EXHIBIT 152
PART 4

CERTIFICATE

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Document Name:	ASTM D5373: Standard Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Laboratory Samples of Coal and Coke
CFR Section(s):	40 CFR 75, Appendix G
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APPROVED

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THE EXECUTIVE DIRECTOR
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WASHINGTON, D.C.

Designation: D 5373 – 93 (Reapproved 1997)

Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Laboratory Samples of Coal and Coke[1]

1. Scope

1.1 These test methods cover the instrumental determination of carbon, hydrogen, and nitrogen in laboratory samples of coal and coke prepared in accordance with Test Methods D 2013 and D 346.

1.2 Within the limitations outlined below, these test methods are applicable to either the air-dry or moisture-free laboratory sample, or both.

1.2.1 For instrumental systems in which the moisture and waters of hydration in the sample are liberated with (and only with) the oxidation products upon combustion, the analyses can be performed on a test specimen of the air-dry sample (Note 1). Concentrations determined on this air-dried basis represent the total carbon (including that present as carbonate), total hydrogen (including that present as water), and total nitrogen.

NOTE 1—These systems are also satisfactory for determining the subject materials in the moisture-free sample.

1.2.2 For systems in which the moisture and hydrates are otherwise liberated, the analysis shall be performed on the moisture-free sample. Values obtained on this basis represent the total carbon, organic hydrogen, and total nitrogen.

1.3 These test methods can be used to provide for the requirements specified in Practice D 3176 for the ultimate analysis.

1.4 The values stated in SI units shall be regarded as the standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in 8.3.1.

2. Referenced Documents

2.1 *ASTM Standards:*

D 346 Test Method for Collection and Preparation of Coke Samples for Laboratory Analysis[2]
D 2013 Test Method for Preparing Coal Samples for Analysis[2]
D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke[2]
D 3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal[2]
D 3176 Practice for Ultimate Analysis of Coal and Coke[2]
D 3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases[2]
D 4621 Guide for Accountability and Quality Control in the Coal Analysis Laboratory[2]
D 5142 Test Methods for the Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures[2]

3. Summary of Test Methods

3.1 Carbon, hydrogen, and nitrogen are determined concurrently in a single instrumental procedure. In some systems, the procedure consists of simply weighing a test specimen, placing the test portion into the instrument, and initiating the (subsequently automatic) analytical process. In other systems, the analytical process may be controlled manually to some degree.

3.2 The actual process can vary substantially from instrument to instrument because a variety of means can be used to effect the primary requirements of the test methods. These test methods provide for the following: (*1*) conversion of the subject materials in an oxygen stream in their entirety to carbon dioxide, water vapor, nitrogen oxides, and ash, respectively; and (*2*) subsequent, quantitative determination of the gases in an appropriate reference gas stream.

3.2.1 The conversion of the subject materials to their corresponding gases occurs largely during combustion of the sample at an elevated temperature in an atmosphere of purified oxygen. The gases that are produced include the following:

3.2.1.1 Carbon dioxide from the oxidation of organic and elemental carbon and the decomposition of carbonate minerals;

3.2.1.2 Hydrogen halides from organic halides (and organic hydrogen, as required);

3.2.1.3 Water vapor from the oxidation of (the remaining) organic hydrogen and the liberation of moisture and waters of hydration;

3.2.1.4 Nitrogen and nitrogen oxides from the oxidation of organic nitrogen and the decomposition of nitrates; and

3.2.1.5 Sulfur oxides from the oxidation of organic sulfur, and the decomposition of sulfide and sulfate minerals.

(*1*) In some systems, sulfurous and sulfuric acids can also be obtained from a combination of the sulfur oxides and the water vapor.

3.2.2 For hydrogen and nitrogen, the required conversion is completed in a two-step process consisting of the following:

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[1] These test methods are under the jurisdiction of ASTM Committee D-5 on Coal and Coke and are the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

Current edition approved March 15, 1993. Published May 1993.

[2] *Annual Book of ASTM Standards*, Vol 05.05.

3.2.2.1 Removal of the halides and sulfur oxides and liberation of the associated hydrogen (as water), by conducting the combustion gases through a series of absorption traps containing appropriate absorbing materials.

3.2.2.2 Reduction of the nitrogen oxides to elemental nitrogen (see Note 2) by passing the resultant gases over copper at an elevated temperature. The carbon dioxide, water vapor, and nitrogen may then be determined via one of several satisfactory detection schemes.

NOTE 2—In this process, residual oxygen is also removed.

3.2.3 In one configuration, the gases are conducted through a series of thermal conductivity detectors and gas absorbers aligned so that, at the water vapor detector level, the gases pass through the sample side of the detector, a water vapor absorber, and the reference side of the detector. At the carbon dioxide detector level, the gases are then conducted through the sample side of the detector, a carbon dioxide absorber, and the reference side of the detector. Finally, the resultant gases, which contain only nitrogen and the carrier gas, pass through the sample side of the nitrogen detector and are vented. At this detector level, high-purity carrier gas is used as the reference gas. In these ways, the detectors determine the thermal conductivities solely of the specified components.

3.2.4 In a second configuration, the carbon dioxide and water vapor are determined by infrared detection, using an aliquot of the combustion gases from which only the halides and sulfur oxides have been removed. These detectors determine the infrared absorption of the pertinent gases at precise wavelength windows so that the absorbances result from only the specified components. In these systems, nitrogen is determined by thermal conductivity, using a second aliquot of the gases, additionally treated to also reduce the nitrogen oxides to nitrogen and to remove the residual oxygen, carbon dioxide, and water vapor.

3.2.5 In a third configuration, which is essentially a modified gas chromatographic system, the nitrogen, carbon dioxide, and water vapor in the treated combustion gases are eluted from a chromatographic column and determined (at appropriate retention times) by thermal conductivity detection.

3.3 In all cases, the concentrations of carbon, hydrogen, and nitrogen are calculated as functions of the following:

3.3.1 Measured instrumental responses,

3.3.2 Values for response per unit mass for the elements (established via instrument calibration), and

3.3.3 Mass of the sample.

3.4 Or to the following: the instrument response is proportional to the gas density, which has been calibrated against a gas density of known concentration.

3.5 A capability for performing these computations automatically can be included in the instrumentation used for these test methods.

4. Significance and Use

4.1 Carbon and hydrogen values are used to determine the amount of oxygen (air) required in combustion processes and for the calculations of efficiency of combustion processes.

4.2 Carbon and hydrogen determinations are used in material balance calculations on coal conversion processes; also, one or the other is used frequently in correlations of chemical and physical properties, such as yields of products in liquefaction reactivity in gasification and the density and porosity of coal.

4.3 Nitrogen data are required to fulfill the requirements of the ultimate analysis, Practice D 3176. Also, the data obtained can be used to evaluate the potential formation of nitrogen oxides as a source of atmospheric pollution.

4.4 Nitrogen data are used for comparing coals and in research. If the oxygen content of coal is estimated by difference, it is necessary to make a nitrogen determination.

5. Apparatus

5.1 Because a variety of instrumental components and configurations can be used satisfactorily for these test methods, no specifications are presented here with respect to overall system design.

5.2 Functionally, however, the following requirements are specified for all approved instruments (Note 3):

NOTE 3—The approval of an instrument with respect to these functions is paramount to these test methods, since such approval tacitly provides approval of both the materials and the procedures used with the system to provide for these functions.

5.2.1 The conditions for combustion of the sample shall be such that (for the full range of applicable samples) the subject components shall be converted completely to carbon dioxide, water vapor (except for hydrogen associated with volatile halides), and nitrogen or nitrogen oxides. Generally, instrumental conditions that effect complete combustion include (*1*) availability of the oxidant, (*2*) temperature, and (*3*) time.

5.2.2 Representative aliquots of the combustion gases shall then be treated for the following reasons:

5.2.2.1 To liberate (as water vapor) hydrogen present as hydrogen halides and sulfur oxyacids; and

5.2.2.2 To reduce (to the element) nitrogen present as nitrogen oxides.

(*1*) The water vapor and nitrogen so obtained shall be included with the materials originally present in these aliquots.

5.2.3 Additional treatment of the test specimens (prior to detection) depends on the detection scheme used for the instrument (Note 4).

NOTE 4—The additional treatments can be provided by the instrumental components used to satisfy 5.2.2.

5.2.3.1 For the configuration described in 3.2.3, the halides proper, sulfur oxides, and residual oxygen shall be removed from the single test specimen in which the water vapor, carbon dioxide, and nitrogen are determined sequentially.

5.2.3.2 For the configuration described in 3.2.4, the test specimen in which the water vapor and carbon dioxide are determined, only the halides and sulfur oxides shall be removed from the gas stream in which the water vapor and carbon dioxide are determined. For combusted gases in which the nitrogen is determined, the water, carbon dioxide, and residual oxygen shall also be removed.

5.2.3.3 For the configuration described in 3.2.5, the halides and sulfur oxides shall be removed from the combusted gases obtained from the single test specimen.

5.2.4 The detection system (in its full scope) shall determine the analytical gases individually and without interference. Additionally, for each analyte, either of the following applies:

5.2.4.1 The detectors themselves shall provide linear responses that correlate directly to concentration over the full range of possible concentrations from the applicable samples, or

5.2.4.2 The system shall include provisions for evaluating nonlinear responses appropriately so that the nonlinear responses can be correlated accurately with these concentrations.

(*1*) Such provisions can be integral to the instrumentation, or they can be provided by (auxiliary) computation schemes.

5.2.5 Finally, except for those systems in which the concentration data are output directly, the instrument shall include an appropriate readout device for the detector responses.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[3] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Helium, Carrier Gas*, as specified by the instrument manufacturer.

6.3 *Oxygen*, as specified by the instrument manufacturer.

6.4 *Additional Reagents*, as specified by the instrument manufacturer. This specification refers to the reagents used to provide for the functional requirements cited in 5.2.2 through 5.2.3.3. These reagents can vary substantially for different instruments; in all cases, however, for systems that are functionally satisfactory (and therefore approved), the reagents recommended by the manufacturer are also tacitly approved. Consequently, these reagents shall be those recommended by the manufacturer.

7. Preparation of Analysis Sample

7.1 The samples shall initially be prepared in accordance with Test Methods D 2013 or D 346.

7.2 If required by characteristics of the instrumental system, reduce the air-dry samples (7.1) typically to pass 75 µm (No. 200 U.S.A. Standard Sieve Series) to obtain test units of the analysis sample in the size range recommended by the instrument manufacturer. If required by characteristics of the instrumental system, as specified in 1.2.2, treat the test specimens in accordance with Test Method D 3173 to provide moisture-free materials solely appropriate for these systems. In this and all subsequent sample handling steps, exercise care to minimize changes in moisture content resulting from exposure to the atmosphere.

8. Instrument Preparation

8.1 Assemble the instrumental system in accordance with the manufacturer's instructions.

8.2 *Adjustment of Response of Measurement System*—Weigh an appropriate test portion of standard reference material (SRM), calibrating agent, or reference coal. Analyze the test portion (see 9.1). Repeat this procedure. Adjust instrument response, as recommended by the manufacturer, until the absence of drift is indicated.

8.3 *Calibration*—Select coal SRMs or other calibrating agents and materials specified by the manufacturer that have certified carbon, hydrogen, and nitrogen values in the range of samples to be analyzed. At least three such SRMs or calibrating agents are recommended for each range of carbon, hydrogen, and nitrogen values to be tested. When possible, two of the SRMs or calibrating agents shall bracket the range of carbon, hydrogen, and nitrogen to be tested, with the third falling within the range.

8.3.1 All coal SRMs should be in accordance with 7.1 and shall be supplied by or have traceability to an internationally recognized certifying organization. **CAUTION:** An indicated problem with linearity of the instrument during calibration can result from contamination of the SRM or calibrating agent as the container becomes depleted. It is therefore recommended that the SRM or calibrating agent be discarded when less than five grams remain in the container.

8.3.2 *Calibration Procedure*—Analyze, as samples, portions of an SRM, reference coal, or calibrating agent chosen to represent the level of carbon, hydrogen, and nitrogen in the samples to be tested. If not required by the characteristics of the instrumental system, use the "as-determined" carbon, hydrogen, and nitrogen values for calibration. These values must have been calculated previously from the certified "dry basis" carbon, hydrogen, and nitrogen values and residual moisture determined using either Test Methods D 3174 or D 5142. Continue analyzing until the results from five consecutive determinations fall within the repeatability interval (see 12.2.1) of these test methods. Calibrate the instrument according to the manufacturer's instructions using these values. Analyze, as samples, two SRMs reference coals or calibrating agents that bracket the range of values to be tested. The results obtained for these samples must be within the stated precision limits of the SRM, reference coal, or calibrating agent, or the calibration procedure must be repeated. Records for all calibrations must be in accordance with Guide D 4621.

8.3.3 *Periodic Calibration Verification and Recalibration*—In accordance with Guide D 4621, analyze a control sample on a periodic basis. Results obtained for the control sample must be within established limits, or all results obtained since the last successful control check must be rejected and the calibration procedure repeated.

9. Procedure

9.1 Analyze a test specimen of the analysis sample in accordance with the manufacturer's instructions.

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[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

10. Calculation

10.1 Calculate the concentrations of carbon, hydrogen, and nitrogen, on the appropriate sample basis, as follows:

$$A = \frac{(B \times C)}{D} \times 100$$

where:

A = % of the analyte,

B = detector response for that analyte,

C = unit mass per detector response established for the analyte during calibration, and

D = mass of test specimen, g.

The calculations can be provided automatically by the instrumental system used for these test methods.

11. Report

11.1 Report results from the carbon, hydrogen, and nitrogen determinations on any of the several common bases that differ solely with respect to moisture. Procedures for converting the as-determined concentrations to the other bases are specified in Practices D 3176 and D 3180.

12. Precision and Bias

12.1 These test methods are highly dependent on the calibration of the equipment.

12.2 The precision of these test methods for the determination of carbon, hydrogen, and nitrogen was calculated from data obtained from coal and coke with the following concentration ranges: carbon (dry-basis) from 48.6 to 90.6 %, hydrogen (dry-basis) from 0.14 to 5.16 %, and nitrogen (dry-basis) from 0.69 to 1.57 %.

12.2.1 *Repeatability*—The difference, in absolute value, between two test results, conducted on portions of the same analysis sample, in the same laboratory, by the same operator, using the same apparatus, shall not exceed the repeatability interval $I(r)$ in more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the repeatability interval, there is reason to question one, or both, of the test results. The repeatability intervals for carbon, hydrogen, and nitrogen are given in Table 1.

TABLE 1 Repeatability and Reproducibility

% Dry Basis	$I(r)$	$I(R)$
Carbon	0.64	2.51
Hydrogen	0.16	0.30
Nitrogen	0.11	0.17

12.2.2 *Example*—Duplicate analyses for carbon exhibited values of 73.26 and 73.62 %. The absolute difference between the two test results is 0.36 %. Since this value does not exceed the $I(r)$ value of 0.64 %, these duplicate analyses are acceptable at the 95 % confidence level.

12.2.3 *Reproducibility*—The difference, in absolute value, between the averages of duplicate determinations conducted in different laboratories on representative samples prepared from the same bulk sample after reducing to 100 % through a 250 Mm (No. 60 U.S.A. Standard Sieve Series) sieve shall not exceed the reproducibility internal $I(R)$ in more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the reproducibility interval, there is reason to question one, or both, of the test results. The reproducibility intervals for carbon, hydrogen, and nitrogen are given in Table 1.

12.2.4 *Example*—Duplicate analysis for hydrogen in one laboratory revealed an average value of 5.15 %, and a value of 4.93 % was obtained in a different laboratory. The difference between the different laboratory value is 0.22 %. Since the laboratory difference is less than the $I(R)$, the two laboratory results are acceptable at the 95 % confidence level.

12.3 *Bias*—Bias is eliminated when the apparatus is calibrated properly against certified reference standards. Proper calibration includes comparison of test data on NIST SRM 1632 or other reagents and materials that have certified carbon, hydrogen, and nitrogen values.

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Document Name: ASTM D5489: Standard Guide for Care Symbols for Care Instructions on Textile Products

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Standard Guide for Care Symbols for Care Instructions Textile Products[1]

This standard is issued under the fixed designation D 5489; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide provides a uniform system of symbols for the disclosure of care instructions on textile products such as apparel, piece goods, and household and institutional articles, hereinafter referred to as "textile," or "textile product."

1.2 This guide provides a comprehensive system of symbols to represent care instructions in order to reduce language-dependent care instructions.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 123 Terminology Relating to Textile Materials[2]

D 3136 Terminology Relating to Care Labels for Textile and Leather Products Other than Textile Floor Coverings and Upholstery[2]

D 3938 Guide for Determining or Confirming Care Instructions for Apparel and Other Textile Consumer Products[3]

2.2 *AATCC Standard:*

A Glossary of AATCC Standard Terminology, Current Edition[4]

2.3 *Other Standards:*

Federal Trade Commission Amendment to Trade Regulation Rule Concerning Care Labeling of Textile Wearing Apparel, and Certain Piece Goods, *Federal Register*, Vol 48, No. 99, May 20, 1983 (cited as 16 CFR 423).[5]

The National Standard of Canada—Care Labelling of Textiles (CAN/CGSB-86.1-M91)[6]

3. Terminology

3.1 *Definitions*—For definitions of terms related to care labeling, refer to Terminology D 3136. For definitions of other textile terms used in this guide, refer to Terminology D 123.

3.1.1 *care instructions, n—in textiles*, a series of directions describing which care practices should refurbish a product without adverse effects, and warnings for those care practices expected to have a harmful effect.

3.1.2 *care label, n—in textiles*, a label or other affixed instuctions that report how a product should be refurbished.

3.1.2.1 *Discussion*—The Federal Trade Commission, in Rule 16 CFK 423, requires care instructions on most apparel and certain other textile items. In relation to these products, the FTC definition states: "Care Label means a permanent label or tag, containing regular care information and instruction, that is attached or affixed in some manner that will not become separated from the product and will remain legible during the useful life of the product."

3.1.3 *care symbol, n*—a pictorial symbol that gives directions for refurbishing a consumer textile product.

4. Significance and Use

4.1 This guide provides symbols and a system for their use by which care instructions for textile products can be conveyed in a simple, space-saving, and easily understood pictorial format that is not language dependent.

4.2 Care symbols are an important means for identifying the appropriate care procedure for home laundering, commercial laundering, professional drycleaning, and coin-operated drycleaning, of textile products.

4.3 Care labeling using symbols can be used by the purchaser to select textiles on the basis of the care method required without knowledge of the language.

4.4 In countries in which a word-based care labeling system is required, the care symbol system may be used as a supplemental system.

4.5 The word-based instructions for each symbol in this guide are harmonious with Terminology D 123, D 3136, the United States Federal Trade Commission Care Labeling Rule, 16 CFR 423 and industry practice. (See Fig. 1 and 2).

4.6 The care label symbol system is based on five basic care symbols representing five operations: washing, bleaching, drying, ironing, and drycleaning.

4.7 One color is used for all care symbols in this care labeling system.

NOTE 1—While this symbol system uses one color, it is harmonious with tri-color systems such as the Canadian system because the instructions are clear whether printed in one or three colors.

4.8 This guide does not specify the type of label material or fabric to use. However, appropriateness for consumer comfort is recommended.

5. Procedure

5.1 *Introduction*—This section defines the basic symbols

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[1] This guide is under the jurisdiction of ASTM Committee D-13 on Textiles and is the direct responsibility of Subcommittee D13.62 on Textile Care Labeling.
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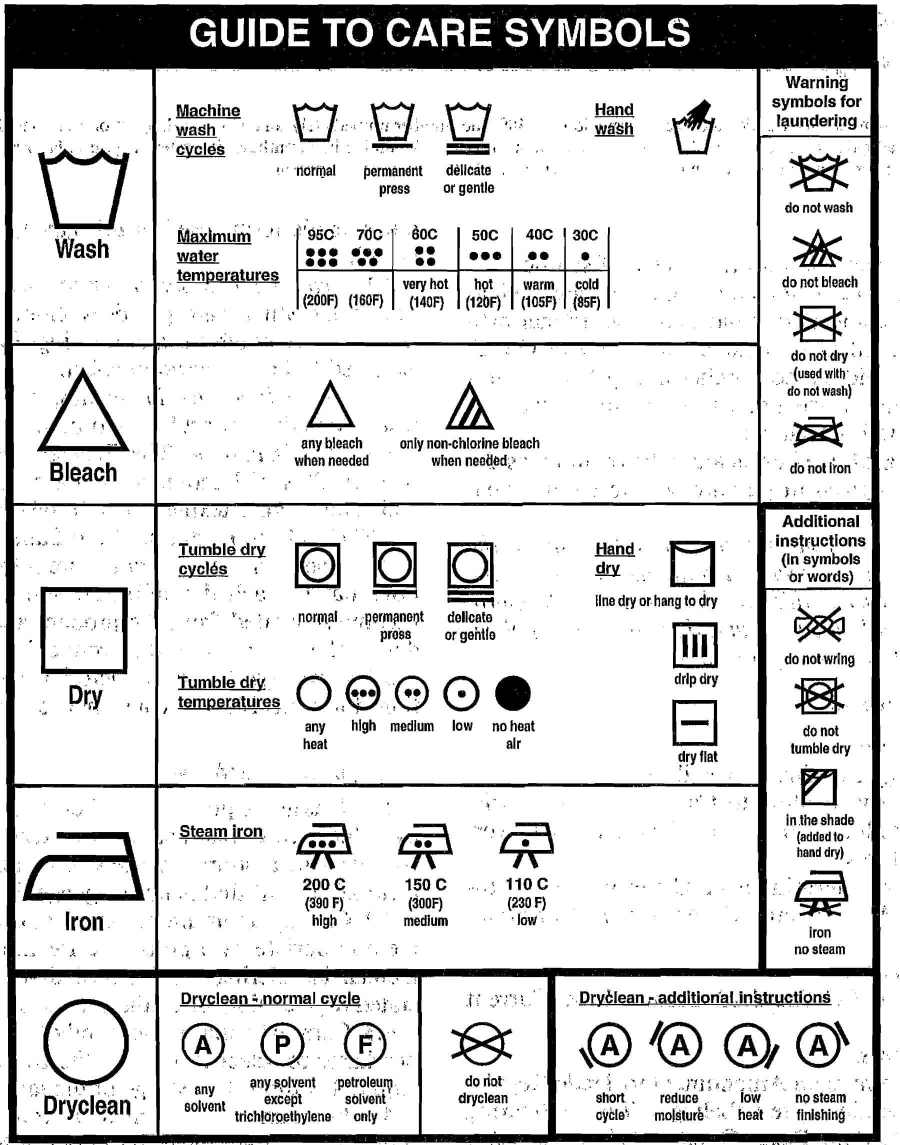
[2] *Annual Book of ASTM Standards*, Vol 07.01.

[3] *Annual Book of ASTM Standards*, Vol 07.02.

[4] *Annual AATCC Technical Manual*, available from the American Association of Textile Chemists and Colorists, P.O. Box 12215, Research Triangle Park, NC 27709.

[5] Available from U.S. Government Printing Office, North Capital and H Streets, NW, Washington, DC 20401.

[6] Available from CGSB, Sales Unit, Ottawa, Canada. (819) 956-0425 or (819) 956-0426.



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NOTE—This figure illustrates the symbols to use for laundering and drycleaning instructions. As a minimum, laundering instructions shall include, in order, four symbols: washing, bleaching, drying, and ironing; and drycleaning instructions shall include one symbol. Additional symbols or words may be used to clarify the instructions.

FIG. 1 Commercial and Home Laundering and Drycleaning Symbols

and prohibitive symbols; the washing, bleaching, drying, ironing, and drycleaning processes; the required number and order of symbols; supplementary care information; and appropriate instructions and labels.

5.2 *Basic Symbols:*

5.2.1 There are five basic symbols: the washtub, triangle, square, iron, and circle.

5.2.2 The washtub represents the washing process, the triangle represents the bleaching process, the square represents the drying process; the iron represents the ironing or pressing process, and the circle represents the drycleaning process (Fig. 3).

5.3 *Prohibitive Symbols*—The prohibitive "X" symbol may be used only when evidence can be provided that the care procedure on which it is superimposed would adversely change the dimensions, hand, appearance, or performance of the textile. (Fig. 4).

5.4 *The Washing Process—The Washtub Symbol:*

5.4.1 The washtub with a water wave represents the washing process in a home laundering or commercial laundering setting.

5.4.2 Additional symbols inside the washtub indicate the suggested water temperature and hand-washing process.

5.4.3 Additional symbols below the tub indicate the permanent press cycle (one underline, minus sign, or bar) and delicate-gentle washing cycle (two underlines, minus signs, or bars).

5.4.4 The numerical or the dot system or both illustrated

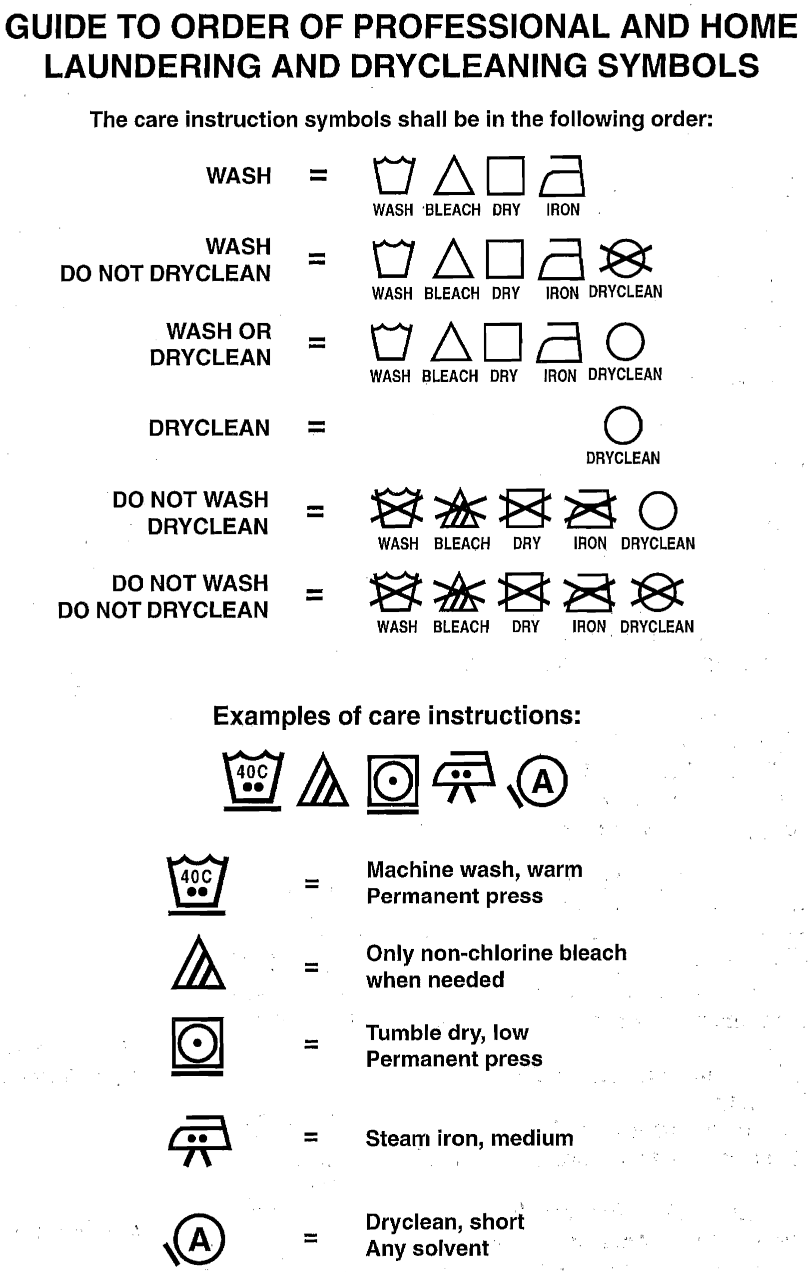

FIG. 2 Guide to Order of Professional and Home Laundering and Drycleaning Symbols

in Fig. 1 may be used to represent the maximum water temperature for machine and hand washing.

5.4.4.1 The six washing temperatures are 30°C (80°F), 40°C (105°F), 50°C (120°F), 60°C (140°F), 70°C (160°F), and 95°C (200°F) and shall be in Celsius when using the numerical water temperature system. The temperature in Farenheit may be included.

NOTE 2—The Fahrenheit temperatures, while not true conversions from Celsius to Fahrenheit, are within the range of tolerance and represent common consumer usage.

5.4.4.2 The symbols used to represent the maximum water temperature in the dot system are: six dots [95°C (200°F)], five dots [70°C (160°F)], four dots [60°C (140°F)], very hot, three dots [50°C (120°F)], hot, two dots [40°C

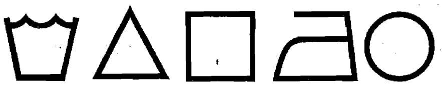

FIG. 3 Basic Symbols

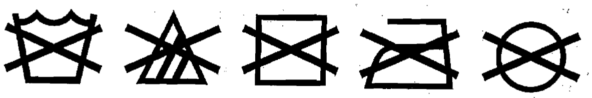

FIG. 4 Prohibitive Symbols

(105°F)], warm, one dot [30°C (85°F)], cool/cold.

5.4.5 To represent the care instruction for a specific wash condition, use the appropriate symbol as illustrated in Fig. 1.

NOTE 3—Consumers may obtain washing machine temperatures that are frequently below the suggested temperature. The actual water temperature obtained when using the washing machine settings of hot, warm, and cold vary in North American by region, season, water heater settings, and regulations governing factory-set mixes. In general, North American washing machines do not have internal heaters.

5.5 *The Bleaching Process—The Triangle Symbol:*

5.5.1 The triangle represents the bleaching process.

5.5.2 The triangle and an additional symbol inside the triangle represent the type(s) of bleach to use.

5.5.3 To represent the care instruction for a specific bleaching condition, use the appropriate symbol as illustrated in Fig. 1.

5.6 *The Drying Process—The Square Symbol:*

5.6.1 The square represents the drying process.

5.6.2 Additional symbols inside the square represent the type of drying process to use, including tumble dry, line dry, drip dry, dry flat, and dry in shade.

5.6.3 Additional symbols below the tumble dry symbol indicate the permanent press cycle (one underline, one minus, or bar) and the delicate-gentle cycle (two underlines, minus signs, bars).

5.6.4 The dot(s) used to represent the dryer temperatures are: three dots (high), two dots (medium), one dot (low), no dots (any heat), and, a solid or filled-in circle (no heat/air).

5.6.5 To represent the care instruction for a specific drying condition, use the appropriate symbol as illustrated in Fig. 1.

5.7 *The Ironing Process—The Hand Iron Symbol:*

5.7.1 The hand iron represents both the hand ironing process and the pressing process on commercial equipment in laundering and cleaning plants.

5.7.2 Additional ironing symbols include dot symbols inside the iron to represent the temperature setting and the steam burst under the iron.

5.7.2.1 The three ironing temperatures are 200°C (392°F), 150°C (302°F), and 110°C (230°F).

5.7.3 To represent the care instruction for a specific ironing condition, use the appropriate symbol as illustrated in Fig. 1.

5.8 *The Drycleaning Process—The Circle Symbol:*

5.8.1 The circle represents the drycleaning process.

5.8.2 A letter enclosed in the drycleaning symbol indicates the type of solvent that is recommended.

5.8.3 Additional symbols with the drycleaning symbol give additional information concerning the drycleaning process.

5.8.4 To represent the care instruction for a specific drycleaning condition, use the appropriate symbol as illustrated in Fig. 1.

5.9 *Required Number and Order of Symbols:*

5.9.1 To represent the laundering process on a care label, a minimum of four care instruction symbols in the following order—washing, bleaching, drying, and ironing—shall be used. (See Fig. 2.)

5.9.1.1 The required symbol may be a prohibitive symbol ("X"), if necessary.

5.9.1.2 Additional warnings and information in symbols or words, as appropriate, shall be placed after or below the four laundering symbols or in the logical refurbishing sequence of the written instructions.

5.9.2 To represent the drycleaning process on a care label, the drycleaning symbol, as a minimum, shall be used. (See Fig. 2.)

5.9.2.1 As appropriate, additional warnings and information shall be placed around, after, or below the drycleaning symbol or in the logical refurbishing sequence of the written instructions.

NOTE 4—The ISO care symbol system requires four care symbols in the following order: washing, bleaching, ironing, and drycleaning. Tumble drying is an optional symbol, which, if used, appears after the four symbols.

TABLE 1 Additional Words to Use with Care Symbols

NOTE—This guide uses symbols to represent many textile refurbishing procedures. Additional words may be needed to clarify specific care procedures. This table of additional words to use with care symbols includes the remaining terms listed in the Federal Trade Commission Care Labeling Glossary of Standard Terms and additional terms in common usage that are not represented by symbols. These terms are illustrative only and are not meant to be an exhaustive list of all terms that might be appropriate or necessary. In general, whatever additional words are needed to state a care procedure that will result in the adequate refurbishment of the item should be used.

Federal Trade Commission Standard Terms	Terms in Common Usage
Preliminary and Laundering Instructions	
do not have commercially laundered	close zippers
small load	do not pretreat
separately	do not soak
with like colors	remove buttons
wash inside out	remove lining
	remove shoulder pads
	remove trim
	wash dark colors separately
warm rinse	wash once before wearing
cold rinse	wash separately
rinse thoroughly	wash with like colors
no spin or do not spin	do not use fabric softener
no wring or do not wring^A	remove promptly
no wring or twist^A	rinse twice
damp wipe only	use oversize washing machine
Bleaching (all terms represented by symbols)	
Drying, All Methods	
no heat^A	tumble dry, air^A
remove promptly	do not tumble dry
line dry in the shade^A	reshape and dry flat
line dry away from heat	block flat to dry
block to dry	
smooth by hand	
Ironing and Pressing	
iron wrong side only	do not iron decal
no steam or do not steam^A	iron reverse side only
steam only	iron right side only
iron damp	warm iron if needed
use press only	use press cloth
Wash or Dryclean (can be represented with symbols)	
Drycleaning, All Methods	
professionally dryclean	fur clean
short cycle^A	use clear solvent
minimum extraction	clear distilled solvent rinse
reduced or low moisture^A	
tumble warm	low heat^A
tumble cool	
cabinet dry warm	
cabinet dry cool	
steam only	
no steam or do not steam	no steam finishing
steam only	
use fluorocarbon solvent	

^{A} Care instruction may be reported in words or a symbol.

5.10 *Supplementary Care Information:*

5.10.1 Additional written care information in English (or the appropriate national language) may be necessary to convey special instructions not covered by the appropriate care symbol or may be legislated by the country of origin. See Fig. 1 and Table 1.

5.10.1.1 For example, the words "remove promptly" shall be added to an article for which this instruction would be appropriate.

5.10.2 The additional words shall appear on the care label in the logical refurbishing sequence.

5.10.3 The written information on a care symbol label shall be brief.

5.10.4 The symbol information and any accompanying detailed written instructions shall be consistent.

5.11 *Appropriate Instructions and Labels:*

5.11.1 It is the responsibility of the manufacturer, or importer to provide the correct information on the care label for refurbishment.

5.11.2 Determining and confirming care instructions requires setting performance specifications, testing, and evaluating the textile product.

5.11.2.1 To aid in determining and confirming appropriate instructions, see Guide D 3938 and published test methods and performance specifications are available in the current *Annual Book of ASTM Standards*, Vols 07.01 and 07.02, and the current Technical Manual of the American Association of Textile Chemists and Colorists (see 2.1 and 2.2).

5.11.3 In the United States, care labels must be attached permanently to apparel except when exempted by the U.S. Federal Trade Commission Regulation (see 2.3).

5.11.4 Permanent care labels shall remain attached and legible for the life of the textile product.

5.11.5 Either the care label or the detailed care instructions shall be visible at the point of purchase.

6. Keywords

6.1 bleaching; care instructions; care label; care symbol; consumer textile product; drycleaning; drying; ironing; permanent care label; pressing; washing

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Standard Test Method for Elements in Water by Inductively Coupled Plasma—Mass Spectrometry[1]

This standard is issued under the fixed designation D 5673; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of dissolved elements in ground water, surface water, and drinking water. It may also be used for the determination of total-recoverable elements in these waters as well as wastewater.[2]

1.2 This test method should be used by analysts experienced in the use of inductively coupled plasma—mass spectrometry (ICP-MS), the interpretation of spectral and matrix interferences and procedures for their correction.

1.3 It is the user's responsibility to ensure the validity of the test method for waters of untested matrices.

1.4 Table 1 lists elements for which the test method applies, with recommended masses and typical estimated instrumental detection limits using conventional pneumatic nebulization. Actual working detection limits are sample dependent and, as the sample matrix varies, these detection limits may also vary. In time, other elements may be added as more information becomes available and as required.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1066 Practice for Sampling Steam[3]
D 1129 Terminology of Terms Relating to Water[3]
D 1192 Specification for Equipment for Sampling Water and Steam in Closed Conduits[3]
D 1193 Specification for Reagent Water
D 3370 Practices for Sampling Water from Closed Conduits[3]

3. Terminology

3.1 *Definitions*—For definitions of other terms used in this test method, refer to Terminology D 1129.

3.2 *Description of Terms Specific to This Standard:*

3.2.1 *calibration blank*—a volume of water containing the same acid matrix as the calibration standards (see 11.1).

3.2.2 *calibration shock solution*—a solution prepared from the stock standard solution(s) to verify the instrument response with respect to analyte concentration.

TABLE 1 Recommended Analytical Mass and Estimated Instrument Detection Limits

Element	Recommended Analytical Mass	Estimated Instrument Detection Limit, µg/L^A
Aluminum	27	0.05
Antimony	121	0.08
Arsenic	75	0.9
Barium	137	0.5
Beryllium	9	0.1
Cadmium	111	0.1
Chromium	52	0.07
Cobalt	59	0.03
Copper	63	0.03
Lead	206, 207, 208	0.08
Manganese	55	0.1
Molybdenum	98	0.1
Nickel	60	0.2
Selenium	82	5.0
Silver	107	0.05
Thallium	205	0.09
Thorium	232	0.03
Uranium	238	0.02
Vanadium	51	0.02
Zinc	66	0.2

^{A} Instrument detection limits (3σ) estimated from seven replicate scans of the blank (1 % v/v HNO_3) and three replicate integrations of a multi-element standard.

3.2.3 *calibration standards*—a series of known standard solutions used by the analyst for calibration of the instrument (that is, preparation of the analytical curve) (see 11.).

3.2.4 *dissolved*—those elements that will pass through a 0.45-µm membrane filter.

3.2.5 *instrumental detection limit*—the concentration equivalent to a signal which is equal to three times the standard deviation of the blank signal at the selected analytical mass(es).

3.2.6 *internal standard*—pure analyte(s) added in known amount(s) to a solution. This is used to measure the relative instrument response to the other analytes that are components of the same solution. The internal standards must be analytes that are not a sample component.

3.2.7 *method detection limit*—the minimum concentration of an analyte that can be identified, measured and reported with 99 % confidence that the analyte concentration is greater than zero. This confidence level is determined from analysis of a sample in a given matrix containing the analyte(s).

3.2.8 *quality control reference solution*—a solution with the certified concentration(s) of the analytes, prepared by an independent laboratory, and used for a verification of the instrument's calibration.

3.2.9 *reagent blank*—a volume of water containing the

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[1] This test method is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

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[2] EPA Test Method: Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma—Mass Spectrometry, Method 200.8.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

same matrix as the calibration standards, carried through the entire analytical procedure.

3.2.10 *total-recoverable*—a term relating to forms of each element that are determinable by the digestion method included in this procedure (see 12.2).

3.2.11 *tuning solution*—a solution that is used to determine acceptable instrument performance prior to calibration and sample analysis.

4. Summary of Test Method

4.1 This test method describes the multi-element determination of trace elements by inductively coupled plasma—mass spectrometry (ICP-MS). Sample material in solution is introduced by pneumatic nebulization into a radiofrequency plasma where energy transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio by a quadrupole mass spectrometer. The ions transmitted through the quadrupole are detected by a continuous dynode electron multiplier assembly and the ion information processed by a data handling system. Interferences relating to the technique must be recognized and corrected for (see Section 7 on interferences). Such corrections must include compensation for isobaric elemental interferences and interferences from polyatomic ions derived from the plasma gas, reagents, or sample matrix. Instrumental drift as well as suppressions or enhancements of instrument response caused by the sample matrix must be corrected for by the use of internal standardization.

5. Significance and Use

5.1 The test method is useful for the determination of element concentrations in many natural waters and wastewaters. It has the capability for the determination of up to 20 elements. High analysis sensitivity can be achieved for some elements that are difficult to determine by other techniques.

6. Interferences

6.1 Several types of interference effects may contribute to inaccuracies in the determination of trace elements. These interferences can be summarized as follows:

6.1.1 *Isobaric Elemental Interferences*—Isobaric elemental interferences are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. All elements determined by this test method have, at a minimum, one isotope free of isobaric elemental interference. Of the analytical isotopes recommended for use with this test method (see Table 2), only molybdenum-98 (ruthenium) and selenium-82 (krypton) have isobaric elemental interferences. If alternative analytical isotopes having higher natural abundance are selected in order to achieve greater sensitivity, an isobaric interference may occur. All data obtained under such conditions must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest. A record of this correction process should be included with the report of the data. It should be noted that such corrections will only be as accurate as the accuracy of the isotope ratio used in the elemental equation for data calculations. Relevant isotope ratios and instrument bias factors should be established prior to the application of any corrections.

TABLE 2 Recommended Analytical Isotopes and Additional Masses That Are Recommended To Be Monitored

Isotope[A]	Element of Interest
27	Aluminum
121, 123	Antimony
75	Arsenic
135, 137	Barium
9	Beryllium
106, 108, 111, 114	Cadmium
52, 53	Chromium
59	Cobalt
63, 65	Copper
206, 207, 208	Lead
55	Manganese
95, 97, 98	Molybdenum
60, 62	Nickel
77, 82	Selenium
107, 109	Silver
203, 205	Thallium
232	Thorium
238	Uranium
51	Vanadium
66, 67, 68	Zinc
83	Krypton
99	Ruthenium
105	Palladium
118	Tin

[A] Isotopes recommended for analytical determination are underlined. These masses were recommended and are reflected in the precision and bias data. Alternate masses may be used but interferences must be documented.

6.1.2 *Abundance Sensitivity*—Abundance sensitivity is a property defining the degree to which the wings of a mass peak contribute to adjacent masses. The abundance sensitivity is affected by ion energy and quadrupole operating pressure. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one. The potential for these interferences should be recognized and the spectrometer resolution adjusted to minimize them.

6.1.3 *Isobaric Polyatomic Ion Interferences*—Isobaric polyatomic ion interferences are caused by ions consisting of more than one atom that have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer in use. These ions are commonly formed in the plasma or interface system from support gases or sample components. Most of the common interferences have been identified, and these are listed in Table 3 together with the method elements affected. Such interferences must be recognized, and when they cannot be avoided by the selection of an alternative analytical isotope, appropriate corrections must be made to the data. Equations for the correction of data should be established at the time of the analytical run sequence as the polyatomic ion interferences will be highly dependent on the sample matrix and chosen instrument conditions.

6.1.4 *Physical Interferences*—Physical interferences are associated with the physical processes that govern the transport of the sample into the plasma, sample conversion processes in the plasma, and the transmission of ions through the plasma—mass spectrometer interface. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the

TABLE 3 Common Molecular Ion Interferences

	Background Molecular Ions	
Molecular Ion	Mass	Element Interference[A]
NH^+	15	. . .
OH^+	17	. . .
OH_2^+	18	. . .
C_2^+	24	. . .
CN^+	26	. . .
CO^+	28	. . .
N_2^+	28	. . .
N_2H^+	29	. . .
NO^+	30	. . .
NOH^+	31	. . .
O_2^+	32	. . .
O_2H^+	33	. . .
$^{36}ArH^+$	37	. . .
$^{36}ArH^+$	39	. . .
$^{40}ArH^+$	41	. . .
CO_2^+	44	. . .
CO_2H^+	45	Sc
ArC^+, ArO^+	52	Cr
ArN^+	54	Cr
$ArNH^+$	55	Mn
ArO^+	56	. . .
$ArOH^+$	57	. . .
$^{40}Ar^{36}Ar^+$	76	Se
$^{40}Ar^{38}Ar^+$	78	Se
$^{40}Ar_2^+$	80	Se
	Matrix Molecular Ions	
Chloride		
$^{35}ClO^+$	51	V
$^{35}ClOH^+$	52	Cr
$^{37}ClO^+$	53	Cr
$^{37}ClOH^+$	54	Cr
$Ar^{35}Cl^+$	75	As
$Ar^{37}Cl^+$	77	Se
Sulphate		
$^{32}SO^+$	48	. . .
$^{32}SOH^+$	49	. . .
$^{34}SO^+$	50	V, Cr
$^{34}SOH^+$	51	V
SO_2^+, S_2^+	64	Zn
$Ar^{32}S^+$	72	. . .
$Ar^{34}S^+$	74	. . .
Phosphate		
PO^+	47	. . .
POH^+	48	. . .
PO_2^+	63	Cu
ArP^+	71	. . .
Group I, II Metals		
$ArNa^+$	63	Cu
ArK^+	79	. . .
$ArCa^+$	80	. . .
Matrix Oxides[B]		
TiO	62 to 66	Ni, Cu, Zn
ZrO	106 to 112	Ag, Cd
MoO	108 to 116	Cd

[A] Method elements or internal standards affected by molecular ions.

[B] Oxide interferences will normally be very small and will only impact the method elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that Ti and Zr isotopes be monitored if samples are likely to contain high levels of these elements. Mo is monitored as a method analyte.

nebulizer (for example, viscosity effects), at the point of aerosol formation and transport to the plasma (for example, surface tension), or during excitation and ionization processes within the plasma itself. High levels of dissolved solids in the sample may contribute deposits of material on the extraction, or skimmer cones, or both, reducing the effective diameter of the orifices and, therefore, ion transmission. Dissolved solids levels not exceeding 0.2 % (w/v) have been recommended to reduce such effects. Internal standardization may be effectively used to compensate for many physical interference effects. Internal standards should have similar analytical behavior to the elements being determined.

6.1.5 *Memory Interferences*—Memory interferences result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the sampler and skimmer cones, and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank consisting of HNO_3 (1+49) in water between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element should be estimated prior to analysis. This may be achieved by aspirating a standard containing elements corresponding to ten times the upper end of the linear range for a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of ten of the method detection limit should be noted. Memory interferences may also be assessed within an analytical run by using a minimum of three replicate integrations for data acquisition. If the integrated signal values drop consecutively, the analyst should be alerted to the possibility of a memory effect, and should examine the analyte concentration in the previous sample to identify if this was high. If a memory interference is suspected, the sample should be re-analyzed after a long rinse period.

7. Apparatus

7.1 *Inductively Coupled Plasma-Mass Spectrometer*—Instrument capable of scanning the mass range 5 to 250 amu with a minimum resolution capability of 1 amu peak width at 5 % peak height. Instrument may be fitted with a conventional or extended dynamic range detection system. See manufacturers instruction manual for installation and operation.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that reagents shall conform to the specifications of the committee on analytical reagents of the American Chemical Society,[4] where such specifications are available. The high sensitivity of inductively coupled plasma—mass spectrometry may require reagents of higher purity. Stock standard solutions are prepared from high-purity metals, oxides, or non-hydroscopic reagent grade salts using Type I, II, or III reagent water and ultrapure acids. Other grades may be used provided it is first ascertained that the reagent is of sufficient

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[4] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Type I, II, or III of Specification D 1193. It is the analyst's responsibility to insure that water is free of interferences.

8.3 *Argon*—High purity grade (99.99 %).

8.4 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl), ultrapure or equivalent.

8.5 *Hydrochloric Acid* (1+1)—Add one volume of hydrochloric acid (sp gr 1.19) to 1 volume of water.

8.6 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3), ultrapure or equivalent.

8.7 *Nitric Acid* (1+1)—Add one volume of nitric acid (sp gr 1.42) to 1 volume of water.

8.8 *Nitric Acid* (1+49)—Add one volume of nitric acid (sp gr 1.42) to 49 volumes of water.

8.9 *Nitric Acid* (1+99)—Add one volume of nitric acid (sp gr 1.42) to 99 volumes of water.

8.10 *Stock Solutions*—Preparation procedures for stock solutions of each element are listed in Table 4.

8.11 *Ammonium Hydroxide* (sp gr 0.902)—Concentrated ammonium hydroxide (NH_4OH), ultrapure or equivalent.

8.12 *Mixed Standard Solutions*—Prepare mixed standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks (see Note 1). Prior to preparing mixed standard solutions, each stock solution needs to be analyzed separately to determine possible interferences on the other analytes or the presence of impurities. Care needs to be taken when preparing the mixed standard solutions to ensure that the elements are compatible and stable.

TABLE 4 Preparation of Metal Stock Solutions[A]

Element or Compound	Weight, g	Solvent
Al	0.1000	10 mL of HCl (sp gr 1.19) + 2 mL of HNO_3 (sp gr 1.42)
Sb	0.1000	0.5 mL of HCl (sp gr 1.19) + 2 mL of HNO_3 (1+1)
As_2O_3	0.1320	1 mL of NH_4OH (sp gr 0.902) + 50 mL of H_2O
$BaCO_3$	0.1437	2 mL of HNO_3 (sp gr 1.42) + 10 mL of H_2O
$BeSO_4 \cdot 4H_2O$	1.9650	50 mL of H_2O, add 1 mL of HNO_3 (sp gr 1.42)
Bi_2O_3	0.1115	5 mL of HNO_3 (sp gr 1.42)
Cd	0.1000	5 mL of HNO_3 (1+1)
CrO_3	0.1923	1 mL of HNO_3 (sp 1.42) + 10 mL H_2O
Co	0.1000	5 mL of HNO_3 (1+1)
Cu	0.1000	5 mL of HNO_3 (1+1)
In	0.1000	10 mL of HNO_3 (1+1)
$PbNO_3$	0.1599	5 mL of HNO_3 (1+1)
MgO	0.1658	10 mL of HNO_3 (1+1)
Mn	0.1000	5 mL of HNO_3 (1+1)
MoO_3	0.1500	1 mL of NH_4OH (sp gr 0.902) + 10 mL of H_2O
Ni	0.1000	5 mL of HNO_3 (sp gr 1.42)
Sc_2O_3	0.1534	5 mL of HNO_3 (1+1)
SeO_2	0.1405	20 mL of H_2O
Ag	0.1000	5 mL of HNO_3 (1+1)
Tb_4O_7	0.1176	5 mL of HNO_3 (sp gr 1.42)
$TlNO_3$	0.1303	1 mL of HNO_3 (sp gr 1.42) + 10 mL of H_2O
$Th(NO_3)_4 \cdot 4H_2O$	0.2380	20 mL of H_2O
$UO_2(NO_3)_2 \cdot 6H_2O$	0.2110	20 mL of H_2O
V	0.1000	5 mL of HNO_2 (1+1)
Y_2O_3	0.1270	5 mL of HNO_3 (1+1)
Zn	0.1000	5 mL of HNO_3 (1+1)

[A] Metal stock solutions, 1.00 mL = 1000 µg of metal. Dissolve the listed weights of each metal or compound as specified in Table 4, then dilute to 100 mL with water. The metals may require heat to increase rate of dissolution. Commercially available standards may be used. Alternate salts or oxides may also be used.

NOTE 1—Mixed calibration standards will vary, depending on the number of elements being determined. Commercially prepared mixed calibration standards of appropriate quality may be used. An example of mixed calibration standards for 20 elements is as follows:

Mixed Standard Solution I		Mixed Standard Solution II
Aluminum	Manganese	Barium
Antimony	Molybdenum	Silver
Arsenic	Nickel	
Beryllium	Selenium	
Cadmium	Thallium	
Chromium	Thorium	
Cobalt	Uranium	
Copper	Vanadium	
Lead	Zinc	

Prepare multi-element mixed standard solutions I and II (1 mL = 10 µg) by pipeting 1.00 mL of each single element stock solution (see Table 4) onto a 100 mL volumetric flask. Add 50 mL of HNO_3 (1+99) and dilute to 100 mL with HNO_3 (1+99).

8.13 *Reagent Blank*—This solution must contain all the reagents and be the same volume as used in the processing of the samples. Carry reagent blank through the complete procedure. Reagent blank must contain the same acid concentration in the final solution as the sample solution used for analysis.

8.14 *Internal Standards*—Internal standards are recommended in all analyses to correct for instrument drift and physical interferences. A list of acceptable internal standards is provided in Table 5. For full mass range scans use a minimum of three internal standards with the use of five suggested. Add internal standards to blanks, samples and standards in a like manner. A concentration of 100 µg/L of each internal standard is recommended.

9. Hazards

9.1 The toxicity or carcinogenicity of each reagent used in this test method has not been precisely defined; however, each chemical should be treated as a potential health hazard. Adequate precautions should be taken to minimize exposure of personnel to chemicals used in this test method.

10. Sampling

10.1 Collect the samples in accordance with the applicable standards, Practice D 1066, Specification D 1192, or Practices D 3370.

10.2 Preserve the samples at the time of collection by immediately adding nitric acid (sp gr 1.42) to adjust the pH to 2. Normally, 2 mL of HNO_3 (sp gr 1.42) is required per

TABLE 5 Internal Standards and Limitations of Use

Internal Standard	Mass	Possible Limitation
Lithium	6	May be present in samples
Scandium[A]	45	Polyatomic ion interference
Yttrium[A]	89	May be present in samples
Rhodium	103	. . .
Indium[A]	115	Isobaric interference by Sn
Tarbium[A]	159	. . .
Holmium	165	. . .
Lutetium	175	. . .
Bismuth[A]	209	May be present in samples

[A] Internal standards recommended for use with this test method. It is also recommended when analyzing a new sample matrix that a scan for the presence of internal standards be performed.

litre of sample. If only dissolved elements are to be determined, filter the sample through a 0.45-µm membrane filter before acidification (see Note 2).

NOTE 2—Depending on the manufacturer, some filters have been found to be contaminated to various degrees with heavy metals. Care should be exercised in selecting a source of these filters. A good practice is to wash the filters first with HNO_3 (1+99) and then with 50 mL of the sample before filtering.

11. Calibration and Standardization

11.1 Calibrate the instrument for the elements chosen over a suitable concentration range by atomizing the calibration blank and mixed standard solutions and recording their concentrations and signal intensities. It is recommended that a minimum of three standards and a blank be used for calibration with one of the standards at three to five times the elements' MDL. It is recommended that the calibration blank and standards be matrix matched with the same acid concentration contained in the samples. Analyze appropriate reference solutions to validate the calibration of the instrument before proceeding to the sample analysis.

12. Procedure

12.1 To determine dissolved elements, add 1 mL of concentrated HNO_3 (sp gr 1.42) to 100 mL of filtered, acid-preserved sample. Proceed with 12.3.

12.2 When determining total-recoverable elements, use 100 mL of a well mixed, acid-preserved sample appropriate for the expected level of elements containing not more than 0.25 % (w/v) total solids.

12.2.1 Transfer the sample to a 125 mL (or larger) beaker or flask and add 2 mL of HNO_3 (1+1) and 1 mL HCl (1+1) and heat on a steam bath or hot plate until the volume has been reduced to near 25 mL, making certain the sample does not boil. Cool the sample, and if necessary, filter or let insoluble material settle to avoid clogging of the nebulizer. Adjust to original sample volume. To determine total-recoverable elements, proceed with 12.3. This method is suitable for the determination of silver in aqueous samples containing concentrations up to 0.1 mg/L. For the analysis of samples containing higher concentrations of silver, succeeding smaller volume, well mixed sample aliquots must be prepared until the analysis solution contains <0.1 mg/L silver.

12.3 Atomize each solution and record signal's intensity or calculated concentration for each mass of interest. Atomize a rinse blank consisting of HNO_3 (1+49) in water between samples.

12.4 Minimum quality control requirements for this method include: initial demonstration of method performance, monitoring of internal standard area counts in each sample (area of internal standard should be within 60–125 % of area in calibration blank), analysis of one reagent blank with each set of samples as continuing check on sample contamination, analysis of a quality control sample with each set of samples as a continuing check on method reference sample recovery, and analysis of calibration check standard every ten analyses as a continuing check on calibration curve (measured values should not exceed ±10 % of concentration).

12.4.1 Demonstrate initial, and continuing, method performance every six months by digesting seven spiked reagent water samples at two through five times estimated detection limit to determine method detection limits (MDL).

12.4.2 Determine detection limits annually or whenever a significant change in background or instrument response is expected.

$$MOL = (t) \times (s)$$

where:

t = students' t value for a 99 % confidence level and with $n-1$ degrees of freedom ($t = 3.14$ for seven replicates), and

s = standard deviation of the replicate analyses.

13. Calculation

13.1 Elemental equations recommended for sample data calculations are listed in Table 6.

13.2 Reagent blanks should be subtracted as appropriate (see 9.13) from the samples. This subtraction is particularly important for digested samples requiring large quantities of acids to complete the digestion (see Note 3).

TABLE 6 Recommended Elemental Equations for Data Calculation

Element	Elemental Equation[A]	Note
Al	$(1.000)\ (^{27}C)$	. . .
Sb	$(1.000)\ (^{121}C)$	. . .
As	$(1.000)\ (^{75}C) - (3.127)\ [(^{77}C) - (0.815)\ (^{82}C)]$	B
Ba	$(1.000)\ (^{137}C)$	. . .
Be	$(1.000)\ (^{9}C)$	. . .
Cd	$(1.000)\ (^{111}C) - (1.073)\ [(^{108}C) - (0.712)\ (^{106}C)]$	C
Cr	$(1.000)\ (^{52}C)$	D
Co	$(1.000)\ (^{59}C)$	. . .
Cu	$(1.000)\ (^{63}C)$	. . .
Pb	$(1.000)\ (^{206}C) + (1.000)\ (^{207}C) + (1.000)\ (^{208}C)$	E
Mn	$(1.000)\ (^{55}C)$	. . .
Mo	$(1.000)\ (^{98}C) - (0.146)\ (^{99}C)$	F
Ni	$(1.000)\ (^{60}C)$	. . .
Se	$(1.000)\ (^{82}C)$	G
Ag	$(1.000)\ (^{107}C)$	. . .
Tl	$(1.000)\ (^{205}C)$	. . .
Th	$(1.000)\ (^{232}C)$	. . .
U	$(1.000)\ (^{238}C)$	. . .
V	$(1.000)\ (^{51}C) - (3.127)\ [(^{53}C) - (0.113)\ (^{52}C)]$	H
Zn	$(1.000)\ (^{66}C)$	. . .
Kr	$(1.000)\ (^{83})$	. . .
Pd	$(1.000)\ (^{105})$	. . .
Ru	$(1.000)\ (^{99})$	. . .
Sn	$(1.000)\ (^{118})$	. . .
Bi	$(1.000)\ (^{209}C)$	I
In	$(1.000)\ (^{115}C) - (0.016)\ (^{118}C)$	J
Sc	$(1.0000)\ (^{45}C)$	K
Tb	$(1.000)\ (^{159}C)$	. . .
Y	$(1.000)\ (^{89}C)$	I

[A] C = calibration blank subtracted counts at specified mass.

[B] Correction for chloride interference with adjustment for Se77. ArCl 75/77 ratio may be determined from the reagent blank.

[C] Correction for MoO interference. An additional isobaric elemental correction should be made if palladium is present.

[D] In 0.4 % v/v HCl, the background from ClOH will normally be small. However the contribution may be estimated from the reagent blank.

[E] Allowance for isotopic variability of lead isotopes.

[F] Isobaric elemental correction for ruthenium.

[G] Some argon supplies contain krypton as an impurity. Selenium is corrected for Kr82 by background subtraction.

[H] Correction for chloride interference with adjustment for Cr53. ClO 51/53 ratio may be determined from the reagent blank.

[I] May be present in environmental samples.

[J] Isobaric elemental correction for tin.

[K] Polyatomic ion interference.

TABLE 7 Regression Equations for Bias and Precision, µg/L, Reagent Water

Element	Applicable Range	Bias	Overall Precision	Single Analyst Precision
Antimony	2.80 to 100	$X = 0.999C + 0.04$	$S_t = 0.013X + 0.61$	$S_o = 0.022X + 0.20$
Arsenic	8.00 to 200	$X = 1.013C + 0.50$	$S_t = 0.031X + 2.74$	$S_o = 0.007X + 2.95$
Barium	8.01 to 200	$X = 1.001C - 0.36$	$S_t = 0.039X + 0.31$	$S_o = 0.024X + 0.25$
Beryllium	2.80 to 100	$X = 1.056C + 0.32$	$S_t = 0.067X + 0.55$	$S_o = 0.038X + 0.11$
Cadmium	4.00 to 100	$X = 1.007C + 0.07$	$S_t = 0.041X + 0.19$	$S_o = 0.022X + 0.10$
Chromium	8.00 to 200	$X = 1.017C + 0.62$	$S_t = 0.066X + 0.48$	$S_o = 0.026X + 1.25$
Cobalt	0.80 to 101	$X = 0.977C + 0.01$	$S_t = 0.28X + 0.06$	$S_o = 0.027X + 0.02$
Copper	4.01 to 100	$X = 1.003C - 0.05$	$S_t = 0.037X + 0.64$	$S_o = 0.016X + 0.51$
Lead	4.00 to 100	$X = 1.043C - 0.31$	$S_t = 0.064X + 1.43$	$S_o = 3.42$
Manganese	8.00 to 200	$X = 0.983C + 0.02$	$S_t = 0.026X + 0.11$	$S_o = 0.027X + 0.06$
Molybdenum	2.80 to 100	$X = 1.012C + 0.20$	$S_t = 0.032X + 0.22$	$S_o = 0.021X + 0.09$
Nickel	4.00 to 100	$X = 1.000C + 0.12$	$S_t = 0.051X + 0.31$	$S_o = 0.017X + 0.40$
Selenium	32.00 to 200	$X = 1.036C - 0.06$	$S_t = 0.051X + 3.24$	$S_o = 0.061X - 0.64$
Silver	0.80 to 200	$X = 0.917C + 0.26$	$S_t = 0.196X + 0.09$	$S_o = 0.053X + 0.08$
Thallium	2.80 to 100	$X = 0.984C + 0.08$	$S_t = 0.035X + 0.09$	$S_o = 0.027X + 0.13$
Thorium	0.80 to 100	$X = 1.0913 + 0.08$	$S_t = 0.036X + 0.13$	$S_o = 0.025X + 0.07$
Uranium	0.80 to 100	$X = 1.026C - 0.02$	$S_t = 0.048X + 0.02$	$S_o = 0.027X + 0.05$
Zinc	8.00 to 200	$X = 1.042C + 0.87$	$S_t = 0.041X + 2.60$	$S_o = 0.030X + 1.42$

TABLE 8 Regression Equations for Bias and Precision, µg/L, Drinking Water

Element	Applicable Range	Bias	Overall Precision	Single Analyst Precision
Antimony	2.80 to 100	$X = 0.983C + 0.03$	$S_t = 0.049X + 0.19$	$S_o = 0.026X + 0.08$
Arsenic	8.00 to 200	$X = 0.993C + 0.57$	$S_t = 0.018X + 2.55$	$S_o = 0.031X + 1.65$
Barium	8.01 to 200	$X = 0.995C - 0.37$	$S_t = 0.045X + 0.97$	$S_o = 0.040X + 0.72$
Beryllium	2.80 to 100	$X = 1.055C + 0.20$	$S_t = 0.057X + 0.28$	$S_o = 0.016X + 0.25$
Cadmium	4.00 to 100	$X = 0.985C + 0.10$	$S_t = 0.031X + 0.65$	$S_o = 0.021X + 0.61$
Chromium	8.00 to 200	$X = 0.990C + 1.45$	$S_t = 0.015X + 2.19$	$S_o = 2.18$
Cobalt	0.80 to 101	$X = 0.964C + 0.06$	$S_t = 0.19X + 0.32$	$S_o = 0.014X + 0.30$
Copper	4.01 to 100	$X = 0.976C - 0.38$	$S_t = 0.063X + 0.86$	$S_o = 0.029X + 0.86$
Lead	4.00 to 100	$X = 1.032C - 0.30$	$S_t = 0.015X + 1.06$	$S_o = 0.011X + 1.13$
Molybdenum	2.80 to 100	$X = 1.013C + 0.07$	$S_t = 0.037X + 0.17$	$S_o = 0.035X + 0.20$
Nickel	4.00 to 100	$X = 0.953C - 0.19$	$S_t = 0.046X + 0.56$	$S_o = 0.023X + 0.91$
Selenium	32.00 to 200	$X = 1.022C + 0.14$	$S_t = 0.056X + 2.10$	$S_o = 0.040X + 2.15$
Thallium	2.80 to 100	$X = 1.010C + 0.01$	$S_t = 0.040X + 0.21$	$S_o = 0.039X + 0.02$
Uranium	0.80 to 100	$X = 1.026C - 0.04$	$S_t = 0.044X + 0.11$	$S_o = 0.022X + 0.07$
Vanadium	32.00 to 200	$X = 1.022C + 0.30$	$S_t = 0.023X + 1.45$	$S_o = 0.023X + 1.38$

NOTE 3—Reagent blank concentrations if the levels can influence the sample results.

13.3 If dilutions were required, apply the appropriate dilution factor to sample values.

13.4 Report results in the calibration concentration units.

14. Precision and Bias[5]

14.1 The precision and bias data for this test method are based on a joint interlaboratory method validation study conducted by the U.S. Environmental Protection Agency and the Association of Official Analytical Chemists.[6]

14.2 The test design of the study meets the requirements of Practice D 2777 for elements listed in this test method with the following exceptions. Thorium in drinking water and vanadium in ground and reagent water did not meet the requirements of 10.3 in Practice D 2777 and are deleted in the test method. In addition, the following elements did not meet the requirements of 10.5 of Practice D 2777 for the concentration levels (in µg/L) tested: Aluminum in reagent (8) and drinking water (12), antimony in ground water (2.0 and 100), manganese in ground (0.8 and 1.2) and drinking water (1.2), molybdenum in ground water (2.8), silver, in ground (0.8 and 2.2) and drinking water (0.8 and 1.2), and zinc in drinking water (8).

14.2.1 The regression equations are based on the results of 13 laboratories for 20 elements tested at six levels, they are outlined in Tables 7, 8, and 9 for reagent water, drinking water, and ground water, respectively.

14.2.2 Type I water was specified for this round robin.

14.2.3 These data may not apply to waters of other matrices; therefore, it is the responsibility of the analyst to ensure the validity of the test method in a particular matrix.

14.2.4 It is the user's responsibility to ensure the validity of precision and bias outside the joint interlaboratory method validation study ranges.

15. Keywords

15.1 elements; mass spectrometry; water

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[5] Supporting data are available from ASTM Headquarters. Request RR:D19-1157.

[6] Longbottom, J. E., et al., "Determination of Trace Elements in Water by Inductively Coupled Plasma-Mass Spectrometry: Collaborative Study," *Journal of AOAC, International* 77, 1994, pp. 1004–1023.

TABLE 9 Regression Equations for Bias and Precision, µg/L, Ground Water

Element	Applicable Range	Bias	Overall Precision	Single Analyst Precision
Aluminum	8.00 to 200	$X = 0.946C + 2.20$	$S_t = 0.169X + 6.22$	$S_o = 0.172X + 0.75$
Arsenic	8.00 to 200	$X = 0.949C + 0.91$	$S_t = 0.048X + 4.52$	$S_o = 0.059X + 4.29$
Barium	8.01 to 200	$X = 1.055C - 0.21$	$S_t = 0.020X + 2.05$	$S_o = 0.014X + 2.08$
Beryllium	2.80 to 100	$X = 1.049C + 0.08$	$S_t = 0.084X + 0.16$	$S_o = 0.043X + 0.06$
Cadmium	4.00 to 100	$X = 0.944C + 0.11$	$S_t = 0.017X + 1.09$	$S_o = 0.029X + 0.01$
Chromium	8.00 to 200	$X = 1.026C + 0.89$	$S_t = 0.067X + 0.68$	$S_o = 0.068X - 0.37$
Cobalt	0.80 to 101	$X = 0.989C - 0.01$	$S_t = 0.057X + 0.09$	$S_o = 0.012X + 0.40$
Copper	4.01 to 100	$X = 0.977C - 0.01$	$S_t = 0.073X + 0.92$	$S_o = 0.077X + 0.35$
Lead	4.00 to 100	$X = 1.012C + 0.15$	$S_t = 0.048X + 1.27$	$S_o = 1.78$
Nickel	4.00 to 100	$X = 1.022C - 0.66$	$S_t = 0.091X + 2.03$	$S_o = 0.008X + 2.75$
Selenium	32.00 to 200	$X = 1.045C - 0.83$	$S_t = 0.037X + 2.97$	$S_o = 0.058X + 1.02$
Thallium	2.80 to 100	$X = 1.023C - 0.06$	$S_t = 0.056X + 0.04$	$S_o = 0.049X - 0.06$
Thorium	0.80 to 100	$X = 1.019C - 0.03$	$S_t = 0.041X + 0.13$	$S_o = 0.027X + 0.04$
Uranium	0.80 to 100	$X = 1.058C - 0.06$	$S_t = 0.039X + 0.17$	$S_o = 0.028X + 0.16$
Zinc	8.00 to 200	$X = 0.962C + 0.07$	$S_t = 0.093X + 0.92$	$S_o = 0.069X + 1.55$

APPENDIX

(Nonmandatory Information)

X1. PRECISION AND BIAS

X1.1 The following regression expressions in Table X1.1 are based on the measured values, X, S_t, and S_o that were derived from data of the joint interlaboratory method validation study conducted by the U.S. Environmental Protection Agency and the Association of Official Analytical Chemists,[6] some of which do not meet the requirements of 10.5 of Practice D 2777, as noted in 14.2 of this test method and were therefore are not included in Tables 7 through 9 in this test method.

TABLE X1.1 Regression Equations for Bias and Precision, µg/L

Element	Applicable Range	Bias	Overall Precision	Single Analyst Precision
		Reagent Water		
Aluminum	(8.00–200)	$X = 0.992C + 1.19$	$S_t = 0.056X + 2.59$	$S_o = 0.042X + 1.27$
		Drinking Water		
Aluminum	(8.00–200)	$X = 0.954C + 2.38$	$S_t = 7.70$	$S_o = 0.013X + 6.17$
Manganese	(8.00–200)	$X = 0.989C + 0.10$	$S_t = 0.047X + 0.29$	$S_o = 0.021X + 0.40$
Silver	(0.80–200)	$X = 0.888C + 0.09$	$S_t = 0.186X + 0.17$	$S_o = 0.164X + 0.18$
Zinc	(8.00–200)	$X = 0.943C + 2.54$	$S_t = 0.048X + 5.27$	$S_o = 0.004X + 5.66$
		Ground Water		
Antimony	(2.80–100)	$X = 1.003C + 0.01$	$S_t = 0.059X + 0.04$	$S_o = 0.058X + 0.02$
Manganese	(8.00–200)	$X = 0.954C - 0.16$	$S_t = 0.103X + 0.14$	$S_o = 0.025X + 0.09$
Molybdenum	(2.80–100)	$X = 1.032C - 0.09$	$S_t = 0.55X + 0.43$	$S_o = 0.042X + 0.27$
Silver	(0.80–200)	$X = 0.858C - 0.00$	$S_t = 0.169X + 0.14$	$S_o = 0.120X - 0.01$

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Standard Test Method for
Gross Calorific Value of Coal and Coke[1]

This standard is issued under the fixed designation D 5865; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method pertains to the determination of the gross calorific value of coal and coke by either an isoperibol or adiabatic bomb calorimeter.

1.2 The values stated in SI units are regarded as the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

D 121 Terminology of Coal and Coke[2]
D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis[2]
D 388 Classification of Coals by Rank[2]
D 1193 Specification for Reagent Water[3]
D 2013 Method of Preparing Coal Samples for Analysis[2]
D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke[2]
D 3177 Test Method for Total Sulfur in the Analysis Sample of Coal and Coke[2]
D 3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases[2]
D 4239 Test Method for Sulfur in the Analysis Sample of Coal and Coke Using High Temperature Tube Furnace Combustion Methods[2]
D 5142 Test Methods for the Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures[2]
E 1 Specification for ASTM Thermometers[4]
E 144 Practice for Safe Use of Oxygen Combustion Bombs[5]
E 178 Practice for Dealing with Outlying Observations[6]

3. Terminology

3.1 *Definitions:*

3.1.1 *calorific value*—the heat produced by combustion of a unit quantity of a substance under specified conditions.

3.1.2 *calorimeter*—a device for measuring calorific value consisting of a bomb, its contents, a vessel for holding the bomb, temperature measuring devices, ignition leads, water, stirrer, and a jacket maintained at specified temperature conditions.

3.1.3 *adiabatic calorimeter*—a calorimeter which has a jacket temperature adjusted to follow the calorimeter temperature so as to maintain zero thermal head.

3.1.4 *isoperibol calorimeter*—a calorimeter which has a jacket of uniform and constant temperature.

3.1.5 *gross calorific value (gross heat of combustion at constant volume),* Q_v (gross)—the heat produced by complete combustion of a substance at constant volume with all water formed condensed to a liquid.

3.1.6 *heat of formation*—the change in heat content resulting from the formation of 1 mole of a substance from its elements at constant pressure.

3.1.7 *net calorific value (net heat of combustion at constant pressure),* Q_p (net)—the heat produced by combustion of a substance at a constant pressure of 0.1 MPa (1 atm), with any water formed remaining as vapor.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *corrected temperature rise*—the calorimeter temperature change caused by the process that occurs inside the bomb corrected for various effects.

3.2.2 *heat capacity*—the energy required to raise the temperature of the calorimeter one arbitrary unit.

NOTE 1—The heat capacity can also be referred to as the energy equivalent or water equivalent of the calorimeter.

4. Summary of Test Method

4.1 The heat capacity of the calorimeter is determined by burning a specified mass of benzoic acid in oxygen. A comparable amount of the analysis sample is burned under the same conditions in the calorimeter. The calorific value of the analysis sample is computed by multiplying the corrected temperature rise, adjusted for extraneous heat effects, by the

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[1] This document is under the jurisdiction of ASTM Committee D-5 on Coal and Coke and is the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

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[2] *Annual Book of ASTM Standards*, Vol 05.05.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] *Annual Book of ASTM Standards*, Vol 14.04.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

heat capacity and dividing by the mass of the sample.

5. Significance and Use

5.1 The gross calorific value can be used to compute the total calorific content of the quantity of coal or coke represented by the sample for payment purposes.

5.2 The gross calorific value can be used for computing the calorific value versus sulfur content to determine whether the coal meets regulatory requirements for industrial fuels.

5.3 The gross calorific value can be used to evaluate the effectiveness of beneficiation processes.

5.4 The gross calorific value can be required to classify coals according to Classification D 388.

6. Apparatus and Facilities

6.1 *Test Area*—An area free from drafts, shielded from direct sunlight and other radiation sources. Thermostatic control of room temperature and controlled relative humidity are desirable.

6.2 *Combustion Bomb*—Constructed of materials that are not affected by the combustion process or the products formed to introduce measurable heat input or alteration of end products. The bomb shall be designed so that all liquid combustion products can be completely recovered by washing the inner surfaces. There shall be no gas leakage. The bomb shall be capable of withstanding a hydrostatic pressure test to 20 MPa (3000 psig) at room temperature without stressing any part beyond its specified elastic limit.

6.3 *Balance*—A laboratory balance capable of weighing the analysis sample to the nearest 0.0001 g. The balance shall be checked weekly, at a minimum, for accuracy.

6.4 *Calorimeter Vessel*—Made of metal with a tarnish-resistant coating, with all outer surfaces highly polished. Its size shall be such that the bomb is completely immersed in water during a determination. A stirrer shall be provided for uniform mixing of the water. The immersed portion of the stirrer shall be accessible to the outside through a coupler of low thermal conductivity. The stirrer speed shall remain constant to minimize any temperature variations due to stirring. Continuous stirring for 10 min shall not raise the calorimeter temperature more than 0.01°C when starting with identical temperatures in the calorimeter, test area and jacket. For calorimeters having a bucket it can be a separate component or integral component of the bomb. The vessel shall be of such construction that the environment of the calorimeter's entire outer boundaries can be maintained at a uniform temperature.

6.5 *Jacket*—A container with the inner perimeter maintained at constant temperature ±0.1°C (isoperibol) or at the same temperature ±0.1°C as the calorimeter vessel (adiabatic) during the test. To minimize convection, the sides, top and bottom of the calorimeter vessel shall not be more than 10 mm from the inner surface of the jacket. Mechanical supports for the calorimeter vessel shall be of low thermal conductivity.

6.6 *Thermometers*:

6.6.1 *Automated Calorimeters*—Platinum resistance or linear thermistor thermometers shall be capable of measuring to the nearest 0.0001°C. Thermometer calibration shall be traceable to a recognized certifying agency.

6.6.2 *Manual Calorimeters*:

6.6.2.1 *Platinum resistance* or *linear thermistor thermometers*, shall be capable of measuring to the nearest 0.0001°C. Thermometer calibration shall be traceable to a recognized certifying agency.

6.6.2.2 *Liquid-in-Glass Thermometers*—Conforming to the requirements for thermometers 56C, 116C, or 117C as prescribed in Specification E 1. Thermometers 56C shall be calibrated at intervals no larger than 2.0°C over the entire graduated scale. The maximum difference in correction between any two calibration points shall be no more than 0.02°C. Thermometers 116C and 117C shall be calibrated at intervals no larger than 0.5°C over the entire graduated scale. The maximum difference in correction between any two calibration points shall not be more than 0.02°C.

6.6.2.3 *Beckman Differential Thermometer*—(Glass enclosed scale, adjustable), having a range of approximately 6°C in 0.01°C subdivisions reading upward and conforming to the requirements for Thermometer 115C, as prescribed in Specification E 1. The thermometer shall be calibrated at intervals no larger than 1°C over the entire graduated scale. The maximum difference in the correction between any two calibration points shall be less than 0.02°C.

6.6.2.4 *Thermometer Accessories*—A magnifier is required for reading liquid-in-glass thermometers to one tenth of the smallest scale division. The magnifier shall have a lens and holder designed so as to minimize errors as a result of parallax.

6.7 *Sample Holder*—An open crucible of platinum, quartz, or base metal alloy. Before use in the calorimeter, heat treat base metal crucibles for a minimum of 4 h at 500°C to ensure the crucible surface is completely oxidized. Base metal alloy crucibles are acceptable, if after three preliminary firings, the weight does not change by more than 0.0001 g.

6.8 *Ignition Fuse*—Ignition fuse of 100-mm length and 0.16-mm (No. 34 B&S gage) diameter or smaller. Nickel-chromium alloy (Chromel C) alloy, cotton thread, or iron wire are acceptable. Platinum or palladium wire, 0.10-mm diameter (No. 38 B&S gage), can be used provided constant ignition energy is supplied. Use the same type and length (or mass) of ignition fuse for calorific value determinations as used for standardization.

6.9 *Ignition Circuit*—A 6- to 30-V alternating or direct current is required for ignition purposes. A step-down transformer connected to an alternating current circuit, capacitors, or batteries can be used. For manually operated calorimeters, the ignition circuit switch shall be of the momentary double-contact type, normally open except when held closed by the operator. An ammeter or pilot light can be used in the circuit to indicate when current is flowing.

6.10 *Controller*—For automated calorimeters, capable of charging the bomb; filling the calorimeter vessel; firing the ignition circuit; recording calorimeter temperatures before, during, and after the test; recording the balance weights; and carrying out all necessary corrections and calculations.

6.11 *Crucible Liner*—Quartz fiber or alundum for lining the crucible to promote complete combustion of samples that do not burn completely during the determination of the calorific value.

7. Reagents

7.1 *Reagent Water*—Conforming to conductivity requirements for Type II of Specification D 1193 for preparation of reagents and washing of the bomb interior.

7.2 *Purity of Reagents*—Use reagent grade chemicals conforming to the specification of the Committee on Analytical Reagents of the American Chemical Society in all tests.[7]

7.3 *Benzoic Acid—Standard* (C_6H_5COOH)—Pellets made from benzoic acid available from the National Institute of Standards and Technology (NIST) or benzoic acid calibrated against NIST standard material. The calorific value of benzoic acid, for use in the calibration calculations, shall be traceable to a recognized certificate value.

7.4 *Oxygen*—Manufactured from liquid air, guaranteed to be greater than 99.5 % pure, and free of combustible matter. Oxygen made by the electrolytic process contains small amounts of hydrogen rendering it unfit unless purified by passage over copper oxide at 500°C.

7.5 *Titration Indicator*—Methyl orange, methyl red, or methyl purple for indicating the end point when titrating the acid formed during combustion. The same indicator shall be used for both calibration and calorific value determinations.

7.6 *Standard Solution*—Sodium carbonate (Na_2CO_3) or other suitable standard solution. Dissolve 3.76 g of sodium carbonate, dried for 24 h at 105°C in water, and dilute to 1 L. One millilitre of this solution is equivalent to 4.2 J (1.0 calorie) in the acid titration.

8. Hazards

8.1 The following precautions are recommended for safe calorimeter operation. Additional precautions are noted in Practice E 144. Also consult the calorimeter equipment manufacturer's installation and operating instructions before using the calorimeter.

8.1.1 The mass of sample and any combustion aid as well as the pressure of the oxygen admitted to the bomb shall not exceed the bomb manufacturer's specifications.

8.1.2 Inspect the bomb parts carefully after each use. Replace cracked or significantly worn parts. Replace O-rings and valve seats in accordance with manufacturer's instruction. For more details, consult the manufacturer.

8.1.3 Equip the oxygen supply cylinder with an approved type of safety device, such as a relief valve, in addition to the needle valve and pressure gage used in regulating the oxygen feed to the bomb. Valves, gages, and gaskets shall meet industry safety codes. Suitable reducing valves and adapters for 3- to 4-MPa (300- to 500-psig) discharge pressure can be obtained from commercial sources of compressed gas equipment. Check the pressure gage annually for accuracy or after any accidental over pressures that reach maximum gage pressure.

8.1.4 During ignition of a sample, the operator shall not extend any portion of the body over the calorimeter.

8.1.5 Do not fire the bomb if the bomb has been dropped or turned over after loading.

8.1.6 Do not fire the bomb if there is evidence of gas leakage when the bomb is submerged in the calorimeter vessel.

8.1.7 For manually operated calorimeters, the ignition switch shall be depressed only long enough to fire the charge.

9. Sample

9.1 The analysis sample is the material pulverized to pass 250-µm (No. 60) sieve, prepared in accordance with either Practice D 346 for coke or Method D 2013 for coal.

10. Determination of the Heat Capacity of the Calorimeter

10.1 *Sample*—Weigh 0.8 to 1.2 g of benzoic acid into a sample holder. Record sample weight to the nearest 0.0001 g.

10.2 *Preparation of Bomb*:

10.2.1 Rinse the bomb with water to wet internal seals and surface areas of the bomb or precondition the calorimeter according to the manufacturer's instructions. Add 1.0 mL of water to the bomb before assembly.

10.2.2 Connect a measured fuse in accordance with manufacturer's guidelines.

10.2.3 Assemble the bomb. Admit oxygen to the bomb to a consistent pressure of between 2 and 3 MPa (20 and 30 atm). The same pressure is used for each heat capacity run. Control oxygen flow to the bomb so as not to blow material from the sample holder. If the pressure exceeds the specified pressure, detach the filling connection and exhaust the bomb. Discard the sample.

10.3 *Preparation of Calorimeter*:

10.3.1 Fill the calorimeter vessel with water at a temperature not more than 2°C below room temperature and place the assembled bomb in the calorimeter. Check that no oxygen bubbles are leaking from the bomb. If there is evidence of leakage, remove and exhaust the bomb. Discard the sample.

10.3.2 The mass of water used for each test run shall be M ± 0.5 g where M is a fixed mass of water. Devices used to supply the required mass of water on a volumetric basis shall be adjusted when necessary to compensate for change in the density of water with temperature.

10.3.3 With the calorimeter vessel positioned in the jacket start the stirrers.

10.4 *Temperature Observations Automated Calorimeters*:

10.4.1 *Stabilization*—The calorimeter vessel's temperature shall remain stable over a period of 30 s before firing. The stability shall be ±0.001°C for an adiabatic calorimeters and ±0.001°C/s or less for an isoperibol calorimeter.

10.4.2 *Extrapolation Method*—Fire the charge, record the temperature rise. The test can be terminated when the observed thermal curve matches a thermal curve which allows extrapolation to a final temperature with a maximum uncertainty of ±0.002°C.

10.4.3 *Full Development Method*—Fire the charge and record the temperature rise until the temperature has stabilized for a period of 30 s in accordance with the stability requirements specified in 10.4.1.

10.5 *Temperature Observations Manual Calorimeters*:

10.5.1 When using ASTM Thermometers 56C, estimate all

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[7] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

readings to the nearest 0.002°C. When using ASTM Thermometers 115C, 116C, or 117C, estimate readings to 0.001°C and 25-Ω resistance thermometer readings to the nearest 0.0001 Ω. Tap or vibrate mercury thermometers just before reading to avoid errors caused by mercury sticking to the walls of the capillary.

10.5.2 Allow 5 min for the temperature of the calorimeter vessel to stabilize. Adjust the jacket temperature to match the calorimeter vessel temperature within 0.01°C and maintain for 3 min.

10.5.3 Fire the charge. Record the time as *a* and the temperature as t_a.

10.5.4 For adiabatic calorimeters adjust the jacket temperature to match that of the calorimeter vessel temperature during the period of the rise. Keep the two temperatures as equal as possible during the period of rapid rise. Adjust to within 0.01°C when approaching the final stabilization temperature. Record subsequent readings at intervals no greater than 1 min until three successive readings do not differ by more than ±0.001°C. Record the first reading after the rate of change has stabilized as the final temperature t_c and the time of this reading as *c*. For isoperibol calorimeters, when approaching the final stabilization temperature, record readings until three successive readings do not differ by more than 0.001°C per min. Record the first reading after the rate of change has stabilized as the final temperature as t_c and the time of this reading as *c*.

10.5.5 Open the calorimeter and remove the bomb. Release the pressure at a uniform rate such that the operation will not be less than 1 min. Open the bomb and examine the bomb interior. Discard the test if unburned sample or sooty deposits are found.

10.6 *Thermochemical Corrections (see* Appendix X1*)*:

10.6.1 *Acid Correction (see* X1.1*)*—One may use either the titration (10.6.1.1) or calculated titration (10.6.1.2) procedure for coal and coke samples.

10.6.1.1 *Titration Method*—Wash the interior of the bomb with distilled water containing the titration indicator (see 7.5) until the washings are free of acid and combine with the rinse of the capsule. Titrate the washings with the standard solutions (see 7.6) using a titration indicator, or a pH or millivolt meter. The number of millilitres of standard Na_2CO_3 used in the titration shall be taken as *e*1.

10.6.1.2 *Calculated Titration Method*—Each calorimeter system shall be tested at several energy levels with benzoic acid pellets weighing 0.8, 1.0, and 1.2 g. This range corresponds to the optimum energy levels of 5000 through 8000 calories. Two runs shall be made at each weight. Plot millilitres of titrant (*y*) versus temperature rise, degree C (*x*), for each calibration and use linear regression to determine the formula for the line $y = m(x) + b$ (see Fig. 1). The resulting formula for a line is the equation for determining the calculated millilitres of titrant (*e*1). The calculated titrant $= m(x) + b$ where *m* and *b* have been determined by linear regression. The temperature rise (*x*) for each test shall be plotted on the graph to determine the calculated acid correction (*e*1) or determined from the equation $y = mx + b$.

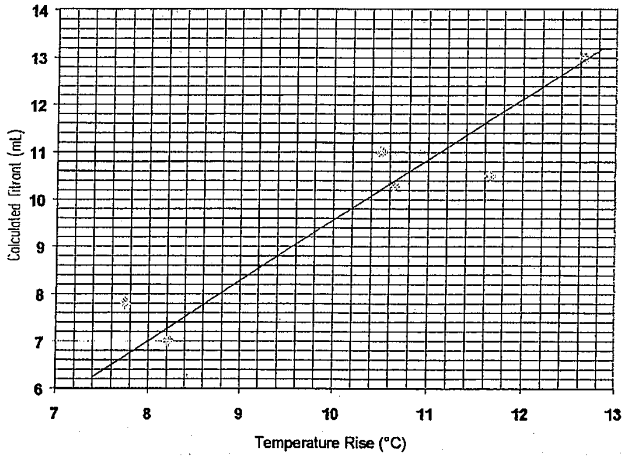

FIG. 1 Titration Versus Temperature Rise

Example:

Sample wt., g	Measured Titration (*y*)	Temperature Rise (*x*)
0.7643	7.8	7.7443
0.8104	7.0	8.2188
1.0392	11.0	10.5114
1.0506	10.3	10.6420
1.1539	10.5	11.6584
1.2562	13.0	12.6491

$$y = m(x) + b \tag{1}$$

Using regression analysis, the above data yield the following data: slope = 1.0826, intercept = −1.1496, and the equiation for the millilitres of titrant $= y = 1.0826x - 1.1496$.

With any given temperature rise (*x*), the value *y* (*e*1) may be determined.

NOTE 2—*m* above represents the slope of the line, whereas in other references in this method *m* represents mass.

NOTE 3—Regression analysis to determine the equation for the millilitres of titrant (*e*1) is to be done without forcing the data through zero.

10.6.2 *Fuse Correction (see* X1.3*)*—Determine the fuse correction using one of the two alternatives:

10.6.2.1 Measure the combined pieces of unburned ignition fuse and subtract from the original length to determine the fuse consumed in firing according to Eq 2.

$$e2 = K_l \times l \tag{2}$$

where:

*e*2 = the correction for the heat of combustion of the firing fuse,
l = the length of fuse consumed during combustion,
K_l = 0.96 J/mm (0.23 cal/mm) for No. 34 B&S gage Chromel C,
K_l = 1.13 J/mm (0.27 cal/mm) for No. 34 B&S gage iron wire, and
K_l = 0.00 J/mm for platinum or palladium wire provided the ignition energy is constant.

or;

10.6.2.2 Weigh the combined pieces of unburned fuse and subtract from the original weight to determine the weight in milligrams of the fuse consumed in firing (m). Remove any ball of oxidized metal from the ends before weighing.

$$e2 = K_m \times m \quad (3)$$

where:

$e2$ = the correction for the heat of combustion of the firing fuse,

m = the weight in mg of fuse consumed during combustion,

K_m = 5.9 J/mg (1.4 cal/mg) for No. 34 B&S gage Chromel C,

K_m = 7.5 J/mg (1.8 cal/mg) for No. 34 B&S gage iron wire, and

K_m = 0.00 J/mg for platinum or palladium wire provided the ignition energy is constant.

When cotton thread is used, employ the correction in J recommended by the instrument manufacturer.

10.7 *Calculation of the Corrected Temperature Rise*—Compute the corrected temperature rise, t, as follows:

$$t = t_c - t_a + C_e + C_r + C_s \quad (4)$$

where:

t = corrected temperature rise, °C;

t_a = initial temperature reading at time of firing;

t_c = final temperature reading;

C_e = thermometer, emergent stem correction (see Eq A1.4);

C_r = radiation correction (see Eq A1.2); and

C_s = thermometer setting correction (see Eq A1.3).

10.7.1 The temperature rise in isoperibol calorimeters require a radiation correction.

10.7.2 Beckman differential thermometers require a setting correction and an emergent stem correction.

10.7.3 Solid-stem ASTM Thermometers 56C do not require emergent stem corrections if all tests are performed within the same 5.5°C interval. If the operating temperature range is beyond this limit, a differential emergent stem correction shall be applied.

10.8 *Calculation of the Heat Capacity*—Calculate the heat capacity (E) of the calorimeter using the following equation:

$$E = [(H_c \times m) + e1 + e2]/t \quad (5)$$

where:

E = the calorimeter heat capacity, J/°C;

H_c = heat of combustion of benzoic acid, as stated in the certificate, J/g;

m = mass of benzoic acid, g;

$e1$ = acid correction from 10.6.1 from either the titration method (10.6.1.1) or the calculated titration (10.6.1.2);

$e2$ = fuse correction from 10.6.2, J; and

t = corrected temperature rise from 10.7,°C.

10.8.1 Using the procedures described in 10.1-10.8 complete a total of ten acceptable test runs. An individual test shall be rejected only if there is evidence of incomplete combustion.

10.8.2 The relative standard deviation of the heat capacity of ten acceptable test runs shall be 0.15 % or less of the average energy equivalent. If after considering the possibility of outliers using criterion established in Practice E 178, this limit is not met, one should review operation of the calorimeter for any assignable cause which should be corrected before repeating the series. Table 1 summarizes a series of heat capacity runs. This table would be applicable regardless of the unit of measure for the heat capacity.

11. Heat Capacity Checks

11.1 The heat capacity value shall be checked a minimum of once a month, after changing any part of the calorimeter, or after changing the oxygen supply. Two procedures are available for heat capacity check: Standard Method and Rolling Average Method.

NOTE 4—Although it is only required to check the heat capacity once a month, this may be inadequate. A more frequent check of heat capacity values is recommended for laboratories making a large number of tests on a daily basis. The frequency of the heat capacity check should be determined to minimize the number of tests that would be affected by an undetected shift in the heat capacity values.

11.2 *Standard Method*:

11.2.1 A single new heat capacity test value shall not differ from the existing heat capacity value by more than ±0.17 %. If this requirement is met, the existing heat capacity value is acceptable. For example: existing heat capacity value is 2402 cal/°C. 2402 cal/°C × 0.0017 = 4.1 cal/°C. If single test value is within 4.1 cal/°C of the 2402 cal/°C value, then the existing heat capacity value is still acceptable.

11.2.2 If the requirements given in 11.2.1 are not met, an additional heat capacity test shall be run. The difference between the two new heat capacity values shall not exceed 0.21 % of the existing heat capacity value. The average of the two new heat capacity values shall not differ from the existing heat capacity value by more than ±0.13 %. If this requirement is met, the existing heat capacity value is acceptable.

11.2.3 If the requirements given in 11.2.2 are not met, two more heat capacity tests shall be run. The range of the four new test values shall not exceed 0.33 % of the existing heat capacity value. The average of the four new heat capacity values shall not differ from the existing heat capacity value by more than ±0.08 %. If this requirement is met, the existing heat capacity value is acceptable.

11.2.4 If the requirements given in 11.2.3 are not met, a fifth and sixth heat capacity test shall be run. The range of the six new test values shall not exceed 0.42 % of the existing heat capacity value. The average of the six new heat capacity values

TABLE 1 Heat Capacity Runs

Note—Variance = s^2 = {Sum Column C − [(Sum Column B)^2/10]}/9 = 89.51.
Standard Deviation = $s = \sqrt{s^2}$ = 9.46.
Relative Standard Deviation = (s/Average) × 100 = 0.09 %.

Run Number	Column A Heat Capacity, J/°C	Column B Difference From Average	Column C (Column B)^2
1	10 257.7	+4.2	17.6
2	10 249.3	−4.2	17.6
3	10 270.2	+16.7	278.9
4	10 253.5	0.0	0
5	10 245.1	−8.4	70.6
6	10 249.3	−4.2	17.6
7	10 240.9	−12.6	158.8
8	10 266.0	+12.5	156.3
9	10 257.7	+4.2	17.6
10	10 245.1	−8.4	70.6
SUM		−0.2	805.6
AVERAGE	E = 10 253.5		

shall not differ from the existing heat capacity value by more than ±0.08 %. If these requirements are met, do not change the existing heat capacity value.

11.2.5 If the requirements given in 11.2.4 are not met, four more heat capacity tests shall be run. The range of the ten new test values shall not exceed 0.50 % of the existing heat capacity value. The average of the ten new heat capacity values shall not differ from the existing heat capacity value by more than ±0.04 %. If this requirement is met, the existing heat capacity value is acceptable.

11.2.6 If requirements given in 11.2.5 are not met, the average value from the ten new test values shall be used to determine a new heat capacity value provided the relative standard deviation of the ten values does not exceed 0.15 %.

11.2.7 The summary of the numerical requirements at each step in checking the heat capacity is given in Table 2.

11.3 *Rolling Average Method*:

11.3.1 A single new heat capacity value shall not differ from the existing heat capacity value by more than ±0.17 %.

11.3.2 Values that serve to confirm existing heat capacity values will be included with the original 10 calibration tests until a total of 20 tests are made. These tests will comprise a database for calculating the mean heat capacity value provided the relative standard deviation of the 20 values does not exceed 0.15 %. Any new calibration check beyond the 20 tests will replace the oldest value in the heat capacity database of 20 tests. A maximum relative standard deviation of 0.15 % shall be maintained for the heat capacity database of 20 tests.

11.3.3 When using a rolling average, the heat capacity data must be continually evaluated for four indications of out-of-control conditions: first, seven consecutive results are higher than the mean; second, seven consecutive results are lower than the mean; third, seven consecutive results are continually increasing; and fourth, seven consecutive results are continually decreasing.

11.3.4 Out-of-control trends indicate that the calorimeter operation is suspect and causes should be identified. Whether or not causes are identified, the calorimeter should be recalibrated according to the procedure in Section 10.

12. Procedure for Coal and Coke Samples

12.1 Weigh 0.8 to 1.2 g of sample into a sample holder. Record the weight to the nearest 0.0001 g (see 12.6.3).

12.2 Follow the procedures as described in 10.2-10.5 for determination of heat capacity. The starting temperature for determinations shall be within ±0.5°C of that used in the determination of the heat capacity.

TABLE 2 Numerical Requirements

Number of Check Runs	Maximum Range $((E_{nmax} - E_{nmin})/E_e) \times 100$	Maximum Difference $((E_{nav} - E_e)/E_e) \times 100$
1	...	±0.17
2	0.21	±0.13
4	0.33	±0.08
6	0.42	±0.08
10	0.50	±0.04

E_e is the existing heat capacity value.
E_{nmin} is the minimum reading in group of heat capacity check runs.
E_{nmax} is the maximum reading in a group of heat capacity check runs.
E_{nav} is the average of the group of heat capacity check runs.

12.3 Carry out a moisture determination in accordance with Test Method D 3173 or Test Methods D 5142 on a separate portion of the analysis sample preferably on the same day but not more than 24 h apart from the calorific value determination so that reliable corrections to other bases can be made.

12.4 Conduct the sulfur analysis in accordance with Test Methods D 3177 or D 4239. From the weight % sulfur, calculate the sulfur corrections (see X1.2):

$$e3 = 55.2\ \text{J/g} \times S \times m\ (13.18\ \text{cal/g} \times S \times m) \quad (6)$$

where:

$e3$ = a correction for the difference between the heat of formation of H_2SO_4 from SO_2 with respect to the formation of HNO_3, J;
S = wt % sulfur in the sample; and
m = mass of sample from 12.1, g.

12.4.1 When titration method is used (see 10.6.1.1), the sulfur correction is

$$e3 = 55.18\ \text{J/g} \times S \times m \text{ or } (13.18\ \text{cal/g} \times S \times m) \quad (7)$$

12.4.2 When the calculated titration method is used (see 10.6.1.2), the sulfur correction is

$$e3 = 94.51\ \text{J/g} \times S \times m \text{ or } (22.57\ \text{cal/g} \times S \times m) \quad (8)$$

12.5 For eight mesh samples, analyze coals susceptible to oxidation within 24 h of preparation.

12.6 Coal or coke that do not burn completely can be treated as follows:

12.6.1 Use a crucible liner of the type recommended in 6.11.

12.6.2 Use a combustion aid such as benzoic acid, ethylene glycol, mineral oil or a gelatin capsule. A minimum of 0.4 g of combustion aid shall be used. Record the weight to the nearest 0.0001 g. Calculate the correction for use of a combustion aid using the following:

$$e4 = Ha \times ma \quad (9)$$

where:

$e4$ = correction for use of a combustion aid,
Ha = heat of combustion of the combustion aid J/g (cal/g), and
ma = mass of combustion aid, g.

12.6.3 Vary the mass of the sample to obtain good ignition and so that the total heat generated is the same as the heat generated during calibration.

13. Calculations

13.1 *Gross Calorific Value*—Calculate the gross calorific value Q_{vad} (gross) using the following equation:

$$Q_{vad}\ (\text{gross}) = [(tE_e) - e1 - e2 - e3 - e4]/m \quad (10)$$

where:

Q_{vad} (gross) = gross calorific value at constant volume as determined, J/g (cal/g);
E_e = the heat capacity of the calorimeter, J/°C (cal/°C);
t = corrected temperature rise according to 10.7, °C;
$e1$ = acid correction according to 10.6.1, J;
$e2$ = fuse correction according to 10.6.2, J;

$e3$ = sulfur correction determined according to 12.4, J;
$e4$ = combustion aid correction determined according to 12.7, J; and
m = mass of the sample, g.

13.1.1 See Appendix X1.2.3 for an example calculation.

13.2 *Net Calorific Value*—Calculate the net calorific value Q_p (net) as follows:

$$Q_p \text{ (net)} = Q_{var} \text{ (gross)} - 215.5 \text{ J/g} \times H_{ar} \tag{11}$$

or:

$$(Q_p \text{ (net)}_{ar} = Q_{var} \text{ (gross)} - 92.67 \text{ Btu/lb} \times H_{ar}) \tag{12}$$

where:
Q_p (net) = net calorific value, at constant pressure;
Q_{var} (gross) = gross calorific value, at constant volume, as-received basis; and
H_{ar} = total hydrogen, %, as-received basis, where hydrogen includes hydrogen in the sample moisture.

Example:

Calorific value as determined Q_{vad} (gross) = 31 420 J/g
Moisture, as determined M_{ad} = 2.13 wt %
Moisture, as received M_{ar} = 8.00 wt %
Hydrogen, as determined H_{ad} = 5.00 wt %

Q_{var} (gross) = Q_{vad} (gross) × $[(100 - M_{ar})/(100 - M_{ad})]$ = 31 420 J/g × [(100 − 8.00)/(100 − 2.13)] = 29 535 J/g

$H_{ar} = [(H_{ad} - 0.1119 \times M_{ad}) \times \{(100 - M_{ar})/(100 - M_{ad})\}] + 0.1119\, M_{ar} = [(5.00 - 0.1119 \times 2.13) \times \{(100 - 8.00)/(100 - 2.13)\}] + 0.1119 \times 8.0 = 5.37$

Q_p (net) = 29 535 J/g − (215.5 × 5.37) = 29 535 J/g − 1153 J/g = 28 388 J/g

14. Report

14.1 Report the calorific value as Q_{vad} (gross) along with the moisture of the sample as determined M_{ad} from 12.3.

14.2 The results of the calorific value can be reported in any of a number of bases differing in the manner the moisture is treated. Procedures for converting the value obtained on an analysis sample to other bases are described in Practice D 3180.

15. Precision and Bias

15.1 *Manual Calorimeters*:

15.1.1 *Repeatability*—The difference in absolute value between two test results calculated to a dry basis (Practice D 3180) performed on two separate test portions of the same analysis sample of 250-µm (No. 60) coal in the same laboratory, by the same operator, using the same equipment with the same heat capacity value shall not exceed the repeatability interval *I(r)* of 115 J/g (50 Btu/lb) more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the repeatability interval, there is reason to question one or both of the test results.

15.1.2 *Reproducibility*—The difference in absolute value between test results calculated to a dry basis (Practice D 3180) performed in different laboratories on representative analysis samples of 250-µm (No. 60) coal shall not exceed the reproducibility interval *I(r)* of 250 J/g (100 Btu/lb) more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the reproducibility interval there is reason to question one or both of the test results.

15.1.3 *Bias*—Bias in the determination of the gross calorific value is eliminated provided samples are treated identically to the benzoic acid used in the determination of the calorimeter heat capacity.

15.2 *Automated Calorimeters*:

15.2.1 *Repeatability*—The repeatability has not been determined.

15.2.2 *Reproducibility*—The reproducibility has not been determined.

15.2.3 *Bias*—Bias in the determination of the gross calorific value is eliminated provided samples are treated identically to the benzoic acid used in the determination of the calorimeter heat capacity.

16. Keywords

16.1 adiabatic calorimeter; bomb calorimeters; calorific value; calorimeter; coal; coke; isoperibol bomb calorimeter

ANNEX

(Mandatory Information)

A1. THERMOMETRIC CORRECTIONS

A1.1 *Thermometer Corrections*—The following corrections shall be made:

A1.1.1 *Calibration Correction*, shall be made in accordance with the calibration certificate furnished by the calibration authority.

A1.1.2 *Radiation Corrections*—Radiation corrections are required to calculate heat loss or gain to the isoperibol water jacket. They are based on the Dickinson formula,[8] the Regnault-Pfaundler formula,[9] or the U.S. Bureau of Mines method.[10] The same method of determining the radiation correction shall be used consistently in the determination of

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[8] Dickinson, H. C., *Bulletin*, U.S. Bureau of Standards, Vol. 11, 1951, p. 189.

[9] Pfaundler, L., *Annalen der Physik* (Leipzig), ANPYA, Vol. 129, 1966, p. 102.

[10] "Methods of Analyzing & Testing Coal and Coke" U.S. Bureau of Mines *Bulletin* 638, XMBUA, 1967, pp. 16–17.

heat capacity and sample measurements.

A1.1.2.1 *Dickinson Formula*:

$$C_r = -r1 \times (b - a) - r2 \times (c - b) \quad \text{(A1.1)}$$

where:

C_r = radiation correction;
$r1$ = rate of rise in temperature per minute in the preliminary period;
$r2$ = rate of rise of temperature per minute in the final period (if temperature is falling, $r2$ is negative;
ta = firing temperature;
tc = final temperature, being the first temperature after which the rate of change is constant;
a = time at temperature ta, min;
b = time at temperature $ta + 0.60\ (tc - ta)$, min; and
c = time at temperature tc, min.

A1.1.2.2 Regnault-Pfaundler Formula:

$$C_r = nr1 + kS \quad \text{(A1.2)}$$

where:

C_r = radiation correction,
n = number of minutes in the combustion period,

$$k = (r1 - r2) / (t'' - t'), \quad \text{(A1.3)}$$

$$S = tn - 1 + (1/2)(ti + tf)nt' \quad \text{(A1.4)}$$

t' = average temperature during the preliminary period,
t'' = average temperature during the final period,

$r1$, $r2$ see A1.1.2.1,

$t1, t2, \ldots tn$ = successive temperature recorded during the combustion period, at 1-min intervals, and

$$tn - 1 = \text{sum of } t1, t2, t3 \ldots tn - 1 \quad \text{(A1.5)}$$

A1.1.2.3 *Bureau of Mines Method*—A table of radiation corrections can be established so that only the initial and final readings are required to determine the calorific value of a fuel. This can be done by carrying out a series of tests using the procedure described in Section 10, using the following conditions. Regulate the amount of sample burned so that a series of determinations is made in which different temperature rises are obtained. For all determinations, keep the water jacket temperature constant, fire the bomb at the same initial temperature, and have the same time, $c - a$, elapse (±2 s) between the initial and final readings. Determine the radiation corrections for each of the series of temperature rises using the Dickinson method (see A1.1.2.1), or the Regnault-Pfaundler method (see A1.1.2.2). These corrections are constant for a given temperature rise. From the series of readings, a table or graph is plotted to show radiation correction versus temperature rise. Once the table or graph is established, the radiation corrections can be obtained from it until there is a major change in the equipment.

A1.1.3 *Setting Correction*

This is necessary for the Beckman thermometer. It shall be made in accordance with the directions furnished by the calibration authority.

A1.1.4 *Differential Emergent Stem Correction*

The calculation of differential stem correction depends upon the way the thermometer was calibrated and how it is used. Two conditions are possible.

A1.1.4.1 *Thermometers Calibrated in Total Immersion and Used in Partial Immersion*

This emergent stem correction is made as follows:

$$Ce = K\,(tf - ti)(tf + ti - L - T) \quad \text{(A1.6)}$$

where:

Ce = emergent stem correction,
K = 0.000 16 for thermometers calibrated in °C,
L = scale reading to which the thermometer was immersed,
T = mean temperature of emergent stem,
ti = initial temperature reading, and
tf = final temperature reading.

Example:

A thermometer was immersed to 16°C; its initial reading, *ti*, was 24.127°C; its final reading, *tf*, was 27.876; the mean temperature of the emergent stem, *T*, was 26°C.

$$Ce = 0.000\,16 \times (28 - 24) \times (28 + 24 - 16 - 26) = 0.0064\text{°C} \quad \text{(A1.7)}$$

A1.1.4.2 *Thermometers Calibrated and Used in Partial Immersion, But at a Different Temperature Than the Calibrated Temperature:*

$$Ce = K\,(tf - ti)(tc - to) \quad \text{(A1.8)}$$

where:

Ce = emergent stem correction,
K = 0.000 16 for thermometers calibrated in °C,
ti = initial temperature reading,
tf = final temperature reading,
to = observed stem temperature, and
tc = stem temperature at which the thermometer was calibrated.

Example:

A thermometer has an initial reading, *ti*, 27°C; a final reading, *tf*, 30°C; the observed stem temperature, *to*, 28°C; and the calibration temperature, *tc*, 22°C.

$$Ce = 0.000\,16 \times (30 - 27) \times (28 - 22) = 0.003\text{°C} \quad \text{(A1.9)}$$

APPENDIX

(Nonmandatory Information)

X1. THERMOCHEMICAL CORRECTIONS

X1.1 *Energy of Formation of Nitric Acid* (*HNO_3*):

X1.1.1 A correction, *e*1, (10.6.1) is applied for the formation of nitric acid.

X1.1.2 (1) HNO_3 is formed in the calorimeter by the following reaction:

$$1/2\ N_2\,(g) + 5/4\,O_2\,(g) + 1/2\,H_2O\,(l) = HNO_3\,(\text{in } 500 \text{ mol } H_2O) \tag{X1.1}$$

X1.1.3 (2) the energy of formation of HNO_3 in approximately 500 mol of water under bomb conditions is minus 59.0 kJ/mol (14.09 Kcal/mole).[11]

X1.1.4 Normal convention assigns a negative value for a heat of formation that is exothermic. By definition, heat released from combustion processes are expressed as positive values. Hence, the negative factors developed for nitric and sulfuric acid corrections are expressed as positive values in the calculations.

X1.1.5 A convenient concentration of Na_2CO_3 is 3.76-g Na_2CO_3/L which gives *e*1 = *V* where *V* is the volume of Na_2CO_3 in millilitres. When H_2SO_4 is produced during the combustion of coal or coke, a part of the correction for H_2SO_4 is present in the *e*1 correction. The remainder is in the *e*3 correction (see X1.2).

X1.2 *Energy of Formation of Sulfuric Acid* (*H_2SO_4*)—By definition (see Terminology D 121), the gross calorific value is obtained when the product of the combustion of sulfur in the sample is SO_2(g). However, in actual bomb combustion processes, all the sulfur is found as H_2SO_4 in the bomb washings.

X1.2.1 A correction *e* 3 is applied for the sulfur that is converted to H_2SO_4. This correction is based upon the energy of formation of H_2SO_4 in solutions, such as will be present in the bomb at the end of a combustion from SO_2. This energy is taken as −303.0 kJ/mol.[12]

X1.2.2 When the bomb washings are titrated, a correction of 2 × 59.0 kJ/mole of sulfur is applied in the *e*1 correction so that the additional correction that is necessary is the difference in the heats of formation for nitric and sulfuric acid and this correction is − 303.0 − (−2 times 59.0) = −185 kJ/mol, or = −58 J/g (13.8 cal/g) of sulfur times the weight of sample in grams times percent sulfur in sample.

X1.2.3 If a 1-g sample is burned, the resulting H_2SO_4 condensed with water formed on the walls of the bomb will have a ratio of about 15 mol of water to 1 mol of H_2SO_4. For this concentration, the energy of the reaction under the conditions of the bomb process is −303 kJ/mole.

$$SO_2\,(g) + 1/2\,O_2\,(g) + H_2O\,(l) = H_2SO_4\,(\text{in } 15 \text{ mol of } H_2O) \tag{X1.2}$$

X1.2.4 The values above are based on a sample containing approximately 5 % sulfur and approximately 5 % hydrogen. The assumption is also made that the H_2SO_4 is dissolved entirely in the water condensed during combustion of the sample.[13]

X1.2.5 For different sample weights or sulfur content, or both, the resultant normality of acid formed can be different, and therefore, the normality of titrant must be adjusted accordingly. Basing the calculation upon a sample of comparatively large sulfur content reduces the possible overall errors, because, for small percentages of sulfur, the correction is smaller.

X1.3 *Fuse Correction*—The energy required to melt a platinum or palladium wire is constant for each experiment if the same amount of platinum or palladium wire is used. As the energy is small, its effect is essentially cancelled out in the relationship between the standardization experiments and the calorific value determinations, and it can be neglected.

X1.4 *Reporting Results in Other Units:*

X1.4.1 The gross calorific value can be expressed in joules per gram, calories per gram, or British thermal units per pound. The relationships between these units are given in the table below:

1 Btu = 1055.06 J
1 calorie = 4.1868 J
1 J/g = 0.430 Btu/lb
1 J/g = 0.239 cal/g
1 cal/g = 1.8 Btu/lb

X1.5 *Sample Calculations:*

X1.5.1 *Heat Capacity*:

$E = [(Hc \times m) + e1 + e2]/t$

$Hc = 26\,435$ J/g,

$m = 1.0047$ g,

$e1 = 43$-J acid correction,

$e2 = 55$-J fuse correction,

$t = 2.6006°C$,

$E = [(26\,435 \text{ J/g} \times 1.0047 \text{ g}) + 43 \text{ J} + 55 \text{ J}]/2.6006°C$, and

$E = 10\,250.4$ J/°C.

X1.6 *Heat of Combustion:*

$Q_{vad}\,(\text{gross}) = [(tE) - e1 - e2 - e3 - e4]/m$;

$E_e = 10\,250.4$ J/°C;

$t = 2.417°C$;

$e1 = 77$-J acid correction;

$e2 = 52$-J fuse correction;

$e3 = 58 \times 1.24\,\% \times 0.7423$ g, sulfur correction;

$e4 = 46\,025 \text{ J/g} \times 0.2043$ g, combustion aid correction;

$m = 0.7423$ g, mass of sample;

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[11] Mott, R. A., and Parker, C., "Studies in Bomb Calorimetry IV—Corrections," Fuel, FUELB, Vol. 34, 1955, p. 303–316.

[12] Calculated from data in National Bureau of Standards Circular 500.

[13] Mott, R. A., and Parker, C., "Studies in Bomb Calorimetry IX—Formation of Sulfuric Acid," *Fuel, Fuel B*, Vol. 37, 1958, p. 371.

Q_{vad} = [(10 250.4 J/°C × 2.417°C) − 77 J − 52 J − 53 J − 9403 J]/0.7423 g; and

Q_{vad} = 20 464 J/g.

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An American National Standard

Standard Practice for
Opacity Monitor Manufacturers to Certify Conformance with Design and Performance Specifications[1]

This standard is issued under the fixed designation D 6216; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the procedure for certifying continuous opacity monitors. It includes design and performance specifications, test procedures, and quality assurance requirements to ensure that continuous opacity monitors meet minimum design and calibration requirements, necessary in part, for accurate opacity monitoring measurements in regulatory environmental opacity monitoring applications subject to 10 % or higher opacity standards.

1.2 This practice applies specifically to the original manufacturer, or to those involved in the repair, remanufacture, or resale of opacity monitors.

1.3 Test procedures that specifically apply to the various equipment configurations of component equipment that comprise either a transmissometer, an opacity monitor, or complete opacity monitoring system are detailed in this practice.

1.4 The specifications and test procedures contained in this practice exceed that of the United States Environmental Protection Agency (USEPA). For each opacity monitor or monitoring system that the manufacturer demonstrates conformance to this practice, the manufacturer may issue a certificate that states that that opacity monitor or monitoring system conforms with all of the applicable design and performance requirements of 40 CFR 60, Appendix B, Performance Specification 1 except those for which tests are required after installation.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres[2]

2.2 *U.S. Environmental Protection Agency Document:*[3]

40 CFR 60 Appendix B, Performance Specification 1

2.3 *Other Documents:*

ISO/DIS 9004 Quality Management and Quality System Elements-Guidelines[4]

ANSI/NCSL Z 540-1-1994 Calibration Laboratories and Measuring Equipment - General Requirements[4]

NIST 260-116 - Filter calibration procedures[5]

3. Terminology

3.1 For terminology relevant to this practice, see Terminology D 1356.

3.2 *Definitions of Terms Specific to This Standard:*

Analyzer Equipment

3.2.1 *opacity, n*—measurement of the degree to which particulate emissions reduce (due to absorption, reflection, and scattering) the intensity of transmitted photopic light and obscure the view of an object through ambient air, an effluent gas stream, or an optical medium, of a given pathlength.

3.2.1.1 *Discussion*—Opacity (Op), expressed as a percent, is related to transmitted light, (T) through the equation:

$$Op = (1 - T)(100). \qquad (1)$$

3.2.2 *opacity monitor, n*—an instrument that continuously determines the opacity of emissions released to the atmosphere.

3.2.2.1 *Discussion*—An opacity monitor includes a transmissometer that determines the *in-situ* opacity, a means to correct opacity measurements to equivalent single-pass opacity values that would be observed at the pathlength of the emission outlet, and all other interface and peripheral equipment necessary for continuous operation.

3.2.2.2 *Discussion*—An opacity monitor may include the following: (*1*) sample interface equipment such as filters and purge air blowers to protect the instrument and minimize contamination of exposed optical surfaces, (*2*) shutters or other devices to provide protection during power outages or failure of the sample interface, and (*3*) a remote control unit to facilitate monitoring the output of the instrument, initiation of zero and upscale calibration checks, or control of other capacity monitor functions.

3.2.3 *opacity monitor model, n*—a specific transmissometer or opacity monitor configuration identified by the specific measurement system design, including: (*1*) the use of specific light source, detector(s), lenses, mirrors, and other optical components, (*2*) the physical arrangement of optical and other

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[1] This practice is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.03 on Ambient Atmospheres and Source Emissions.

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[2] *Annual Book of ASTM Standards*, Vol 11.03.

[3] Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

[4] Available from American National Standards Institute, 11 W. 42nd St., 13th floor, New York, NY 10036.

[5] Available from National Institute of Standards and Technology, Gaithersburg, MD 20899.

principal components, (*3*) the specific electronics configuration and signal processing approach, (*4*) the specific calibration check mechanisms and drift/dust compensation devices and approaches, and (*5*) the specific software version and data processing algorithms, as implemented in a particular manufacturing process, at a particular facility and subject to an identifiable quality assurance system.

3.2.3.1 *Discussion*—Changing the retro-reflector material or the size of the retro-reflector aperture is not considered to be a model change unless it changes the basic attributes of the optical system.

3.2.4 *opacity monitoring system, n*—the entire set of equipment necessary to monitor continuously the in-stack opacity, average the emission measurement data, and permanently record monitoring results.

3.2.4.1 *Discussion*—An opacity monitoring system includes at least one opacity monitor with all of its associated interface and peripheral equipment and the specific data recording system (including software) employed by the end user. An opacity monitoring system may include multiple opacity monitors and a common data acquisition and recording system.

3.2.5 *optical density (OD), n*—a logarithmic measure of the amount of incident light attenuated.

3.2.5.1 *Discussion*—OD is related to transmittance and opacity as follows:

$$\mathrm{OD} = \log_{10}(1/\mathrm{T}) = -\log_{10}(\mathrm{T}) = -\log_{10}(1-\mathrm{Op}), \qquad (2)$$

where Op is expressed as a fraction.

3.2.6 *transmittance, n*—the fraction of incident light within a specified optical region that passes through an optical medium.

3.2.7 *transmissometer, n*—an instrument that passes light through a particulate-laden effluent stream and measures *in situ* the optical transmittance of that light within a specified wavelength region.

3.2.7.1 *Discussion*—Single-pass transmissometers consist of a light source and detector components mounted on opposite ends of the measurement path. Double-pass instruments consist of a transceiver (including both light source and detector components) and a reflector mounted on opposite ends of the measurement path.

3.2.7.2 *Discussion*—For the purposes of this practice, the transmissometer includes the following mechanisms (1) means to verify the optical alignment of the components and (2) simulated zero and upscale calibration devices to check calibration drifts when the instrument is installed on a stack or duct.

3.2.7.3 *Discussion*—Transmissometers are sometimes referred to as *opacity analyzers* when they are configured to measure opacity.

Analyzer Zero Adjustments and Devices

3.2.8 *dust compensation, n*—a method or procedure for systematically adjusting the output of a transmissometer to account for reduction in transmitted light reaching the detector (apparent increase in opacity) that is specifically due to the accumulation of dust (that is, particulate matter) on the exposed optical surfaces of the transmissometer.

3.2.8.1 *Discussion*—The dust compensation is determined relative to the previous occasion when the exposed optics were cleaned and the dust compensation was reset to zero. The determination of dust accumulation on surfaces exposed to the effluent must be limited to only those surfaces through which the light beam passes under normal opacity measurement and the simulated zero device or equivalent mechanism necessary for the dust compensation measurement.

3.2.8.2 *Discussion*—The dust accumulation for all of the optical surfaces included in the dust compensation method must actually be measured. Unlike zero drift, which may be either positive or negative, dust compensation can only reduce the apparent opacity. A dust compensation procedure can correct for specific bias and provide measurement results equivalent to the *clean window* condition.

3.2.8.3 *Discussion*—The opacity monitor must provide a means to display the level of dust compensation. Regulatory requirements may impose a limit on the amount of dust compensation that can be applied and require that an alarm be activated when the limit is reached.

3.2.9 *external zero device, n*—an external device for checking the zero alignment of the transmissometer by simulating the zero opacity condition for a specific installed opacity monitor.

3.2.10 *simulated zero device, n*—an automated mechanism within the transmissometer that produces a simulated clear path condition or low level opacity condition.

3.2.10.1 *Discussion*—The simulated zero device is used to check zero drift daily or more frequently and whenever necessary (for example, after corrective actions or repairs) to assess opacity monitor performance while the instrument is installed on the stack or duct.

3.2.10.2 *Discussion*—The proper response to the simulated zero device is established under clear path conditions while the transmissometer is optically aligned at the installation pathlength and accurately calibrated. The simulated zero device is then the surrogate, clear path calibration value, while the opacity monitor is in service.

3.2.10.3 *Discussion*—Simulated zero checks do not necessarily assess the optical alignment, the reflector status (for double-pass systems), or the dust contamination level on all optical surfaces. (See also 6.9.1.)

3.2.11 *zero alignment, n*—the process of establishing the quantitative relationship between the simulated zero device and the actual clear path opacity responses of a transmissometer.

3.2.12 *zero compensation, n*—an automatic adjustment of the transmissometer to achieve the correct response to the simulated zero device.

3.2.12.1 *Discussion*—The zero compensation adjustment is fundamental to the transmissometer design and may be inherent to its operation (for example, continuous adjustment based on comparison to reference values/conditions, use of automatic control mechanisms, rapid comparisons with simulated zero and upscale calibration drift check values, and so forth) or it may occur each time a calibration check cycle (zero and upscale calibration drift check) is performed by applying either analog or digital adjustments within the transmissometer.

3.2.12.2 *Discussion*—For opacity monitors that do not distinguish between zero compensation and dust compensation, the accumulated zero compensation may be designated as the dust compensation. Regulatory requirements may impose a limit on the amount of dust compensation that can be applied and require that an alarm be activated when the limit is reached.

3.2.13 *zero drift*, *n*—the difference between the opacity monitor response to the simulated zero device and its nominal value (reported as percent opacity) after a period of normal continuous operation during which no maintenance, repairs, or external adjustments to the opacity monitor took place.

3.2.13.1 *Discussion*—Zero drift may occur due to changes in the light source, changes in the detector, variations due to internal scattering, changes in electronic components, or varying environmental conditions such as temperature, voltage or other external factors. Depending on the design of the transmissometer, particulate matter (that is, dust) deposited on optical surfaces may contribute to zero drift. Zero drift may be positive or negative.

Calibrations and Adjustments

3.2.14 *attenuator*, *n*—a glass or grid filter that reduces the transmittance of light.

3.2.15 *calibration drift*, *n*—the difference between the opacity monitor response to the upscale calibration device and its nominal value after a period of normal continuous operation during which no maintenance, repairs, or external adjustments to the opacity monitor took place.

3.2.15.1 *Discussion*—Calibration drift may be determined after determining and correcting for zero drift. For opacity monitors that include automatic zero compensation or dust compensation features, calibration drift may be determined after zero drift or dust compensation, or both, are applied.

3.2.16 *calibration error*, *n*—the sum of the absolute value of the mean difference and confidence coefficient for the opacity values indicated by an optically aligned opacity monitor (laboratory test) or opacity monitoring system (field test) as compared to the known values of three calibration attenuators under clear path conditions.

3.2.16.1 *Discussion*—The calibration error indicates the fundamental calibration status of the opacity.

3.2.17 *external adjustment*, *n*—either (*1*) a physical adjustment to a component of the opacity monitoring system that affects its response or its performance, or (*2*) an adjustment applied by the data acquisition system (for example, mathematical adjustment to compensate for drift) which is external to the transmissometer and control unit, if applicable.

3.2.17.1 *Discussion*—External adjustments are made at the election of the end user but may be subject to various regulatory requirements.

3.2.18 *intrinsic adjustment*, *n*—an automatic and essential feature of an opacity monitor that provides for the internal control of specific components or adjustment of the opacity monitor response in a manner consistent with the manufacturer's design of the instrument and its intended operation.

3.2.18.1 *Discussion*—Examples of intrinsic adjustments include automatic gain control used to maintain signal amplitudes constant with respect to some reference value, or the technique of ratioing the measurement and reference beams in dual beam systems. Intrinsic adjustments are either non-elective or are configured according to factory recommended procedures; they are not subject to change from time to time at the discretion of the end user.

3.2.19 *upscale calibration device*, *n*—an automated mechanism (employing a filter or reduced reflectance device) within the transmissometer that produces an upscale opacity value.

3.2.19.1 *Discussion*—The upscale calibration device is used to check the upscale drift of the measurement system. It may be used in conjunction with the simulated zero device (for example, filter superimposed on simulated zero reflector) or a parallel fashion (for example, zero and upscale (reduced reflectance) devices applied to the light beam sequentially). (See also 6.9.2.)

Opacity Monitor Location Characteristics

3.2.20 *installation pathlength*, *n*—the installation flange-to-flange separation distance between the transceiver and reflector for a double-pass transmissometer or between the transmitter and receiver for a single-pass transmissometer.

3.2.21 *monitoring pathlength*, *n*—the effective single pass depth of effluent between the receiver and the transmitter of a single-pass transmissometer, or between the transceiver and reflector of a double-pass transmissometer at the installation location.

3.2.22 *emission outlet pathlength*, *n*—the physical pathlength (single pass depth of effluent) at the location where emissions are released to the atmosphere.

3.2.22.1 *Discussion*—For circular stacks, the emission outlet pathlength is the internal diameter at the stack exit. For non-circular outlets, the emission outlet pathlength is the hydraulic diameter. For rectangular stacks:

$$D = (2LW)/(L + W), \qquad (3)$$

where L is the length of the outlet and W is the width of the stack exit.

3.2.23 *pathlength correction factor (PLCF)*, *n*—the ratio of the emission outlet pathlength to the monitoring pathlength.

3.2.23.1 *Discussion*—The PLCF is used to calculate the equivalent single pass opacity that would be observed at the stack exit.

3.2.23.2 *Discussion*—A number of similar terms are found in the literature, manufacturer operating manuals, and in common usage. OPLR (optical pathlength ratio) and STR (stack taper ratio) are common. The OPLR is equal to one half of the pathlength correction. Refer to the instrument manufacturer for the proper factor.

Opacity Monitor Optical Characteristics

3.2.24 *angle of projection (AOP)*, *n*—the total angle that contains all of the visible (photopic) radiation projected from the light source of the transmissometer at a level greater than 2.5 % of its peak illuminance.

3.2.25 *angle of view (AOV)*, *n*—the total angle that contains all of the visible (photopic) radiation detected by the photodetector assembly of the transmissometer at a level greater than 2.5 % of the peak detector response.

3.2.26 *instrument response time*, *n*—the time required for the electrical output of an opacity monitor to achieve 95 % of a step change in the path opacity.

3.2.27 *mean spectral response, n*—the mean response wavelength of the wavelength distribution for the effective spectral response curve of the transmissometer.

3.2.28 *optical alignment indicator, n*—a device or means to determine objectively the optical alignment status of opacity monitor components.

3.2.29 *peak spectral response, n*—the wavelength of maximum sensitivity of the transmissometer.

3.2.30 *photopic, n*—a region of the electromagnetic spectrum defined by the response of the light-adapted human eye as characterized in the "Source C, Human Eye Response" contained in 40CFR60, Appendix B, Performance Specification 1.

4. Summary of Practice

4.1 A comprehensive series of specifications and test procedures that opacity monitor manufacturers must use to certify opacity monitoring equipment (that is, that the equipment meets minimum design and performance requirements) prior to shipment to the end user is provided. The design and performance specifications are summarized in Table 1.

4.2 Design specifications and test procedures for (*1*) peak and mean spectral responses, (*2*) angle of view and angle of projection, (*3*) insensitivity to supply voltage variations, (*4*) thermal stability, (*5*) insensitivity to ambient light, and (*6*) an optional procedure for opacity monitors with external zero devices that states or other regulatory agencies might require are included. The manufacturer periodically selects and tests for conformance with these design specifications an instrument that is representative of a group of instruments) produced during a specified period or lot. Non-conformance with the design specifications requires corrective action and retesting. Each remanufactured opacity monitor must be tested to demonstrate conformance with the design specifications. The test frequency, transmissometer installation pathlength (that is, set-up distance) and pathlength correction factor for each design specification test are summarized in Table 2.

4.3 This practice includes manufacturer's performance specifications and test procedures for (*1*) instrument response time, (*2*) calibration error, (*3*) optical alignment sight performance - homogeneity of light beam and detector. It also includes a performance check of the spectral response of the instrument. Conformance with these performance specifications is determined by testing each opacity monitor prior to shipment to the end user. (The validity of the results of the calibration error test depends upon the accuracy of the installation pathlength measurements, which is provided by the end user.) The test frequency, transmissometer installation pathlength (that is, set-up distance) and pathlength correction factor for each performance specification test are summarized in Table 3.

4.4 This practice establishes appropriate guidelines for QA programs for manufacturers of continuous opacity monitors, including corrective actions when non-conformance with specifications is detected.

5. Significance and Use

5.1 Continuous opacity monitors are required to be installed at many stationary sources of air pollution by federal, state, and local air pollution control agency regulations. EPA regulations regarding the design and performance of opacity monitoring systems for sources subject to "Standards of Performance for New Stationary Sources" are found in 40 CFR 60, Subpart A General Provisions, §60.13 Monitoring Provisions, Appendix B, Performance Specification 1, and in applicable source-specific subparts. Many states have adopted these or very similar requirements for opacity monitoring systems.

TABLE 1 Summary of Manufacturer's Specifications and Requirements

Specification	Requirement
Spectral response	peak and mean spectral response between 500 and 600 nm: less than 10% of peak response below 400 nm and above 700 nm
Angle of view, angle of projection	≤4° for all radiation above 2.5 % of peak
Insensitivity to supply voltage variations	±1.0 % opacity max. change over specified range of supply voltage variation, or ±10 % variation from the nominal supply voltage
Thermal stability	±2.0 % opacity change per 40°F change over specified operational range
Insensitivity to ambient light	±2.0 % opacity max. change from sunrise to sunset with at least one 1-h average solar radiation level of ≥ 900 W/m^2
External audit filter access	required
External zero device repeatability - Optional	±1.0 % opacity
Automated calibration checks	check of all active analyzer internal optics with power or curvature, all active electronic circuitry including the light source and photodetector assembly, and electric or electro-mechanical systems used during normal measurement operation
Simulated zero check device	simulated condition during which the energy reaching the detector is between 90 and 190 % of the energy reaching the detector under actual clear path conditions
Upscale calibration check device	check of the measurement system where the energy level reaching the detector is between the energy levels corresponding to 10 % opacity and the highest level filter used to determine calibration error
Status indicators	manufacturer to identify and specify
Pathlength correction factor security	manufacturer to specify one of three options
Measurement output resolution	0.5 % opacity over measurement range from -5 % to 50 % opacity, or higher value
Measurement and recording frequency	sampling and analyzing at least every 10 s: calculate averages from at least 6 measurements per minute
Instrument response time	≤10 s to 95 % of final value
Calibration error	≤3 % opacity for the sum of the absolute value of mean difference and 95 % confidence coefficient for each of three test filters
Optical alignment indicator - (uniformity of light beam and detector)	clear indication of misalignment at or before the point where opacity changes ±2 % due to misalignment as system is misaligned both linearly and rotationally in horizontal and vertical planes
Calibration device repeatability	≤1.5 % opacity

TABLE 2 Manufacturer's Design Specifications – Test Frequency, Set-Up Distance, and Pathlength Correction Factor

Manufacturer's Design Specification	Test Frequency	Set-Up Distance	Pathlength Correction Factor
Spectral Response	annually, and following failure of spectral response performance check[A]	1 to 3 m when measured (not applicable when spectral response is calculated)	NA
Angle of view, angle of projection	monthly, or 1 in 20 units (whichever is more frequent)	3 m	NA
Insensitivity to supply voltage variations	monthly, or 1 in 20 units (whichever is more frequent)	3 m	1.0
Thermal stability	annually[B]	3 m (external jig for tests)	1.0
Insensitivity to ambient light	annually[B]	3 m	1.0
External zero device repeatability - optional	annually[B]	3 m	1.0
Additional design specifications[C]	as applicable		

[A] The spectral response is determined annually for each model and whenever there is a change in the design, manufacturing process, or component that might affect performance. Reevaluation of the spectral response is necessary when an instrument fails to meet the spectral response performance check.

[B] Annually, and whenever there is a change in the design, manufacturing process, or component that might affect performance.

[C] The manufacturer shall certify that the opacity monitor design meets the applicable requirements for (*a*) external audit filter access, (*b*) external zero device (if applicable), (*c*) simulated zero and upscale calibration devices, (*d*) status indicators, (*e*) pathlength correction factor security, (*f*) measurement output resolution, and (*g*) measurement recording frequency.

TABLE 3 Manufacturer's Performance Specification – Test Applicability, Set-Up Distance and Pathlength Correction Factor

Manufacturer's Performance Specification	Test Applicability	Set-Up Distance	Pathlength Correction Factor
Instrument response time	each instrument	per actual installation	per actual installation
Calibration error	each instrument	per actual installation[A]	per actual installation[A]
Acceptable tolerance comparing test to actual conditions		±10 % reset clear path zero values for subsequent monitoring[B]	±10 %, use actual value for all subsequent monitoring[B]
Optical alignment indicator - (uniformity of light beam and detector)	each instrument	per actual installation	per actual installation
Spectral response performance check	each instrument	per actual installation	per actual installation
Calibration device repeatability	each instrument	per actual installation	per actual installation

[A] Default test values are provided for use where the installation pathlength and pathlength correction factor can not be determined.

[B] When actual measurements are within ±10 % tolerance, a field performance audit can be performed rather than a field calibration error test at the time of installation.

5.2 Regulated industrial facilities are required to report continuous opacity monitoring data to control agencies on a periodic basis. The control agencies use the data as an indirect measure of particulate emission levels and as an indicator of the adequacy of process and control equipment operation and maintenance practices.

5.3 EPA Performance Specification 1 provides minimum specifications for opacity monitors and requires source owners or operators of regulated facilities to demonstrate that their installed systems meet certain design and performance specifications. Performance Specification 1 allows, as an alternative to testing each instrument, manufacturers to demonstrate conformance with certain design specifications by selecting and testing representative instruments.

5.4 Previous experience has demonstrated that EPA Performance Specification 1 does not address all of the important design and performance parameters for opacity monitoring systems. The additional design and performance specifications included in this practice are needed to eliminate many of the performance problems that have been encountered. This practice also provides purchasers and vendors flexibility, by designing the test procedures for basic transmissometer components or opacity monitors, or in certain cases, complete opacity monitoring systems. However, the specifications and test procedures are also sufficiently detailed to support the manufacturer's certification and to facilitate independent third party evaluations (if desired) of the procedures.

5.5 Purchasers of opacity monitoring equipment meeting all of the requirements of this practice are assured that the opacity monitoring equipment meets all of the design requirements of EPA Performance Specification 1, and additional design specifications that eliminate many of the operational problems that have been encountered in the field. Purchasers can rely on the manufacturer's published operating range specifications for ambient temperature and supply voltage. These purchasers are also assured that the specific instrument has been tested at the point of manufacture and demonstrated to meet the manufacturer's performance specifications for instrument response time, calibration error (based on pathlength measurements provided by the end user), optical alignment, and the spectral response performance check requirement. Conformance with the requirements of this practice ensures conformance with all of the requirements of 40CFR60, Appendix B, Performance Specification 1 except those requirements for which tests are required after installation.

5.6 The original manufacturer, or those involved in the repair, remanufacture, or resale of opacity monitors can use this practice to demonstrate that the equipment components or opacity monitoring systems provided meet appropriate design and performance specifications.

5.7 The applicable test procedures and specifications of this practice are selected to address the equipment and activities that are within the control of the manufacturer; they do not mandate testing of the opacity system data recording equipment or reporting.

5.8 This practice also may serve as the basis for third party independent audits of the certification procedures used by manufacturers of opacity monitoring equipment.

6. Procedure—Design Specification Verification

6.1 *Test Opacity Monitor Selection, Test Frequency, and Summary of Tests*:

6.1.1 Perform the design specification verification procedures in this section for each representative model or configuration involving substantially different optics, electronics, or software before being shipped to the end user.

6.1.2 At a minimum, select one opacity monitor from each month's production, or one opacity monitor from each group of twenty opacity monitors, whichever is more frequent. Test this opacity monitor for (*1*) angle of view, (*2*) angle of projection, and (*3*) insensitivity to supply voltage variations. If any design specification is unacceptable, institute corrective action according to the established quality assurance program and remedy the cause of unacceptability for all opacity monitors produced during the month or group of twenty. In addition, test all of the opacity monitors in the group and verify conformance with the design specifications before shipment to the end users.

NOTE 1—The selected opacity monitor may be the first opacity monitor produced each month, or the first opacity monitor in each group of twenty, provided that it is representative of the entire group.

6.1.3 At a minimum, test one opacity monitor each year for (*1*) spectral response, (*2*) thermal stability, and (*3*) insensitivity to ambient light. If any design specification is unacceptable, institute corrective action according to the established quality assurance program and remedy the cause of unacceptability for all affected opacity monitors. In addition, retest another representative opacity monitor after corrective action has been implemented to verify that the problem has been resolved.

6.1.4 Certify that the opacity monitor design meets the applicable requirements (see 6.7-6.13) for (*1*) external audit filter access, (*2*) external zero device (if applicable), (*3*) simulated zero and upscale calibration devices, (*4*) status indicators, (*5*) pathlength correction factor security, (*6*) measurement output resolution, and (*7*) measurement recording frequency. Maintain documentation of tests and data necessary to support certification.

6.2 *Spectral Response*:

NOTE 2—The purpose of the spectral response specifications is to ensure that the transmissometer measures the transmittance of light within the photopic range. The spectral response requirements ensure some level of consistency among opacity monitors because the determination of transmittance for effluent streams depends on the particle size, wavelength, and other parameters. The spectral response requirements also eliminate potential interfering effects due to absorption by various gaseous constituents except NO_2 which can be an interferent if present in abnormally high concentrations or over long pathlengths, or both. The spectral response requirements apply to the entire transmissometer. Any combination of components may be used in the transmissometer so long as the response of the entire transmissometer satisfies the applicable requirements.

6.2.1 *Test Frequency*— See 6.1.3. In addition, conduct this test (*1*) anytime a change in the manufacturing process occurs or a change in a component that may affect the spectral response of the transmissometer occurs or (2) on each opacity monitor that fails the spectral response performance check in 7.10.

6.2.2 *Specification*— The peak and mean spectral responses must occur between 500 nm and 600 nm. The response at any wavelength below 400 nm and above 700 nm must be less than 10 % of the peak spectral response. Calculate the mean spectral response as the arithmetic mean value of the wavelength distribution for the effective spectral response curve of the transmissometer.

6.2.3 *Spectral Response Design Specification Verification Procedure*—Determine the spectral response of the transmissometer by either of the procedures in 6.2.4 (Option 1) or 6.2.5 (Option 2), then calculate the mean response wavelength from the normalized spectral response curve according to 6.2.6. Option 1 is to measure the spectral response using a variable slit monochromator. Option 2 is to determine the spectral response from manufacturer-supplied data for the active optical components of the measurement system.

6.2.4 *Option 1, Monochromator*—Use the following procedure:

6.2.4.1 Verify the performance of the monochromator using a NIST traceable photopic band pass filter or light source, or both.

6.2.4.2 Set-up, optically align, and calibrate the transmissometer for operation on a pathlength of 1 to 3 m.

6.2.4.3 Connect an appropriate data recorder to the transmissometer and adjust the gain to an acceptable measurement level.

6.2.4.4 Place the monochromator in the optical path with the slit edge at an appropriate distance from the permanently mounted focusing lenses.

6.2.4.5 Use the monochromator with a range from 350 nm to 750 nm or greater resolution. Record the response of the transmissometer at each wavelength in units of optical density or voltage.

6.2.4.6 Cover the reflector for double-pass transmissometers, or turn off the light source for single-pass transmissometers, and repeat the test to compensate measurement values for dark current at each wavelength.

6.2.4.7 Determine the spectral response from the opacity monitor double pass response and the monochromator calibration.

6.2.4.8 Graph the raw spectral response of the transmissometer over the test range.

6.2.4.9 Normalize the raw response curve to unity by dividing the response at 10 nm intervals by the peak response.

6.2.5 *Option 2, Calculation from Manufacturer Supplied Data*—Obtain data from component suppliers that describes the spectral characteristics of the light source, detector, filters, and all other optical components that are part of the instrument design and affect the spectral response of the transmissometer. Ensure that such information is accurately determined using reliable means and that the information is representative of the specific components used in current production of the transmissometer under evaluation. Update the information at least every year or when new components are used, or both. Keep the information and records necessary to demonstrate its applicability to the current spectral response determination on file. Using the component manufacturer-supplied data, calculate the effective spectral response for the transmissometer as follows:

6.2.5.1 Obtain the spectral emission curve for the source. The data must be applicable for the same voltages or currents, or both, as that used to power the source in the instrument.

6.2.5.2 Obtain the spectral sensitivity curve for the detector that is being used in the system.

6.2.5.3 Obtain spectral transmittance curves for all filters and other active optical components that affect the spectral response.

6.2.5.4 Perform a point-wise multiplication of the data obtained in 6.2.5.1-6.2.5.3, at 10 nm intervals, over the range

350 to 750 nm, to yield the raw response curve for the system.

6.2.5.5 Normalize the raw response curve to unity by dividing the response at 10 nm intervals by the peak response.

6.2.6 Using the results from Option 1 or 2, as applicable, determine conformance to the specifications in 6.2.2. Then calculate the mean response wavelength (response-weighted average wavelength) by (*1*) multiplying the response at 10 nm intervals by the wavelength, (*2*) summing all the products, and (*3*) dividing by the sum of all 10 nm interval responses. Verify that this result is greater than 500 nm but less than 600 nm.

6.2.7 *Monitor-Specific Performance Check Limits*—Establish the monitor-specific performance check limits for use in conducting the Spectral Response Performance Check (7.10) as follows:

NOTE 3—The equivalent single-pass opacity from 6.2.7.2 and the single-pass opacity results corresponding to the applicable shifts from 6.2.7.3 bound the acceptable limits for the spectral response performance check.

6.2.7.1 Obtain a photopic transmission filter that has (*1*) a peak transmission ≥70 %, (*2*) maximum transmission between 550 nm and 560 nm, (*3*) half-maximum transmission between 500 nm and 520 nm, (*4*) half-maximum transmission between 600 nm and 620 nm, (*5*) transmission <10 % at any wavelength less than 450 nm or greater than 650 nm, and (*6*) a traceable calibration. Calibrate and verify the transmittance of the photopic filter as a function of wavelength initially and at least annually.

6.2.7.2 Calculate the expected single-pass opacity (assuming PLCF=1) that would result from inserting the photopic transmission filter into the clear-stack path of the transmissometer by (*1*) performing a point-wise multiplication of the photopic transmission filter curve with the normalized transmissometer response curve (obtained from 6.2.4.9 or 6.2.5.5), (*2*) summing the products, (*3*) dividing by the sum of the 10 nm responses to form the single-pass transmission, and (*4*) calculating the equivalent single-pass opacity.

6.2.7.3 Repeat the calculations in 6.2.7.2, except use (*1*) the normalized transmissometer curve shifted by +20 nm or the amount which would cause the peak or mean spectral response to shift to the limiting value of 600 nm, whichever shift is less, and (*2*) the normalized transmissometer curve shifted by −20 nm or the amount which would cause the peak or mean spectral response to shift to the limiting value of 500 nm, whichever shift is less.

6.2.7.4 Repeat the calculations with any design changes involving the source, detector(s), or light transmitting optics. Although failure of the spectral response performance check in 7.10 does not necessarily mean that the transmissometer response is no longer within the photopic range, it is a sufficient basis to warrant additional investigation, including reevaluation of the spectral response and performance check limits, explanation, and documentation of the problem.

6.3 *Angle of View and Angle of Projection*:

NOTE 4—The purpose of the angle of view (AOV) and angle of projection (AOP) design specifications is to minimize the effects of light scattering in the measurement path when determining transmittance or opacity.

6.3.1 *Test Frequency*— See 6.1.2. Manufacturers that demonstrate and document using good engineering practice that a specific design results in an AOP of less than 0.5° are not required to perform the following AOP or AOV tests.

6.3.2 *Specification*— The total AOP and the total AOV must each be no greater than 4°. Transmissometers with an AOP of less than 0.5° are exempt from the AOV or AOP specification.

6.3.3 *AOV and AOP Design Specification Verification Procedure*—Conduct the AOV and AOP tests using the procedures given in 6.3.4-6.3.13.

6.3.4 *Transmissometer Configuration*—Conduct the AOV and AOP tests with the complete transmissometer assembly, including all parts of the measurement system that may impact the results. Provide a justification of (*1*) exactly what is included and excluded from the AOV and AOP tests and (*2*) any test procedure modifications necessary to accommodate particular designs, such as those that may be required for dual beam designs that are chopped and synchronously detected. Include the justifications with documentation of the results.

6.3.5 *Set-Up*—Focus and configure the transmissometer for a flange-to-flange installation separation distance of 3 m.

6.3.6 *Test Fixture*— Set up the AOV test fixture that incorporates (*1*) a movable light source along arcs of 3 m radius relative to the first optical surface encountered by the light beam entering the detector housing assembly, in both the horizontal and vertical directions relative to the normal installation orientation, and (*2*) recording measurements at 2.5 cm increments along the arc. Similarly, set up the AOP test fixture that incorporates (*1*) a movable photodetector along an arc of 3 m radius relative to the final optical surface encountered by the light beam exiting the transmitter housing assembly, in both the horizontal and vertical directions relative to the normal installation orientation, and (*2*) recording measurements at 2.5 cm increments along the arc.

NOTE 5—It is helpful to mount on test stands the detector and transmitter housings for single-pass transmissometers, or the transceiver for double-pass transmissometers.

6.3.7 *Alternative Test Fixture*—For the AOV test, at a distance of 3 m from a stationary light source, mount the detector housing on a turntable that can be rotated (both horizontally and vertically) in increments of 0.5° [28.6 min], corresponding to measurements displaced 2.5 cm along the arc, to a maximum angle of 5° (corresponding to a distance of 26 cm along the arc) on either side of the alignment centerline. Similarly, for the AOP test, mount transmitter housing on the turntable at a distance of 3 m relative to a stationary photodetector.

NOTE 6—If the turntable is capable of rotating only in either the horizontal or vertical direction, the detector or transmitter housing may be mounted on its side or bottom (as appropriate) to simulate the other direction.

6.3.8 *Light Source*— For the AOV test, use a small non-directional light source (less than 3 cm wide relative to the direction of movement) that (*1*) includes the visible wavelengths emitted by the light source installed in the transmissometer, (*2*) provides sufficient illuminance to conduct the test but doe snot saturate the detector, (*3*) does not include lenses or focusing devices, and (*4*) does not include non-directional characteristics, that is, the intensity in the 20° sector facing the detector assembly varies by less than ±10 %.

NOTE 7—A light source that does not meet the non-directional criteria may still be used for the AOV test, if a specific procedure is followed. This procedure is given in 6.3.9.

6.3.9 *Alternative Light Source*—For the AOV test, if the light source does not meet the non-directional criteria, rotate the light source in the vertical and horizontal planes about its normal optical axis as it is pointed at the entrance aperture of the instrument under test in order to obtain the maximum response from the instrument under test at each position in the test procedure.

6.3.10 *AOV Test Procedure*—Test the entire detector assembly (that is, transceiver for double-pass transmissometers or receiver/detector for a single-pass transmissometer). If applicable, include the mounting flanges normally supplied with the opacity monitor. Use an appropriate data recorder to record continuously the detector response during the test.

NOTE 8—Alternative AOV test procedures are necessary for certain designs. For example, a transmissometer with an optical chopper/modulator responds only to light modulated at a certain frequency. An external chopper/modulator used in conjunction with the test light source must match both the phase and duty cycle for accurate results. If this cannot be done, the manufacturer may either (*1*) provide additional electronics to drive another similar external source in parallel wit the internal source or (2) modify the detector electronics so that its response may be used to accurately evaluate the AOV of the test transmissometer. The manufacturer must take appropriate measures to ensure (*1*) that the background, or ambient light, and detector offsets do not significantly reduce the accuracy of the AOV measurements, (2) that the field of view restricting hardware normally included with the instrument are not modified in any way, and (*3*) that good engineering practice is followed in the design of the test configuration to ensure an accurate measurement of AOV.

6.3.10.1 Align the test light source at the center position and observe the detector assembly response. Optimize the test light source and optical chopper/modulator (if applicable) to maximize the detector assembly response. If the detector response is not within the normal operating range (that is, 25 to 200 % of the energy value equivalent to a clear path transmittance measurement for the transmissometer), adjust the test apparatus (for example, light source power supply) to achieve a detector response in the acceptable range.

6.3.10.2 Position the test light source on the horizontal arc 26 cm from the detector centerline (5°) and record the detector response. Move the light source along the arc at intervals not larger than 2.5 cm (or rotate the turntable in increments not larger than 0.5°) and record the detector response for each measurement location. Continue to make measurements through the aligned position and on until a position 26 cm (5°) on the opposite side of the arc from the starting position is reached. Record the response for each measurement location and over the full test range; continue recording data for all positions up to 26 cm (5°) even if no response is observed at an angle of ≤26 cm (5°) from the centerline.

6.3.10.3 Repeat the AOV test on an arc in the vertical direction relative to the normal orientation of the detector housing.

6.3.10.4 For both the horizontal and vertical directions, calculate the relative response of the detector as a function of viewing angle (response at each measurement location as a percentage of the peak response). Determine the maximum viewing angle for the horizontal and vertical directions yielding a response greater than 2.5 % of the peak response. Determine conformance to the specification in 6.3.2. Report these angles as the angle of view. Report the relative angle of view curves in both the horizontal and vertical directions. Document and explain any modifications to the test procedures as described in 6.3.11.

6.3.11 *AOP Test Procedure*—Perform this test for the entire light source assembly (that is, transceiver for double-pass transmissometers or transmitter for single-pass transmissometers). The test may also include the mounting flanges normally supplied with the opacity monitor. Conduct the AOP test using the procedures in either 6.3.12 or 6.3.13.

6.3.12 *Option 1*—Use a photodetector (*1*) that is less than 3 cm wide relative to the direction of movement, (*2*) that is preferably of the same type and has the same spectral response as the photodetector in the transmissometer, (*3*) that is capable of detecting 1 % of the peak response, and (*4*) that does not saturate at the peak illuminance (that is, when aligned at the center position of the light beam. Use an appropriate data recorder to record continuously the photodetector response during the test.

6.3.12.1 Perform this test in a dark room. If the external photodetector output is measured in a dc-coupled circuit, measure the ambient light level in the room (must be <0.5 % of the peak light intensity to accurately define the point at which 2.5 % peak intensity occurs). If the external photodetector is measured in an ac-coupled configuration, demonstrate that (*1*) ambient light level in the room, when added to the test light beam, does not cause the detector to saturate, and (2) turning on and off the ambient lights does not change the detected signal output. Include documentation for these demonstrations in the report.

6.3.12.2 Position the photodetector on the horizontal arc 26 cm from the projected beam centerline (5°) and record the response. Move the photodetector along the arc at ≤2.5-cm intervals (or rotate the turntable in ≤0.5° increments) until a position 26 cm (5°) on the opposite side of the arc is reached. Record the response for each measurement location and over the full test range; continue recording data for all positions up to 26 cm (5°) even if no response is observed at an angle of ≤26 cm (5°) from the centerline.

6.3.12.3 Repeat the AOP test on an arc in the vertical direction relative to the normal orientation of the detector housing.

6.3.12.4 For both the horizontal and vertical directions, calculate the relative response of the photodetector as a function of projection angle (response at each measurement location as a percentage of the peak response). Determine the maximum projection angle for the horizontal and vertical directions yielding a response greater than 2.5 % of the peak response. Determine conformance to the specification in 6.3.2. Report these angles as the angle of projection. Report the relative angle of projection curves in both the horizontal and vertical directions.

6.3.13 *Option 2*—Use this test procedure for only transmissometer designs that have previously met the AOP specification using Option 1 procedure during the preceding 12 months. Ensure that the light beam is focused at the actual flange-to-flange separation distance of the transmissometer.

6.3.13.1 Perform this test in a darkened room. Project the light beam onto a target located at a distance of 3 m from the transceiver/transmitter. Focus the light beam on the target.

6.3.13.2 Measure the beam dimensions (for example, diameter) on the target in both the horizontal and vertical directions. Calculate the maximum total angle of projection (that is, total subtended angle) based on the separation distance and beam dimensions. Compare this result to the previously measured AOP result obtained using Option 1. If the AOP results obtained by Option 1 and Option 2 do not agree within ±0.3°, repeat the test using Option 1.

6.3.13.3 Report the greater AOV result of Option 1 or Option 2 as the AOV for the test instrument.

6.4 *Insensitivity to Supply Voltage Variations*:

NOTE 9—The purpose of this design specification is to ensure that the accuracy of opacity monitoring data is not affected by supply voltage variations over ±10 % from nominal or the range specified by the manufacturer, whichever is greater. This specification does not address rapid voltage fluctuations (that is, peaks, glitches, or other transient conditions), emf susceptibility or frequency variations in the power supply.

6.4.1 *Test Frequency*— See 6.1.2.

6.4.2 *Specification*— The opacity monitor output (measurement and calibration check responses, both with and without compensation, if applicable) must not deviate more than ±1.0 % single pass opacity for variations in the supply voltage over ±10 % from nominal or the range specified by the manufacturer, whichever is greater.

6.4.3 *Design Specification Verification Procedure*:

6.4.3.1 Determine the acceptable supply voltage range from the manufacturer's published specifications for the model of opacity monitor to be tested. Use a variable voltage regulator and a digital voltmeter to monitor the rms supply voltage to within ±0.5 %. Measure the supply voltage over ±10 % from nominal, or the range specified by the manufacturer, whichever is greater.

6.4.3.2 Set-up and align the opacity monitor (transceiver and reflector for double-pass opacity monitors, or transmitter and receiver for single-pass opacity monitors) at a measurement pathlength of 3 m. Use a pathlength correction factor of 1.0. Calibrate the instrument using external attenuators at the nominal operating voltage. Insert an external attenuator with a nominal value between 10 and 20 % single-pass opacity into the measurement path and record the response. Initiate a calibration check cycle and record the low level and upscale responses.

6.4.3.3 Do not initiate any calibration check cycle during this test procedure except as specifically required. Decrease the supply voltage in increments of 2 % of the nominal value and record the one-minute or more frequent measurement response to the attenuator at each voltage (after the instrument response has stabilized) until the minimum value is reached. Initiate a calibration check cycle at the minimum supply voltage and record the low level and upscale responses. Reset the supply voltage to the nominal value and then increase the supply voltage in increments of 2 % of the nominal value and record the measurement response to the attenuator at each voltage (after the instrument response has stabilized) until the maximum value is reached. Initiate a calibration check cycle at the maximum supply voltage and record the low level and upscale responses, both with and without compensation, if applicable.

6.4.3.4 Determine conformance to specifications in 6.4.2.

6.5 *Thermal Stability*:

NOTE 10—The purpose of this design specification is to ensure that the accuracy of opacity monitoring data is not affected by ambient temperature variations over the range specified by the manufacturer.

6.5.1 *Test Frequency*— See 6.1.3. Repeat this test anytime there is a major change in the manufacturing process or change in a major component that could affect thermal stability.

6.5.2 *Specification*— The opacity monitor output output (measurement and calibration check responses, both with and without compensation, if applicable) must not deviate more than ±2.0 % single pass opacity for every 22.2°C (40°F) change in ambient temperature over the range specified by the manufacturer.

6.5.3 *Design Specification Verification Procedure*:

6.5.3.1 Determine the acceptable ambient temperature range from the manufacturer's published specifications for the model of opacity monitor to be tested. Use a climate chamber capable of operation over the specified range. If the climate chamber cannot achieve the full range (for example, cannot reach minimum temperatures), clearly state the temperature range over which the opacity monitor was tested and provide additional documentation of performance beyond this range to justify operating at lower temperatures.

6.5.3.2 Set-up and align the opacity monitor (transceiver and reflector for double-pass opacity monitors, or transmitter and receiver for single-pass opacity monitors) at a measurement pathlength of 3 m. Use a pathlength correction factor of 1.0. If the opacity monitor design introduces purge air through the housing that contains optical components of the transceiver, transmitter, or detector, operate the purge air system during this test. If the purge air does not contact internal optics and electronics, the air purge system need not be operative during the test.

NOTE 11—For double-pass systems with reflectors that can be shown to be insensitive to temperature, this test may be performed using a zero reference similar to an external zero jig, but one that is designed specifically to evaluate the temperature stability of the instrument for this test. This device must be designed to be temperature invariant so that the test evaluates the stability of the instrument, not the stability of the zero reference. Another acceptable approach is to construct a test chamber where the reflector is mounted outside the chamber at a constant temperature. The control unit, if applicable, need not be installed in the climate chamber if it is to be installed in a controlled environment by the end user.

6.5.3.3 Establish proper calibration of the instrument using external attenuators at a moderate temperature that is, 21.1 ± 2.8°C (70 ± 5°F). Insert an external attenuator with a single-pass value between 10 and 20 % opacity into the measurement path and record the response. Initiate a calibration check cycle and record the low level and upscale responses.

NOTE 12—Grid filters are recommended for these tests to eliminate temperature dependency of the attenuator value.

6.5.3.4 Do not initiate any calibration check cycle during this test procedure except as specifically stated. Continuously record the temperature and measurement response to the attenuator during this entire test. Decrease the temperature in the climate chamber at a rate not to exceed 11.1°C (20°F) per hour until the minimum temperature is reached. Note data recorded during brief periods when condensation occurs on optical surfaces due to temperature changes. Allow the opacity monitor to remain at the minimum temperature for at least one hour and then initiate a calibration check cycle and record the low level and upscale responses with and without compensation, if applicable. Return the opacity monitor to the initial temperature and allow sufficient time for it to equilibrate and for any condensed moisture on exposed optical surfaces to evaporate. Increase the temperature in the climate chamber at a rate not to exceed 11.1°C (20°F) per hour until the maximum temperature is reached. Allow the opacity monitor to remain at the maximum temperature for at lest one hour and then initiate a calibration check cycle and record the low level and upscale responses.

NOTE 13—The notations when condensation occurs are for explanatory purposes only.

6.5.3.5 Determine conformance to specifications in 6.5.2.

6.6 *Insensitivity to Ambient Light*:

NOTE 14—The purpose of this design specification is to ensure that opacity monitoring data are not affected by ambient light.

6.6.1 *Test Frequency*— See 6.1.3. Repeat this test anytime there is a major change in the manufacturing process or change in a major component that could affect the opacity monitor sensitivity to ambient light.

6.6.2 *Specification*— The opacity monitor output (measurement and calibration check responses, both with and without compensation, if applicable) must not deviate more than ±2.0 % single pass opacity when exposed to ambient sunlight over the course of a day.

6.6.3 *Design Specification Verification Procedure*:

6.6.3.1 Perform this test (*1*) at a time of maximum insolation, on a clear day where light scattering from atmospheric haze, clouds, or particulate matter are at a minimum, (*2*) when at least one 1-h solar radiation average is ≥900 W/m ^{2}, and (*3*) for a specific opacity monitor that has successfully completed the spectral response, thermal stability tests, and other design specification verification procedures.

6.6.3.2 Set-up the opacity monitor outside, with the light path in a horizontal position, and where it will be directly exposed to sunlight for the entire day. Use mounting flanges of normal length, and attach the flanges to mounting plates that extend at least 0.305 m (12 in.) above, below, and to both sides of the mounting flanges. Paint the interior surfaces of the mounting flanges and the facing surfaces of the mounting plates white. Optically align the opacity monitor (transceiver and reflector for double-pass opacity monitors, or transmitter and receiver for single-pass opacity monitors) at a measurement pathlength of 3 m on an approximate east-west axis aligned with the transit of the sun. Use a pathlength correction factor of 1.0. Calibrate the instrument using external attenuators prior to the test. Insert an external attenuator with a single-pass value between 10 and 20 % opacity into the measurement path and record the response. Initiate a calibration check cycle and record the low level and upscale responses.

6.6.3.3 Use a cosine corrected total solar radiation monitor that (*1*) is capable of detecting light from 400 to 1100 nm, (*2*) has been calibrated under natural daylight conditions to within ±5 % against industry standards, (*3*) has a sensitivity of at least 90 μA/100 W/m ^{2}, and (*4*) has a linearity with a maximum deviation of less than 1 % up to 3000 W/m^2. Place the solar radiation monitor on top of the transceiver for double-pass opacity monitors, or detector for single-pass opacity monitors. If weather covers are supplied with all opacity monitors, install the solar radiation monitor on top of the weather cover. Measure the total solar radiation according to the manufacturer's instructions.

6.6.3.4 Continuously record the opacity monitor response to the attenuator and the output of the solar radiation monitor for a period from two hours before sunrise to two hours after sunset. Record the ambient temperature during this period. Do not conduct calibration check cycles during this test more frequently than once per 24-h period or the longest interval recommended in the manufacturer's published specifications. Document and report the frequency of conducting calibration check cycles during the insensitivity to ambient light test.

6.6.3.5 If necessary, correct the measurement data for changes in instrument response due to ambient temperature variation by running a separate test with the same instrument shielded from the sunlight. Determine the maximum percent deviation in the measurement response for any six minute period during the test.

6.6.3.6 Determine conformance with the specifications 6.6.2.

6.7 *External Audit Filter Access*:

NOTE 15—The opacity monitor design must accommodate independent assessments of the measurement system response to commercially available external (that is, not intrinsic to the instrument) audit filters. These calibration attenuators may be placed within the mounting flange, air purge plenum, or other location after the projected light beam passes through the last optical surface of the transceiver or transmitter. They may also be placed in a similar location at the other end of the measurement path prior to the light beam reaching the first optical surface of the reflector or receiver. The external audit filter access design must ensure (*a*) the filters are used in conjunction with a zero condition based on the same energy level, or within 5 % of the energy reaching the detector under actual clear path conditions, (*b*) the entire beam received by the detector will pass through the attenuator, and (*c*) the attenuator is inserted in a manner that minimizes interference from the reflected light.

6.7.1 Insert the external audit filter into the system.

6.7.2 Determine whether the entire beam received by the detector passes through the attenuator and that interference from reflected light is minimal.

6.7.3 Determine whether the zero condition corresponds to the same energy level reaching the detector as when actual clear path conditions exist

6.8 *External Zero Device—Optional*:

NOTE 16—The opacity monitor design may include an external, removable device for checking the zero alignment of the transmissometer. Such a device may provide an independent means of simulating the zero opacity condition for a specific installed opacity monitor over an extended period of time and can be used by the operator to periodically verify the accuracy of the internal simulated zero device. The external zero device must be designed: (*1*) to simulate the zero opacity condition based on the same energy level reaching the detector as when actual clear path conditions exist; (*2*) to produce the same response each time it is installed on the transmissometer; and (*3*) to minimize the chance that inadvertent adjustments will affect the zero level response produced by the device. The opacity monitor operator is responsible for the proper storage and are of the external zero device and for reverifying the proper calibration of the device during all clear path zero alignment tests.

NOTE 17—The purpose of this design specification is to ensure that the external zero device design and mounting procedure will produce the same response each time that the device is installed on the transmissometer.

6.8.1 *Test Frequency*— If the optional external zero device is supplied with any opacity monitors of the subject model, select and perform this test for one representative external zero device manufactured each year for the opacity monitor model certified by this practice.

6.8.2 *Specification*— The opacity monitor output must not deviate more than ±1.0 % single pass opacity for repeated installations of the external zero device on a transmissometer.

6.8.3 *Design Specification Verification Procedure*—Perform this test using an opacity monitor that has successfully completed the tests to demonstrate insensitivity to ambient light (6.6) and which is set up and properly calibrated for a measurement pathlength of 3 meters. Install the external zero device and make any necessary adjustment to it so that it produces the proper zero opacity response from the test transmissometer. Remove the external zero device and return the test transmissometer to operation and verify that the opacity monitor output indicates 0.0 ± 0.5 % opacity. Without making any adjustments to the external zero device or the test opacity monitor, install and remove the external zero device five times. Record the zero response of the test opacity monitor to the external zero device and to the clear path condition after it is returned to operation after each installation.

6.8.4 Determine conformance with the design specification in 6.8.3.

6.9 *Calibration Check Devices*:

NOTE 18—Opacity monitors covered by this practice must include automated mechanisms to provide calibration checks of the installed opacity monitor.

6.9.1 *Simulated Zero Device*—Establish the proper response to the simulated zero device under clear path conditions while the transmissometer is optically aligned at the installation pathlength and accurately calibrated. Certify that the simulated zero device conforms to the following:

6.9.1.1 The simulated zero device produces a simulated clear path condition or low level opacity condition, where the energy reaching the detector is between 90 and 190 % of the energy reaching the detector under actual clear path conditions. Corrections for energy levels other than 100 % are permitted provided that they do not interfere with the instrument's ability to measure opacity accurately.

6.9.1.2 The simulated zero device provides a check of all active analyzer internal optics with power or curvature, all active electronic circuitry including the light source and photodetector assembly, and electric or electro-mechanical systems, and hardware and/or software used during normal measurement operation.

NOTE 19—The simulated zero device allows the zero drift to be determined while the instrument is installed on the stack or duct. Simulated zero checks, however, do not necessarily assess the optical alignment, status of the reflector (for double-pass systems), or the level of dust contamination of all optical surfaces.

6.9.2 *Upscale Calibration Device*—Certify that the device conforms to the following:

6.9.2.1 The upscale calibration device measures the upscale calibration drift under the same optical, electronic, software, and mechanical components as are included in the simulated zero check.

6.9.2.2 The upscale calibration device checks the pathlength corrected measurement system response where the energy level reaching the detector is between the energy levels corresponding to 10 % opacity and the highest level filter used to determine calibration error.

6.9.2.3 The upscale calibration check response is not altered by electronic hardware or software modification during the calibration cycle and is representative of the gains and offsets applied to normal effluent opacity measurements.

NOTE 20—The upscale calibration device may employ a neutral density filter or reduced reflectance device to produce an upscale drift check of the measurement system. The upscale calibration device may be used in conjunction with the simulated zero device (for example, neutral density filter superimposed on simulated zero reflector) or in a parallel fashion (for example, zero and upscale [reduced reflectance] devices applied to the light beam sequentially).

6.10 *Status Indicators*:

NOTE 21—Opacity monitors must include alarms or fault condition warnings to facilitate proper operation and maintenance of the opacity monitor. Such alarms or fault condition warnings may include lamp/source failure, purge air blower failure, excessive zero or calibration drift, excessive zero or dust compensation, and so forth.

6.10.1 Specify the conditions under which the alarms or fault condition warnings are activated.

6.10.2 Verify the conditions of activations in 6.10.1 on an annual basis.

6.10.3 Certify the that the system's visual indications, or audible alarms, as well as electrical outputs can be recorded as part of the opacity data record and automatically indicate when either of the following conditions are detected:

6.10.3.1 A failure of a sub-system or component which can be reasonably expected to invalidate the opacity measurement, or

6.10.3.2 A degradation of a subsystem or component which requires maintenance to preclude resulting failure.

6.11 *Pathlength Correction Factor (PLCF) Security*:

NOTE 22—The opacity monitoring system must display and record all measured opacity values (including effluent opacity measurements, zero and upscale calibration checks, and zero or dust compensation values) corrected to the emission outlet pathlength.

6.11.1 Certify that the system has been designed and constructed so that the value of the pathlength correction factor

6.11.1.1 Cannot be changed by the end user, or

6.11.1.2 Is recorded during each calibration check cycle, or

6.11.1.3 The system must provides an alarm when the value is changed from the certified value.

6.11.2 Document the option(s) that are selected and write corresponding instructions. Provide them to the end user to minimize the likelihood that the PLCF will be changed inadvertently.

6.12 *Measurement Output Resolution*:

6.12.1 Certify that the opacity monitor output, including visual measurement displays, analog outputs, or digital outputs, or combinations thereof, have a resolution ≤0.5 % opacity over the measurement range from –4.0 % opacity to 50 % opacity or higher value.

NOTE 23—The 0.5 % opacity resolution is required for determining calibration error or achieving conformance with applicable regulatory requirements.

6.13 *Measurement and Recording Frequency*:

6.13.1 Certify that each opacity monitor is designed and constructed to do the following:

6.13.1.1 To complete a minimum of one cycle of sampling and analyzing for each successive 10-s period.

6.13.1.2 To calculate average opacity values from 6 or more data points equally spaced over each 1-min period included in the average (for example, 6 measurements per 1-min average or 36 measurements per 6-min average),

6.13.1.3 To record values for each averaging period.

NOTE 24—Most regulations require recording of six-min average opacity values, however, some regulatory agencies require calculation of one-minute or other less than 6-min average values.

7. Procedure—Manufacturer's Performance Specifications

7.1 *Required Performance Tests*—Test each instrument prior to shipment to ensure that the opacity monitor meets manufacturer's performance specifications for instrument response time, calibration error, and optical alignment sight performance. Conduct a performance check of the spectral response for each instrument.

NOTE 25—These tests are performed for the specific transmissometer components (transceiver and reflector for double-pass opacity monitors or transmitter and receiver for single-pass opacity monitors), the specific control unit (if included in the installation), and any other measurement system components that are supplied by the manufacturer. The data recording system that will be employed by the end user is not required to be evaluated by these tests. Additional field tests are necessary to evaluate the complete opacity monitoring system after it is installed at the end user's facility. The field test procedures may be simplified when certain conditions are met in the conduct of the manufacturer's performance specification tests.

7.2 *Representative Test Conditions*:

7.2.1 Conduct the manufacturer's performance specification tests under conditions that are representative of the specific intended installation, whenever possible. Obtain from the end user accurate information about the installation pathlength (that is, flange-to-flange separation distance), monitoring pathlength, emission outlet pathlength, and the applicable opacity standard. Use the applicable opacity standard, monitoring pathlength, and emission outlet pathlength to select appropriate attenuators for the calibration error test and to establish the pathlength correction factor for the opacity monitor being tested. Set-up and test the transmissometer components at the same installation pathlength and the same pathlength correction factor as that of the field installation.

NOTE 26—When these conditions are met, the equivalent *clear path* setting for an external zero device can be established in conjunction with the manufacturer's calibration error test. This device can then be used in subsequent field calibration error tests to verify performance of the opacity monitor. If both the actual installation pathlength and the pathlength correction factors are within ±10 % of the values used for the manufacturer's calibration error test, the manufacturer's calibration error test results are valid and representative for the installation.

7.2.2 If actual pathlength values differ by >2 %, but ≤10 % relative to that used for the manufacturer's calibration error test, repeat the zero alignment (for installation pathlength errors) or reset the pathlength correction factor (for pathlength correction errors) prior to subsequent opacity monitoring.

NOTE 27—A field performance audit may be substituted for the field calibration error test when the above criteria are satisfied.

7.2.3 If the actual installation pathlength and pathlength correction factors exceed ±10 % of the values used for the manufacturer's calibration error test, repeat the calibration error test.

7.3 *Default Test Conditions*—If the installation pathlength, monitoring pathlength, and emission outlet pathlength cannot be determined by the manufacturer (for example, opacity monitor is intended for future resale, opacity monitor will serve as backup for multiple installations, construction of facility is not complete and so forth), test the opacity monitor at an installation pathlength of 5 m and use a pathlength correction factor of 1.0. If an opacity monitor is designed for a range of measurement pathlengths that does not include 5 m, test the opacity monitor at the middle of the range specified by the manufacturer (see example in Note 28). If the applicable opacity standard cannot be determined, assume a standard of 20 % opacity for the selection of attenuators used for the calibration error test.

NOTE 28—Example: If an opacity monitor is designed for measurement pathlengths from 6 to 12 m, use a pathlength of 9 m.

7.4 *Test Set-Up*—Conduct the performance tests of the opacity monitor in a clean environment in an area protected from manufacturing or other activities that create dust, mist, fumes, smoke, or any other ambient condition that will interfere with establishing a clear path opacity condition.

7.4.1 Set-up the transmissometer components on test stands that will facilitate adjustments to, and maintenance of, the optical alignment throughout the test procedure.

7.4.2 Use the appropriate installation pathlength as determined from 7.2, if possible, or 7.3, if necessary.

7.4.3 Adjust the focus of the transmissometer for the installation pathlength, if applicable.

7.4.4 Optically align the transmissometer components according to the written procedures of the manufacturer.

7.4.5 Verify that the alignment sight indicates proper alignment.

7.4.6 Enter the proper pathlength correction factor (if applicable) for the opacity monitor.

7.4.7 Establish proper calibration of the measurement system according to the manufacturer's written procedures.

7.4.8 Connect the opacity monitor to an appropriate data recorder for documenting the performance test results. At a minimum, use a data recorder that

7.4.8.1 Is capable of resolving 0.25 % opacity,

7.4.8.2 Has been accurately calibrated and verified according to the manufacturer's QA procedures, and

7.4.8.3 Has a sufficiently fast response to measure the instrument response time.

7.5 *Selection of Calibration Attenuators*—Using the applicable pathlength correction factor and opacity standard values from 7.2 (if possible) or 7.3 (if necessary), select calibration attenuators that will provide an opacity monitor response corrected to single-pass opacity values for the emission outlet pathlength in accordance with the following:

Applicable Standard	10 to 19 % opacity	≥20 % opacity
Low level:	5 to 10 %	10 to 20 %
Mid level:	10 to 20 %	20 to 30 %
High level:	20 to 40 %	30 to 60 %

NOTE 29—The manufacturer may elect to use additional calibration attenuators in the calibration error test. The use of additional calibration attenuators may be advantageous in demonstrating the linear range of the measurement system. Alternate calibration attenuator values may be used where required by applicable regulatory requirements (for example, state or local regulations, permit requirements, and so forth).

7.6 *Attenuator Calibrations*—Calibrate the attenuators used for the manufacturer's calibration at the frequency and according to the procedures specified in 40 CFR 60, Appendix B, Performance Specification 1, 7.1.3. For transmissometers operating over narrow bandwidths, determine the attenuator calibration values for the actual operating wavelengths of the transmissometer.

7.7 *Instrument Response Time*:

NOTE 30—The purpose of the instrument response time test is to demonstrate that the instantaneous output of the opacity monitor is capable of tracking rapid changes in effluent opacity, using the instantaneous output or signal input used to generate averages. It includes the transmissometer components and the control unit if one is included for the particular installation. The instrument response time test does not include the opacity monitoring system permanent data recorder. (A separate field test should be conducted to verify the ability of the system to properly average or integrate and record 6-min opacity values.)

7.7.1 *Specification*— The instrument response time must be less than or equal to 10 s.

7.7.2 *Instrument Response Time Test Procedure* —Using a high-level calibration attenuator, alternately insert the filter five times and remove it from the transmissometer light path.

7.7.2.1 For each filter insertion and removal, determine the amount of time required for the opacity monitor to display 95 % of the step change in opacity on the data recorder used for the test. For upscale response time, determine the time it takes to reach 95 % of the final, steady upscale reading. For downscale response time, determine the time it takes for the display reading to fall to 5 % of the initial upscale opacity reading.

7.7.2.2 Calculate the mean of the five upscale response time measurements and the mean of the five downscale response time measurements. Report each of the scale and downscale response time determinations and the mean upscale and downscale response times.

7.7.3 Determine conformance with the specification in 7.7.1. If the response time is not acceptable, take corrective action and repeat the test.

7.8 *Calibration Error*:

NOTE 31—The calibration error test is performed to demonstrate that the opacity monitor is properly calibrated and can provide accurate and precise measurements.

7.8.1 *Specification*— The calibration error must be ≤3 % opacity as calculated ad the sum of the absolute value of the mean difference and confidence coefficient for each of three test attenuators.

7.8.2 *Calibration Error Test Procedure*:

7.8.2.1 Zero the instrument. Insert the calibration attenuators (low-, mid- and high-level) into the light path between the transceiver and reflector (or transmitter and receiver).

7.8.2.2 While inserting the attenuator, ensure that the entire beam received by the detector passes through the attenuator and insert the attenuator in a manner that minimizes interference from the reflected light.

NOTE 32—See also Note 15. The placement and removal of the attenuator must be such that measurement of opacity is performed over a sufficient period to obtain a stable response from the opacity monitor.

7.8.2.3 Make a total of five non-consecutive readings for each filter. Record the opacity monitoring system output readings in single-pass percent opacity.

7.8.2.4 Subtract the single-pass calibration attenuator values corrected to the stack exit conditions from the opacity monitor responses. Calculate the arithmetic mean difference, standard deviation, and confidence coefficient of the five measurements value. Calculate the calibration error as the sum of the absolute value of the mean difference and the 95 % confidence coefficient for each of the three test attenuators. Report the calibration error test results for each of the three attenuators.

$$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i \qquad (4)$$

where:
x = arithmetic mean,
x_i = individual measurements, and
n = number of data points.

$$S_d = \sqrt{\frac{\sum_{i=l}^{n} x_i^2 - \frac{(\sum_{i=l}^{n} x_i)^2}{n}}{n-1}} \qquad (5)$$

where:
s_d = standard deviation.

$$CC = t_{0.975}\frac{S_d}{\sqrt{n}} \qquad (6)$$

where:
$t_{0.975}$ = t-value ($t_{0.975} = 2.776$ for $n = 5$), and
CC = confidence coefficient

7.8.2.5 Determine conformance with the specification in 7.8.1. If the calibration error test results are not acceptable, take corrective action, recalibrate the opacity monitor according to the manufacturer's written instructions, and repeat the calibration error test.

7.9 *Optical Alignment Indicator – (Uniformity of Light Beam and Detector)*:

NOTE 33—Each transmissometer must provide a means for visually determining that the instrument is optically aligned. The purpose of this specification is to ensure that the alignment device is capable of clearly indicating when the transmissometer components are misaligned. The performance test procedure will also detect opacity monitors where the accuracy of opacity measurements is adversely affected by the use of the light beams having non-uniform intensity, or the use of non-uniform detectors, or inefficient or poor quality retro-reflector material.

7.9.1 *Specification*— The alignment sight must clearly indicate that the unit is misaligned when an error of ±2 % single-pass opacity occurs due to shifts in the optical alignment of the transmissometer components. For opacity monitor designs that include automatic beam steering (that is beam position sensing and an active means for adjusting alignment so that centered alignment is maintained even with slowly changing misalignment conditions), an alarm must be activated when the alignment is varied beyond the manufacturer's specified range of angular tolerance is unable to maintain alignment.

NOTE 34—Modifications of the alignment indicator test procedures for systems with beam steering are included in 7.9.7.

7.9.2 *Alignment Indicator Performance Test Procedure*—Conduct the alignment indicator test according to the procedures in 7.9.3-7.9.7.

NOTE 35—The test procedure can be modified to accommodate moving of either component of the transmissometer to achieve equivalent geometric misalignment as described in 7.9.3-7.9.6. Alignments tests may be performed in the horizontal or vertical planes of the instrument and the instrument components may be turned on their side to accommodate the tests.

7.9.3 *Set-up*:

7.9.3.1 Set up the transmissometer on test stands that allow adjustments for the rotational and translational misalignment tests.

7.9.3.2 Optically align the transceiver and reflector (double-pass opacity monitor) or transmitter and receiver (single-pass opacity monitor) according to the manufacturer's written instructions. Verify that all alignment indicator devices show proper alignment.

7.9.3.3 Conduct the alignment indicator performance test with a clear path condition. Alternatively, insert an external attenuator that produces a response ≤10 % single-pass opacity into the measurement path turn it approximately 3° from normal to the light path to eliminate surface reflection, and record the indicated opacity.

7.9.4 *Case 1: Single and Double Pass Opacity Monitors*:

7.9.4.1 Slowly tilt the transceiver (double-pass opacity monitor) or transmitter (single-pass opacity monitor) upward in the vertical plane (for example, adjust the appropriate alignment bolts or mounting mechanism) relative to the reflector (double-pass opacity monitor) or receiver (single-pass opacity monitor) until an error of ±2 % opacity is first indicated on the data recorder. Verify that the alignment indicator shows misalignment.

7.9.4.2 Illustrate and record the alignment indicator and the degree of misalignment shown.

7.9.4.3 Return the system to its aligned condition.

7.9.4.4 Repeat the entire procedure by tilting the transceiver in the opposite (downward) direction.

7.9.4.5 Repeat the rotational misalignment check of the transceiver or transmitter in the horizontal plane (both to the left and right) and again illustrate and record the visual depiction of the alignment for each step of the procedure.

7.9.5 *Case 2, Single-Pass Opacity Monitors Only*

7.9.5.1 Slowly tilt the receiver in the vertical plane (for example, adjust the appropriate alignment bolts or mounting mechanism) until an error of ±2 % opacity is first indicated on the data recorder, Verify that the alignment indicator shows misalignment.

7.9.5.2 Illustrate and record the alignment indicator and the degree of misalignment shown.

7.9.5.3 Return the system to its aligned condition and again draw the alignment indicator.

7.9.5.4 Repeat the entire procedure by tilting the receiver in the opposite direction.

7.9.5.5 Repeat the rotational misalignment check of the receiver in the horizontal plane (both to the left and right) and again illustrate and record the visual depiction of the alignment for each step of the procedure.

7.9.6 *Case 3: Single and Double Pass Opacity Monitors*:

7.9.6.1 Achieve lateral misalignment of the transceiver or transmitter relative to the reflector or receiver by slowly moving either assembly linearly to the left until a positive or negative error of ±2 % opacity is first indicated on the data recorder. Verify that the alignment indicator shows lateral misalignment of the transceiver or transmitter relative to reflector or receiver.

7.9.6.2 Illustrate and record the alignment indicator and the degree of misalignment shown.

7.9.6.3 Return the system to its aligned condition and again draw the alignment indicator.

7.9.6.4 Repeat the entire procedure by moving the same component to the right.

7.9.6.5 Repeat the test in the vertical plane (both above and below the aligned position) and again illustrate and record the visual depiction of the alignment for each step of the procedure.

7.9.7 *Assessment*—Determine conformance with the specification in 7.9.1.

NOTE 36—The performance of the alignment indicator is acceptable if: (*1*) for each case of rotational or translational misalignment, misalignment is clearly shown when an error of ±2 % single-pass opacity first occurs in each direction, and (2) proper alignment status is consistently indicated when the opacity monitor is optically aligned. A clear indication of misalignment is one that is objectively apparent relative to reference marks or conditions; an alignment device that requires a subjective judgement of the degree of misalignment is not acceptable.

7.9.8 *Automatic Beam Steering*:

7.9.8.1 If the design includes automatic beam steering, investigate each case of rotational and translational misalignment. Vary the alignment over the manufacturer's specified range of angular tolerance for which the alignment is maintained and for which the opacity is maintained within ±2 % single pass opacity.

7.9.8.2 During each misalignment test, record the angular misalignment where the alarm is activated.

7.9.8.3 Determine conformance with the specification in 7.9.2.

NOTE 37—Acceptable performance is indicated if (*1*) the alarm is activated or misalignment is clearly show at, or before, the angular tolerance specified by the manufacturer is reached, and (*2*) for translational misalignment, an alarm is activated or misalignment is clearly shown at, or before, an error of ±2 % single-pass opacity first occurs in each direction.

7.10 *Special Response Performance Check*:

NOTE 38—This performance check provides a simple method to ensure that the special response of each instrument satisfies the spectral response design specifications of 6.2. The performance check uses a photopic transmission filter placed in the measurement beam and comparison with a range of expected responses determined from Monitor-Specific Performance Check Limits (6.2.7).

7.10.1 *Specification*— The transmissometer response to the photopic transmission filter used to establish performance check limits, after correction of the response to account for the applicable pathlength correction factor, must be within ±2 % opacity of the range defined by the maximum and minimum responses (determined in 6.2.7 in units of percent opacity.

7.10.2 *Spectral Response Performance Check Procedure*:

7.10.2.1 Insert the photopic transmission filter used to establish the performance check limits into the opacity monitor measurement beam after the calibration error test has been completed. Record the opacity monitor response to the photopic filter in units of percent opacity.

7.10.2.2 Correct the opacity monitor response to the equivalent value corresponding to a pathlength correction factor of 1.

7.10.2.3 Compare the corrected response to the acceptable limits and determine conformance with the specification in 7.10.1.

7.10.2.4 If an unacceptable result is obtained, do not assume that the spectral response design requirements are met. Investigate the causes for an unacceptable result. Unless a clear explanation of the problem is apparent, repeat Spectral Response Design Specifications Verification Procedure (6.2.3) and recalibrate the photopic filter.

7.11 *Calibration Device Value and Repeatability*:

NOTE 39—The purpose of this specification is to verify that the upscale calibration device response is repeatable and provides results consistent with the use of external filters. This specification may also be applied to additional internal calibration check devices (values), if provided in the instrument.

7.11.1 *Specification*— The 95 % confidence coefficient for repeated measurements of the upscale calibration device must be less than 1.5 % opacity. The upscale calibration device must be assigned a value relative to the calibration error test results for the specific opacity monitor.

7.11.2 *Calibration Device Repeatability Test Procedure*— Perform this procedure immediately after successfully completing the calibration error test for the opacity monitor. Do not make any adjustments to the opacity monitor until after this procedure has been completed. Make seven non-consecutive measurements of each internal device and record the opacity monitor responses.

7.11.2.1 Calculate the 95 % confidence coefficient using the same procedures as that used in the calibration error test. (See 7.8.2.4).

7.11.2.2 Determine conformance to the specification in 7.11.1.

7.11.2.3 Assign values to the upscale calibration device(s) relative to the calibration error test results. Construct a calibration curve by linear regression analysis through zero of the results of the calibration error test (x-axis are correct values and y-axis are the corresponding opacity monitor responses). Using the mean of the five measurement responses for each upscale calibration device as the y-axis value, determine in corresponding x-axis value from the calibration curve. Assign this value to the internal upscale calibration device.

8. Quality Assurance Guidelines for Opacity monitor Manufacturers

8.1 *General*—The products shall be manufactured under a quality program that ensures that like products, subsequently made, have the same reliability and quality as those originally examined to determine compliance with this design specification. To establish and maintain such a program, the manufacturer shall be guided by industry practice, its quality controls, and by this set of guidelines. These guidelines are supported by various standards and by industry practice.

8.1.1 *Applicable Documents*—This document is an adaptation of and the referred to the following standards for additional guidance:

ISO/DIS 9004 Quality Management and Quality System Elements-Guidelines

ANSI/NCSL Z 540-1-1994, Calibration Laboratories and Measuring Equipment - General Requirements

8.1.2 *General Vocabulary*—Terms used in this document are defined by:

ISO 842, Quality Vocabulary

8.2 *Quality System*:

8.2.1 *Management Responsibility*:

8.2.1.1 *Quality Policy*— The management of a company shall develop and promulgate a corporate quality policy. Management shall ensure that the corporate policy is understood, implemented and maintained.

8.2.1.2 *Quality Objectives*—Based on this policy, key quality objectives shall be defined, such as fitness for use, performance, reliability, safety, and so forth.

8.2.1.3 *Quality Management Systems*—A documented system shall be developed, established and implemented for the product as a means by which stated quality policies and objectives can be realized. The quality system should ensure that: (*1*) it is understood and effective; (2) products actually do satisfy customer expectations; (*3*) emphasis is placed on problem prevention rather than dependence on detection after occurrence; (*4*) causes, not only symptoms, of a problem are found, and that corrections are comprehensive, touching any activity that has a bearing on quality; and (*5*) feedback is generated that can be used at the product or process design stage for correcting problems and improving product. Management shall provide the resources essential to the implementation of quality policies and objectives.

8.2.2 *Quality System Documentation and Records*—The elements requirements and provisions adopted for the quality management system shall be documented in a systematic and orderly manner.

8.2.2.1 Documentation shall be legible, clean, readily, identifiable and maintained in an orderly manner.

8.2.2.2 The quality management system shall establish and require the means for identification, collection, filing, storage, maintenance, retrieval and disposition of pertinent quality documentation and records. Methods shall be established for making changes, modifications, revisions or additions to the contents of applicable documentation in a controlled manner.

8.3 *Corrective Action Program*:

8.3.1 *Introduction*— There shall be a comprehensive defect analysis/corrective action program for reporting and following-up on product and program deficiencies.

8.3.2 *Assignment of Responsibility*:

8.3.2.1 The responsibility and authority for instituting corrective action shall be defined as part of the quality system.

8.3.2.2 The coordination, recording and monitoring of corrective action shall be assigned to a specific person or group within the organization. (The analysis and execution of any corrective action may involve a variety of people from such areas as sales, design, production engineering, production and/or quality control.)

8.3.3 *Deficiencies*:

8.3.3.1 Deficiencies shall be evaluated in terms of their potential impact on product quality, reliability, safety, performance and customer satisfaction.

8.3.3.2 The relationship between cause and effect should be determined. The root cause should be determined before planning and implementing corrective measures. Careful analysis shall be given to the product and all related processes, operations, records, and so forth.

8.3.3.3 Controls of processes and procedures shall be implemented to prevent recurrence of the problem. When the corrective measures are implemented, their effect shall be monitored in order to ensure desired goals are met.

8.3.3.4 Permanent changes resulting from corrective action shall be incorporated into the work instructions, manufacturing processes, product specifications and/or the quality manual.

8.4 *Quality System Certification*—Companies with ISO 9001/9002 certification, companies meeting the requirements of ANSI/ASQC Q90 (Q91 or Q92), companies meeting the requirements of nationally recognized test laboratories (NRTLs) where the manufactured product bears the mark (or marks) of the NRTL(s), or companies with an equivalent independently and periodically verified quality system, and which adopt this specification as part of their product definition shall be deemed to meet all of the above quality assurance guidelines. Companies meeting these conditions shall attach the applicable certification to the manufacturer's certification of conformance report as proof of such designation.

9. Report

9.1 Summarize the design and performance (see Table 1, Table 2 and Table 3) data in the report. See Fig XI.1 for an example.

9.2 Include all descriptive information, such as:

9.2.1 Manufacturer or supplier information,

9.2.2 Opacity monitor information,

9.2.3 User information, and

9.2.4 Installation information.

10. Keywords

10.1 continuous opacity monitor; design specification; performance specification; transmissometer

APPENDIX

(Nonmandatory Information)

X1. DATA SUMMARY FORM

X1.1 Fig. X1.1 is an example data form to summarize data to certify conformance with design and performance specifications.

This document is provided by (company name), an (original manufacturer, supplier, remanufacturer, or service facility), of/for opacity monitoring systems that are intended to comply with standards of performance established by the US EPA 40CFR60, Appendix B, Performance Specification 1, Performance Specifications for Opacity Monitors. This EPA specification references the above ASTM Standard Practice, which may be used by the manufacturer or supplier to demonstrate that the designated opacity monitor meets those performance requirements that can be tested and verified by the supplier prior to field installation. Data in this summary document (Part I) have been generated in compliance with the procedures and specifications shown in the ASTM Standard Practice, SPXXXX. These data confirm that the designated opacity monitor meets or exceeds the requirements of this Standard Practice.

I. MANUFACTURER/SUPPLIER INFORMATION

Company name ____________________________
Location _________________________________

II. OPACITY MONITOR INFORMATION

Model______________________
Transceiver type ______________
Transceiver serial no. __________
Reflector type ________________
Reflector serial no. ___________
Control unit serial no. __________
Software version no. ___________

III. USER INFORMATION

Company _____________________________
Plant ___________________________________
Process/boiler __________________________
Location ________________________________

IV. INSTALLATION INFORMATION

Monitoring pathlength (depth of effluent) ________
Installation pathlength (flange to flange) _________
Emission outlet pathlength (stack exit) __________
PLCF or (OPLR) ________
Facility opacity standard, % opacity ___________

FIG. X1.1 Data Summary Form

PART I—DESIGN AND PERFORMANCE SPECIFICATIONS-TESTED AT MANUFACTURER'S FACILITY

Conformance with design specifications is demonstrated by testing two separate opacity monitors, each of which is representative of standard production. One opacity monitor is selected and tested annually and the other is selected from either a production lot of instruments not to exceed 20 in size, or from monthly production, and tested in accordance with procedures described in the SPXXXX. The tests associated with each of the above selected analyzers is required to be repeated anytime there is a critical component change that is substantial, hardware or software change, or manufacturing process change that could affect performance with respect to said design specifications. The test data derived from each of the above two described analyzers is summarized below.

1. Design Specifications Verified Through Tests Prescribed For An Annually, or More Often, Selected Opacity Monitor

The opacity monitor that tested to demonstrate the following design parameters was selected on the basis of an annual selection ____ , or following a major change in the design or construction of the monitor ____.

OPACITY MONITOR INFORMATION

Model______________________
Transceiver type ______________
Transceiver serial no. ___________
Reflector type ________________
Reflector serial no. _____________
Control unit serial no. __________
Software version no. ____________

TESTS PERFORMED BY:
______________________________ DATE: ______________

TEST DATA REVIEWED AND CERTIFIED BY:
______________________________ DATE: ______________

1.1 ***Spectral Response***

Data listed below were obtained by calculation ____, or by actual measurement using a monochrometer ____. A description of either the calculations or the measurement test setup are included in Attachment A.

Parameter	**Specification**	**Actual Test Result**
Peak response	Between 500-600 nm	
Mean response	Between 500-600 nm	
Max response beyond 700 nm	Less than 10% of peak	
Max response less than 400 nm	Less than 10% of peak	

FIG. X1.1 Data Summary Form *(continued)*

1.2 *Thermal Stability*

Parameter	Specification	Actual Test Result
Tested range, min temp	Mfgr specified, °C (°F)	
Tested range, max temp	Mfgr specified, °C (°F)	
Nominal measurement value	0-10% opacity	
Measurement drift, max deviation from nominal measurement value	≤ 2% opacity/40 °C (°F)	
Zero drift from nominal without compensation	≤ 2% opacity/40 °C (°F)	
Zero drift from nominal with compensation	≤ 2% opacity/40 °C (°F)	
Span drift from nominal without compensation	≤ 2% opacity/40 °C (°F)	
Span drift from nominal with compensation	≤ 2% opacity/40 °C (°F)	

1.3 *Insensitivity to Ambient Light*

Parameter	Specification	Actual Test Result
Max solar intensity	900 W/m^2	
Nominal measurement value	0-10% opacity	
Measurement drift, max deviation from nominal measurement value	≤ 2% opacity	
Drift was corrected for thermal effects, yes or no	Mfgr specified	
Zero drift from nominal without compensation	≤ 2% opacity	
Zero drift from nominal with compensation	≤ 2% opacity	
Span drift from nominal without compensation	≤ 2% opacity	
Span drift from nominal with compensation	≤ 2% opacity	

1.4 *Calibration Device Availability*

Parameter	Specification	Availability/Value
External zero device	Optional	
Ext zero device repeatability	≤ 1% opacity	
External filter access	To be available	

FIG. X1.1 Data Summary Form *(continued)*

1.5 *Zero/upscale calibration check apparatus*

Parameter	Specification	Test Result
Indicated response to simulated zero calibration device	0 ± 0.5% opacity	
Simulated Zero Check	Simulated condition during which the energy reaching the detector is between 90 and 190% of the energy reaching the detector under actual clear path conditions *	
Response to upscale calibration device without electronic hardware or software modification	+10% opacity to highest calibration error attenuator value	
Does automatic zero and span calibration devices check all active optics and electronics?	Required	
Is automatic correction provided for zero drift?	Mfgr to specify (Y/N)	
	If yes, specify freq	
Is automatic correction provided for dust accumulation on exposed optics?	Mfgr to specify (Y/N)	
	If yes, specify freq	
Is automatic correction provided for span/cal drift?	Mfgr to specify (Y/N)	
	If yes, specify freq	

Note: * Negative opacity values of this magnitude can be calculated from the detector or preamplifier output by measuring the equivalent optical energy detected in the clear path condition and that produced by the zero calibration check device.

1.6 *PLCF (OPLR) Security Precautions*

Condition	Specification	As Supplied (Y/N)
Original certified value is fixed and not adjustable by user	One or more of listed conditions to be provided	
Value is output with zero and span values during each calibration cycle	One or more of listed conditions to be provided	
Flag (alarm) is activated when changed from original certified or permanently recorded value	One or more of listed conditions to be provided	

FIG. X1.1 Data Summary Form *(continued)*

1.7 ***Faults and Alarm (Mfgr to specify)***

Fault Conditions (Note 1&3)	**Specified Indication**	**Actual Indication**
Conditions tested	Audible or visual, and electrical	Tested output
	Same	
	Same	
	Same	
Alarm Conditions (Note 2&3)	**Specified Indication**	**Actual Indication**
Conditions tested	Audible or visual, and electrical	Tested output
	Same	
	Same	
	Same	
	Same	
	Same	

Note 1) Fault conditions are those conditions which, when they occur, are deemed by the manufacturer to result in performance which is not in compliance with this performance specification.

Note 2) Alarm conditions are those conditions for which the manufacturer recommends review and/or corrective action by trained service personnel as appropriate to prevent further deterioration of instrument performance which could result in performance not in compliance with this specification.

Note 3) Manufacturer may use other nomenclature to designate either general or specific alarms and/or faults, as long as they are appropriately defined in the operators manual.

1.8 ***Miscellaneous***

Parameter	**Specification**	**Test Result**
Resolution of visual measurement indication, if provided	≤ 0.5% opacity	
Resolution of analog output measurement indication	≤ 0.5% opacity	
Resolution of serial digital output, if provided	≤ 0.5% opacity	
Bipolar range of visual measurement indication	+50% opacity or more to -4% opacity or less	
Capability of analog output measurement indication to indicate negative values to at least -4% opacity	Required	
Are means available to monitor daily zero and span drift before correction?	Optional	
Is span drift corrected for zero drift in above method?	Optional	
Are means available to monitor dust accumulation on exposed optical surfaces?	Optional	

FIG. X1.1 Data Summary Form *(continued)*

What surfaces are monitored for dust accumulation?	Mfgr to specify, if applicable	
Is an alarm provided for excessive dust accumulation?	Mfgr to specify, if automatic correction is provided	
What level of dust accumulation triggers the above alarm?	Mfgr to specify, if applicable	
Is dust level measured separately from the accumulative zero drift?	Mfgr to specify	
Are all dust (if provided), zero, and span values corrected to stack exit conditions?	Required	
What is the normal update interval for opacity measurements?	10 sec max	
Do longer term opacity averages include at least 6 approximately equaly distributed individual measurement values per minute?	Required	

FIG. X1.1 Data Summary Form *(continued)*

2. Design Specifications Verified Through Tests Prescribed for an Opacity Monitor Selected on a Monthly Basis, or From Each Production Lot

The opacity monitor that was selected and tested to demonstrate the following design parameters was selected on the basis of a monthly selection ____, a manufacturing lot not to exceed 20 in size ____, or following a major change in the design or construction of the monitor ____.

OPACITY MONITOR INFORMATION
Model ____________________
Transceiver type ______________
Transceiver serial no. ___________
Reflector type _______________
Reflector serial no. ___________
Control unit serial no. __________
Software version no. ___________

TESTS PERFORMED BY:
______________________________ DATE: _______________

TEST DATA REVIEWED AND CERTIFIED BY:
______________________________ DATE: _______________

2.1 *Angle of View*

Transmissometer is exempt from angle of view specification because the angle of projection has been demonstrated to be less than______degrees.

Portion of opacity monitor included in the test: ________________________
____

Portion of opacity monitor excluded in the test: ________________________
____

Light source used in the test: directional _____, or non-directional _____
Were detector/measurement electronics modified to measure response to designated light source: (Y/N) _______. If so, describe modifications in attachment B.

Parameter	**Specification**	**Actual Test Result**
Angle of view, horizontal	≤ 4° for all radiation providing a response of ≥ 2.5% of peak response	
Angle of view, vertical	≤ 4° for all radiation providing a response of ≥ 2.5% of peak response	

FIG. X1.1 Data Summary Form *(continued)*

2.2 *Angle of Projection*

Transmissometer is exempt from angle of projection specification because the angle of projection has been demonstrated to be less than______degrees.

Option 1 Procedure
Portion of opacity monitor included in the test:____________________________

Portion of opacity monitor excluded in the test: ___________________________

Photodetector used in test: ____

____ If tests were conducted with dc coupled measurement circuit, was ambient light level less than 0.5% of peak light intensity? (Y/N) _______

____ If tests were conducted with ac coupled measurement circuit, was it demonstrated that ambient light levels did not saturate the detector? (Y/N) ______; and was it demonstrated that turning on/off ambient lights did not affect measurements? (Y/N) ______.

Parameter	**Specification**	**Actual Test Result**
Angle of projection, horizontal	≤ 4° for all radiation providing a response of ≥ 2.5% of peak response	
Angle of projection, vertical	≤ 4° for all radiation providing a response of ≥ 2.5% of peak response	

Option 2 Procedure (For transmissometer designs that have previously met the AOP specification using Option 1 procedure during the preceding 12 months)
Distance from transceiver/transmitter to target _____________
Beam dimension (diameter) in the vertical direction ___________
Beam dimension (diameter) in the horizontal direction __________
Option 2 Result: (total subtended angle):___________ degrees.
Option 1 Result (angle of projection): _____________ degrees
Difference (Option 1 result minus Option 2 result) ___________ degrees. If the results do not agree within 0.3 degrees, repeat the test using Option 1.

FIG. X1.1 Data Summary Form *(continued)*

2.3 ***Insensitivity to supply voltage variations***

Manufacturers specified nominal voltage: ___________
Manufacturers specified operating voltage range, if specified: ____________________

Parameter	**Specification**	**Actual Test Value**
Min test voltage	-10% from nom, or mfgrs min specified operating voltage, whichever is lesser	
Max test voltage	+10% from nom, or mfgrs max specified operating voltage, whichever is greater	
Nominal measurement value	0-10% opacity	
Measurement drift, max deviation from nominal measurement value from nominal to max ac voltage	± 1% opacity	
Measurement drift, max deviation from nominal measurement value from nominal to min ac voltage	± 1% opacity	
Zero drift from nominal to min ac voltage without compensation	± 1% opacity	
Zero drift from nominal to min ac voltage with compensation	± 1% opacity	
Span drift from nominal to min ac voltage without compensation	± 1% opacity	
Span drift from nominal to min ac voltage with compensation	± 1% opacity	
Zero drift from nominal to max ac voltage without compensation	± 1% opacity	
Zero drift from nominal to max ac voltage with compensation	± 1% opacity	
Span drift from nominal to max ac voltage without compensation	± 1% opacity	
Span drift from nominal to max ac voltage with compensation	± 1% opacity	

FIG. X1.1 Data Summary Form *(continued)*

3. Performance Specifications Verified by Tests Prescribed for Each Specific Opacity Monitor.

The following tests were performed individually on the specific instrument described in the beginning of this test report. Further, the following signatures attest to the fact that the design and performance specifications tested on previous opacity monitors, as described in Sections 1 and 2, are representative of the design and performance of this specific monitor.

TESTS PERFORMED BY:

_________________________________ DATE: _________________

TEST DATA REVIEWED AND CERTIFIED BY:

_________________________________ DATE: _________________

3.1 *Calibration error*

Filter	Specify Group, Group I or II	Actual Filter Value	Specified Cal Error	Actual Cal Error
Low	Group		3%	
Mid	Group		3%	
High	Group		3%	

Note: Group I filters are 5-10, 10-20, 20-40 percent opacity (low, mid, high)
Group II filters are 10-20, 20-30, 30-60 percent opacity (low, mid, high)

3.2 *Misalignment indication*

This opacity monitor uses (a) manual alignment and visual alignment sighting device (Y/N) ____, or b) automatic beam steering (Y/N) ______.

3.2.1 *For manually aligned opacity monitors with visual alignment sighting indicator:*

A. Rotational misalignment

Parameter	Specification	Test Result
Nominal measurement value	0-10% opacity	
Indication of centered alignment	Acceptable? (Y/N)	
Clear indication of misalignment for rotational misalignment for transceiver/transmitter in upward vertical direction which causes 2% opacity change	Acceptable? (Y/N)	
Clear indication of misalignment for rotational misalignment for transceiver/transmitter in downward vertical direction which causes 2% opacity change	Acceptable? (Y/N)	
Clear indication of misalignment for rotational misalignment for transceiver/transmitter in horizontal right direction which causes 2% opacity change	Acceptable? (Y/N)	
Clear indication of misalignment for rotational	Acceptable? (Y/N)	

FIG. X1.1 Data Summary Form *(continued)*

misalignment for transceiver/transmitter in horizontal left direction which causes 2% opacity change		

B. Lateral misalignment, same test conditions

Parameter	**Specification**	**Test Result**
Clear indication of misalignment for lateral movement to the left which causes 2% opacity change	Acceptable? (Y/N)	
Clear indication of misalignment for lateral movement to the right which causes 2% opacity change	Acceptable? (Y/N)	
Clear indication of misalignment for lateral movement in upward direction which causes 2% opacity change Clear indication of misalignment with above movement	Acceptable? (Y/N)	
Clear indication of misalignment for lateral movement in downward direction which causes 2% opacity change Clear indication of misalignment with above movement	Acceptable? (Y/N)	

3.2.2 *For opacity monitors with automatic beam steering.*

Parameter	**Specification**	**Test Result**
Nominal measurement value	0-10% opacity	
Indication of centered alignment	Acceptable? (Y/N)	
Degree of rotational misalignment for transceiver/transmitter in upward vertical direction which triggers alarm	Mfgr to specify	
Degree of rotational misalignment for transceiver/transmitter in downward vertical direction which triggers alarm	Mfgr to specify	
Degree of rotational misalignment for transceiver/transmitter in horizontal right direction which triggers alarm	Mfgr to specify	
Degree of rotational misalignment for transceiver/transmitter in horizontal left direction which triggers alarm	Mfgr to specify	

FIG. X1.1 Data Summary Form *(continued)*

Parameter	Specification	Test Result
Lateral movement of transceiver/transmitter in upward vertical direction which causes an indication of misalignment	Mfgr to specify movement	
What is maximum deviation of opacity from nom when opacity monitor is misaligned as above from centered to value noted?	≤ 2% opacity	
Lateral movement of transceiver/transmitter in downward vertical direction which causes an indication of misalignment	Mfgr to specify movement	
What is maximum deviation of opacity from nom when opacity monitor is misaligned as above from centered to value noted?	≤ 2% opacity	
Lateral movement of transceiver/transmitter in horizontal right direction which causes an indication of misalignment	Mfgr to specify movement	
What is maximum deviation of opacity from nom when opacity monitor is misaligned as above from centered to value noted?	≤ 2% opacity	
Lateral movement of transceiver/transmitter in horizontal left direction which causes an indication of misalignment	Mfgr to specify movement	
What is maximum deviation of opacity from nom when opacity monitor is misaligned as above from centered to value noted?	≤ 2% opacity	

3.3 *Spectral Response Repeatability*

Date of photopic filter calibration ____________________

Peak transmission of photopic filter ______________________

Calculated nominal response of analyzer to photopic filter ____________________, % opacity

Calculated allowable variation of the response to photopic filter:

OP, high _______________, OP, low ____________________

Above opacity values converted to stack exit values according to the specific PLCF (OPLR) established for this installation: PLCF (OPLR) ________________

OPc, nom ____________, OPc, high _______________, OPc, low _______________

Actual measured response of the instrument to listed photopic filter _____________ % opacity

Is measured response within previously calculated range (Y/N) _____________

FIG. X1.1 Data Summary Form *(continued)*

3.4 ***Intrinsic opacity monitor settings/adjustments***

List all configurable parameters to obtain the performance described in this report. These parameters typically include calibration check intervals, calibration check correction, procedure settings relating to flange-to-flange separation distance, range, averaging time, alarms, etc.

4. Quality Assurance Program

4.1 ***ISO, ANSI/ASQC, or other Quality System Certification***

Is the company which prepared this report certified according to ISO quality standards, ANSI/ASQC (QC 90 or 91) or other applicable quality standard (Y/N) ____________. If so, to what classification ________________ and on what date _______________. Attach certificate of such designation as attachment C.

4.2 ***QA Guideline Compliance***

Has the company which prepared this document established and maintained a QA/QC program that is in compliance with the guidelines specified in the ASTM SPXXXXX, (Y/N) ________. If so, please attach a description of the quality program in attachment C, and indicate the person responsible for the integrity of this quality program ___________________________________

Suppliers who comply with 4.1 and not 4.2 are required to supply all supporting test data with this report.

FIG. X1.1 Data Summary Form *(continued)*

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

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Document Name: ASTM D6228: Standard Test Method for Determination of Sulfur Compounds in Natural Gas and Gaseous Fuels by Gas Chromatography and Flame Photometric Detection

CFR Section(s): 40 CFR 60.334(h)(1)

Standards Body: American Society for Testing and Materials

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THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: D 6228 – 98

Standard Test Method for Determination of Sulfur Compounds in Natural Gas and Gaseous Fuels by Gas Chromatography and Flame Photometric Detection[1]

This standard is issued under the fixed designation D 6228; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method provides for the determination of individual volatile sulfur-containing compounds in gaseous fuels by gas chromatography (GC) with flame photometric detection (FPD). The detection range for sulfur compounds is from 20 to 20 000 picograms (pg) of sulfur. This is equivalent to 0.02 to 20 mg/m^3 or 0.014 to 14 ppmv of sulfur based upon the analysis of a 1-mL sample.

1.2 This test method describes a GC-FPD method using a specific capillary GC column. Other GC-FPD methods, with differences in GC column and equipment setup and operation, may be used as alternative methods for sulfur compound analysis with different range and precision, provided that appropriate separation of the sulfur compounds of interest can be achieved.

1.3 This test method does not intend to identify all individual sulfur species. Total sulfur content of samples can be estimated from the total of the individual compounds determined. Unknown compounds are calculated as monosulfur-containing compounds.

1.4 The values stated in SI units are to be regarded as standard. The values stated in inch-pound units are for information only.

1.5 *This standard does not purport to address all the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1072 Test Method for Total Sulfur in Fuel Gases[2]

D 1265 Practice for Sampling Liquefied Petroleum (LP) Gases–Manual Method[3]

D 1945 Test Method for Analysis of Natural Gas by Gas Chromatography[2]

D 3609 Practice for Calibration Techniques Using Permeation Tubes[4]

D 4468 Test Method for Total Sulfur in Gaseous Fuels by Hydrogenolysis and Rateometric Colorimetry[2]

D 4626 Practice for Calculation of Gas Chromatographic Response Factors[5]

D 5287 Practice for Automatic Sampling of Gaseous Fuels[2]

D 5504 Test Method for Determination of Sulfur Compounds in Natural Gas and Gaseous Fuels by Gas Chromatography and Chemiluminescence Detection[2]

E 840 Practice for Using Flame Photometric Detectors in Gas Chromatography[6]

2.2 *EPA Standards:*

EPA–15 Determination of Hydrogen Sulfide, Carbonyl Sulfide and Carbon Disulfide Emissions from Stationary Sources, 40 CFR, Chapter 1, Part 60, Appendix A

EPA–16 Semicontinuous Determination of Sulfur Emissions from Stationary Sources, 40 CFR, Chapter 1, Part 60, Appendix A

3. Terminology

3.1 *Abbreviations*:

3.1.1 A common abbreviation of a hydrocarbon compound is to designate the number of carbon atoms in the compound. A prefix is used to indicate the carbon chain form, while a subscript suffix denotes the number of carbon atoms, for example, normal decane = n-C_{10}, isotetradecane = i-C_{14}.

3.1.2 Sulfur compounds commonly are referred to by their initials, chemical or formula, for example, methyl mercaptan = MeSH, dimethyl sulfide = DMS, carbonyl sulfide = COS, di-*t*-butyl trisulfide = DtB-TS, and tetrahydothiophene = THT or thiophane.

4. Summary of Test Method

4.1 Sulfur analysis ideally is performed on-site to eliminate potential sample deterioration during storage. The reactive nature of sulfur components may pose problems both in sampling and analysis. Samples should be collected and stored in containers that are nonreactive to sulfur compounds, such as

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[1] This test method is under the jurisdiction of ASTM Committee D-3 on Gaseous Fuels and is the direct responsibility of Subcommittee D03.05 on Determination of Special Constituents of Gaseous Fuels.

Current edition approved March 10, 1998. Published May 1998.

[2] *Annual Book of ASTM Standards*, Vol 05.05.

[3] *Annual Book of ASTM Standards*, Vol 05.01.

[4] *Annual Book of ASTM Standards*, Vol 11.03.

[5] *Annual Book of ASTM Standards*, Vol 05.02.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

Tedlar[7] bags. Sample containers should be filled and purged at least three times to ensure representative sampling. Laboratory equipment also must be inert, well conditioned, and passivated with a gas containing the sulfur compounds of interest to ensure reliable results. Frequent calibration and daily verification of calibration curve using stable standards are required. Samples should be analyzed within 24 h of collection to minimize sample deterioration. If the stability of analyzed sulfur components is proved experimentally, the time between collection and analysis may be lengthened.

4.2 A 1-mL sample of the fuel gas is injected into a gas chromatograph where it is passed through a 60-m, 0.53-mm inside diameter (ID), thick film, methyl silicone liquid phase, open tubular partitioning column, or a similar column capable of separating sulfur components.

4.3 *Flame Photometric Detectors*—When combusted in a hydrogen-rich flame, sulfur compounds emit light energy characteristic to all sulfur species. The light is detected by a photomultiplier tube (PMT). The PMT response is proportional to the concentration or the amount of sulfur. All sulfur compounds including sulfur odorants can be detected by this technique.

4.4 *Other Detectors*—This test method is written primarily for the flame photometric detector. The same gas chromatographic (GC) method can be used with other sulfur-specific detectors provided they have sufficient sensitivity and selectivity to all sulfur compounds of interest in the required measurement range.

4.5 *Other GC Test Methods*—The GC test methods using sulfur chemiluminescence, reductive rateometric, and electrochemical detectors are available or under development.

5. Significance and Use

5.1 Many sources of natural gas and petroleum gases contain varying amounts and types of sulfur compounds, which are odorous, corrosive to equipment, and can inhibit or destroy catalysts used in gas processing. Their accurate measurement is essential to gas processing, operation, and utilization.

5.2 Small amounts, typically, 1 to 4 ppmv of sulfur odorant compounds, are added to natural gas and liquefied petroleum (LP) gases for safety purposes. Some odorant compounds can be reactive and may be oxidized, forming more stable compounds having lower odor thresholds. These gaseous fuels are analyzed for sulfur odorants to help ensure appropriate odorant levels for safety.

5.3 This test method offers a technique to determine individual sulfur species in gaseous fuel and the total sulfur content by calculation. Gas chromatography is used commonly and extensively to determine other components in gaseous fuels including fixed gas and organic components (see Test Method D 1945). This test method dictates the use of a specific GC technique with one of the more common detectors for measurement.

6. Apparatus

6.1 *Chromatograph*—Any gas chromatograph that has the following performance characteristics can be used.

6.1.1 *Sample Inlet System*—Gas samples are introduced to the gas chromatograph using an automated or manually operated stainless steel gas sampling valve enclosed in a heated valve oven, which must be capable of operating continuously at a temperature of 50°C above the temperature at which the gas was sampled. TFE-fluorocarbon tubing made of fluorinated ethylene propylene (FEP), 316 stainless steel tubing, or other tubing made of nonpermeable, nonsorbing, and nonreactive materials, as short as possible and heat traced at the same temperature, should be used for transferring the sample from a sample container to the gas-sampling valve. A 1.0-mL sampling loop made of nonreactive materials, such as deactivated fused silica or 316 stainless steel is used to avoid possible decomposition of reactive sulfur species. Other size fixed-volume sampling loops may be used for different concentration ranges. A 1- to 2-m section of deactivated precolumn attached to the front of the analytical column is recommended. The precolumn is connected directly to the gas sampling valve for on-column injection. The inlet system must be well conditioned and evaluated frequently for compatibility with trace quantities of reactive sulfur compounds, such as *tert*-butyl mercaptan.

6.1.2 *Digital Pressure Transmitter*—A calibrated stainless steel pressure/vacuum transducer with a digital readout may be equipped to allow sampling at different pressures to generate calibration curves.

6.1.3 *Column Temperature Programmer*—The chromatograph must be capable of linear programmed temperature operation over a range from 30 to 200°C, in programmed rate settings of 0.1 to 30°C/min. The programming rate must be sufficiently reproducible to obtain retention time repeatability of 0.05 min (3 s).

6.1.4 *Carrier and Detector Gas Control*—Constant flow control of carrier and detector gases is critical to optimum and consistent analytical performance. Control is best provided by the use of pressure regulators and fixed flow restrictors. The gas flow rate is measured by any appropriate means and the required gas flow indicated by the use of a pressure gage. Mass flow controllers, capable of maintaining gas flow constant to ±1 % at the required flow rates also can be used. The supply pressure of the gas delivered to the gas chromatograph must be at least 69 kPa (10 psi) greater than the regulated gas at the instrument to compensate for the system back pressure. In general, a supply pressure of 552 kPa (80 psig) will be satisfactory.

6.1.5 *Detector*—A flame photometric detector calibrated in the sulfur-specific mode is used for this test method. Other detectors as mentioned in 4.4 will not be covered in this test method. This detector may be obtained from various manufacturers; however, there are variations in design. The pulsed flame photometric detector (PFPD) is one of the new FPD designs. The pressure and flow rate of the hydrogen and air gases used in the detector may be different. The selection of which detector to use should be based on its performance for the intended application. The detector should be set according to the manufacturer's specifications and tuned to the best performance of sensitivity and selectivity as needed.

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[7] Registered trademark. Available from DuPont de Nemours, E. I., & Co., Inc., Barley Mill Plaza, Bldg. 10, Wilmington, DE 19880–0010.

6.1.5.1 When sulfur-containing compounds are burned in a hydrogen-rich flame, they quantitatively produce a S_2* species in an excited state (Eq 1 and Eq 2). The light emitted from this species is detected by a photomultiplier tube (PMT) (Eq 3). A 393-nm bandpass optical filter normally is used to enhance the selectivity of detection. The selectivity normally is about 10^4 to 1 by mass of sulfur to mass of carbon.

$$RS + O_2 \rightarrow n\,CO_2 + SO_2 \tag{1}$$

$$2\,SO_2 + 4\,H_2 \rightarrow 4\,H_2O + S_2{}^* \tag{2}$$

$$S_2{}^* \rightarrow S_2 + h\nu \tag{3}$$

where:

$h\nu$ = emitted light energy.

6.1.5.2 The intensity of light is not linear with the sulfur concentration but is proportional approximately to the square of the sulfur concentration. The relationship between the FPD response (R_D) and the sulfur concentration (S) is given by Eq 4 and Eq 5. The n-factor usually is less than 2.0.

$$R_D \,\alpha\, [S]^n \tag{4}$$

$$\text{Log}\,[S] \,\alpha\, 1/n\, \text{Log}\, R \tag{5}$$

where:

n = exponential factor (1.7 to 2.0).

6.1.5.3 The linear calibration curve can be made using a log-log plot. Some instruments use an electronic linearizer to produce a signal with direct linear response. The dynamic range of this linear relationship is about 1×10^3.

6.2 *Column*—A 60- by 0.53-m ID fused silica open tubular column containing a 5-µm film thickness of bonded methyl silicone liquid phase is used. The column shall provide adequate retention and resolution characteristics under the experimental conditions described in 7.3. Other columns, which can provide equivalent separation can be used, as well.

6.3 *Data Acquisition*:

6.3.1 *Recorder*—A 0- to 1-mV range recording potentiometer or equivalent, with a full-scale response time of 2 s or less can be used.

6.3.2 *Integrator*—The use of an electronic integrating device or computer is recommended. The device and software must have the following capabilities:

6.3.2.1 Graphic presentation of the chromatogram.

6.3.2.2 Digital display of chromatographic peak areas.

6.3.2.3 Identification of peaks by retention time or relative retention time, or both.

7. Reagents and Materials

7.1 *Sulfur Permeation Tube Standards*—Gaseous standards generated from individual or a combination of certified permeation tubes at a constant temperature and flow rate shall be used for all calibrations. Each permeation tube will be weighed to the nearest 0.1 mg on a periodic basis after the permeation rate has equilibrated and remains constant. The standard concentration is calculated by mass loss and dilution gas flow rate. Impurities permeated from each tube must be detected, measured, and accounted for in the mass loss, if they are present above a level of 0.1 % of the permeated sulfur species. See Practice D 3609.

7.2 *Compressed Cylinder Gas Standards*—As an alternative, blended gaseous sulfur standards may be used if a means to ensure accuracy and stability of the mixture is available. These mixtures can be a source of error if their stability during storage cannot be guaranteed.

NOTE 1—**Warning:** Sulfur compounds may be flammable and harmful or fatal if ingested or inhaled.

7.3 *Carrier Gas*—Helium or nitrogen of high purity (99.999 % min purity) (**Warning**—See Note 2). Additional purification is recommended by the use of molecular sieves or other suitable agents to remove water, oxygen, and hydrocarbons. Available pressure must be sufficient to ensure a constant carrier gas flow rate (see 6.1.4).

NOTE 2—**Warning:** Helium and nitrogen used are compressed gases under high pressure.

7.4 *Hydrogen*—Hydrogen of high purity (99.999 % min purity) is used as fuel for the flame photometric detector (FPD) (**Warning**—See Note 3).

NOTE 3—**Warning:** Hydrogen is an extremely flammable gas under high pressure.

7.5 *Air*—High-purity (99.999 % min purity) compressed air is used as the oxidant for the flame photometric detector (FPD) (**Warning**—See Note 4).

NOTE 4—**Warning:** Compressed air is a gas under high pressure that supports combustion.

8. Preparation of Apparatus and Calibration

8.1 *Chromatograph*—Place in service in accordance with the manufacturer's instructions. Typical operating conditions are shown in Table 1 .

8.2 *FPD*—Place the detector in service in accordance with the manufacturer's instructions. Hydrogen and air flows are critical and must be adjusted properly in accordance with the instruction furnished by the manufacturer. With the FPD flame ignited, monitor the signal to verify compliance with the signal noise and drift specified by the manufacturer. The FPD flame should be maintained to give consistent and optimum sensitivity for the detection range.

8.2.1 *Sample Injection*—A sample loop of 1.0 mL of suitable size for sample injection may be used for performance check. A linear calibration curve may be determined by using standards of varying concentrations or by injecting a single calibration standard at different pressures from 13.3 to 133 kPa (100 to 1000 torr). If the latter method is used, the concentration of sulfur component for calibration is calculated using the following equation.

$$S_n = (P_s/P_o) \times C_n \tag{6}$$

TABLE 1 Gas Chromatographic Operating Parameters

Gas Sample Loop:	1.0 mL at 120°C
Injection Type:	On-column
Carrier Gas:	He at 11.0 mL/min or at a flow rate allowing CH_4 elutes at approximately 2.1 min
Column Oven:	30°C hold 1.5 min, 15°C/min to 200°C, hold 8 min, or as needed
Detector:	H_2/air ratio specified by manufacturer, 250°C, 20 mL/min, helium makeup gas

where:

S_n = calculated concentration of the sulfur compound in the sampled gas on mole or volume basis,
P_s = sampling pressure as absolute,
P_o = laboratory ambient pressure as absolute, and
C_n = concentration of the sulfur compound in the calibration standard.

8.2.2 *Detector Response Calibration*—Analyze the calibration gas and obtain the chromatograms and peak areas. Determine the linear range of detector response using sample injection techniques illustrated in 8.2.1. A log/log plot or a linearized plot may be constructed with the linear correlation factor calculated. Calculate the relative sulfur response factor of each sulfur compound at ambient pressure by:

$$F_n = (S_n/A_n) \times L_n \quad (7)$$

where:

F_n = sulfur response factor of compound,
S_n = concentration of the sulfur compound in the sampled gas on mole or volume basis,
A_n = peak area of the sulfur compound measured, and
L_n = moles of sulfur in the compound.

Example:

Assume 1.0 ppmv of dimethyl sulfide (DMS) injected with a 1.0-mL sample loop.

1-ppmv DMS = 62.13/22.41 = 2.772 mg/m^3 (from Table 2). 1.0 mL of 1-ppmv DMS = 2772-pg DMS = 2772 × 51.61 % = 1430-pg S. If the peak area of DMS response is 15 850 counts, the response factor F_n (S/peak) is 1430/15 850 × 1 = 9.02 × 10^2 (pg sulfur/unit area). The response factor (F_n) of 1.0-mL injection = 1.0/15 850 × 1 = 63 × 10^{-6} (ppmv DMS/unit area).

All *mono*-sulfur compounds should have approximately the same response factor. The response factor (F_n) of each sulfur compound should be within 10 % of F_n for dimethyl sulfide. The day-to-day variation of F_n should not be greater than 5 %. The detector should be maintained and flow rates readjusted to optimize the detector performance if F_n exceeds this limitation. Table 2 lists common sulfur compounds found in gaseous fuel and their properties for calculation.

8.2.3 *Interferences*—There are two types of interferences that must be minimized for reliable quantitation.

8.2.3.1 *Hydrocarbon Quenching*—Hydrocarbons produce a quenching effect on sulfur detection as a result of the formation of a large amount of carbon dioxide in the flame suppressing the formation of SO_2. The quenching can be minimized by optimizing the chromatographic conditions to separate the sulfur components of interest from large hydrocarbons present in the sample matrix. A flame ionization detector (FID) or thermal conductivity detector (TCD) can be used to identify the presence of interfering hydrocarbons. Sample dilution or injection of a smaller volume of sample may be used to alleviate the quenching effect if sulfur concentration is significantly higher than the method detection limit.

TABLE 2 Physical Properties of Common Sulfur Compounds

Compound	Relative Molecular Mass (Molecular Weight)	% 5	Boiling Point, °C	Vapor Pressure kPa at 37.78°C
H_2S	34.08	94.09	−60.3	. . .
COS	60.08	53.37	−50.2	. . .
MeSH	48.11	66.65	6.2	214
EtSH	62.13	51.61	35.0	112
DMS	62.13	51.61	37.3	103
CS_2	76.14	84.23	46.5	. . .
iPrSH	76.16	42.10	52.6	61
TBM	90.19	35.55	64.0	41
nPrSH	76.16	42.10	67.0	35
MES	76.16	42.10	67.0	36
THT	88.17	36.37	120	4.6
di-EtS	90.19	35.55	92.0	. . .
DMDS	94.20	68.08	109.7	. . .
di-Et-DS	122.25	52.46	154.0	. . .

8.2.3.2 *Self-Quenching*—In other cases, the reverse of the reaction shown in Eq 3 produces self-quenching. This arises when the emitted light is reabsorbed before reaching the photomultiplier. It occurs when a very large concentration of any sulfur species is present in the flame above and beyond the linearity range of the detector. It often generates an M-shape peak with the inverted signal at a component's peak apex, which mistakenly can be identified as two close eluting compounds. Sample dilution or smaller sample injection may eliminate this effect.

8.3 *Chromatography*—A chromatogram of typical natural gas analysis is illustrated in Fig. 1 (relative response versus retention time). The retention times of selected sulfur components are listed for reference (Table 3). They may vary considerably depending on the chromatographic conditions. The eluting sequence and spread of sulfur peaks and their peak shape should remain the same. Adequate resolution defined as baseline separation of adjacent peaks shall be achieved. The baseline separation of two peaks is defined as the FPD signal of the first compound returns to a point at least below 5 % of the smallest peak of two.

9. Procedure

9.1 *Sampling and Preparation of Sample Aliquots*:

9.1.1 *Gas Samples*—Samples should be supplied to the laboratory in specially conditioned high-pressure sample containers or in Tedlar[7] bags at atmospheric pressure. The sample must be analyzed as soon as possible, preferably within 24 h of sampling.

9.2 *Instrument Setup*—Set up the GC-FPD in accordance with the chromatograph operating parameters listed in Table 1.

9.3 *Instrument Performance*—Analyze selected standards to verify the chromatographic performance (see 8.3), retention times (Table 3), and the response factors (see 8.2.2). The calibration made at the beginning and the end of each run or series of runs within 24-h period shall not exceed ±5 %.

9.4 *External Standard Calibration*—At least twice a day or as frequently as necessary, analyze the calibration standard mix to verify the calibration curve determined in 8.2.1 and 8.2.2 and determine the standard response factors for the sample analysis.

9.5 *Sample Analysis*—Evacuate and purge the lines from the sample container through the sample loop in the gas chromatograph. Inject 1.0 mL with a gas sampling valve as in 8.2.1. If the sample size exceeds the linear range of the detector, reduce the sample size using a smaller loop or lower sampling pressure. Run the analysis in accordance with the conditions specified in Table 1. Obtain the chromatographic

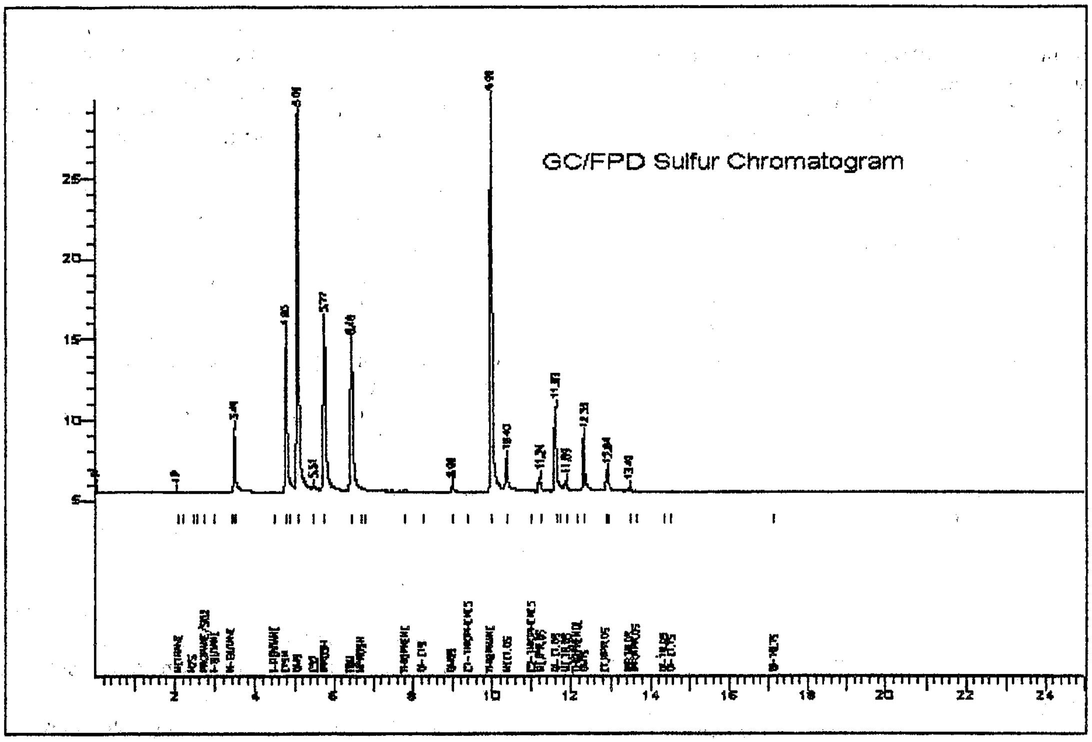

FIG. 1 Chromatogram of a Composite Odorized Natural Gas

TABLE 3 Retention Times of Various Sulfur Components

RT, min	Compound	*RT*, min	Compound	*RT*, min	Compound
2.09	methane	5.50	CS_2	11.23	M-iPr-DS
2.20	ethane	5.80	iPrSH	11.62	DEDS
2.45	H_2S	6.45	TBM	11.74	M-nPr-DS
2.55	COS	6.70	nPrSH	11.90	M-tB-DS
2.65	propane	6.80	MES	12.35	DMTS
3.00	*i*-butane	7.80	thiophene	12.87	E-nPr-DS
3.40	*n*-butane	8.25	DES	12.98	DiPr-DS
3.52	MeSH	9.00	DMDS	13.50	iPr-tB-DS
4.50	*i*-pentane	9.42	*M*-thiophenes	13.65	iPr-nPr-DS
4.75	EtSH	9.95	THT	14.35	DtB-DS
4.90	*n*-pentane	10.37	MEDS	14.55	DEt-TS
5.10	DMS	11.00	C_{27}thiophenes	17.15	DtB-TS

data by means of a potentiometric record (graphic), digital integrator, or computer-based chromatographic data system. Examine the graphic display or digital data for any errors, for example, over-range component data, and repeat the injection and analysis, if necessary. The difference between corresponding peak areas of repeated runs should not exceed 5 %.

9.6 *Compound Identification*—Sulfur compounds are identified by their retention times established during calibration. All compounds without matching standards are identified as individual unknowns.

10. Calculation

10.1 Determine the chromatographic peak area of each component and use the response factor (Eq 7) obtained from the calibration run to calculate the amount of each sulfur compound present corrected for injection pressure. The amount of each unknown sulfur compound is calculated using the response factor of the closest adjacent identified compound, unless that compound shows abnormal peak shape.

$$C_n = (A_n/F_n)\,(P_o/P_s)\,/\,L_n \tag{8}$$

where:

C_n = concentration of the sulfur compound in the gas on mole or volume basis,
A_n = peak area of the sulfur compound measured,
F_n = sulfur response factor of compound,
P_o = laboratory ambient pressure,
P_s = sampling pressure, and
L_n = moles of sulfur in the compound.

11. Report

11.1 Report the identification and concentration of each individual sulfur compound in ppmv. Report the sum of all sulfur components detected to the nearest ppmv or pg as total sulfur.

12. Precision and Bias

12.1 *Precision*—The precision of this test method is determined based on a sulfur standard mix in methane, which is stable during the testing period. The statistical examination of the laboratory test results is as follows:

12.1.1 *Repeatability (Single-Operator and Apparatus)*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed the following values by only one case in twenty.

Sulfur Compound	ppmv	Repeatability
COS	2.07	±0.06
DMS	3.63	±0.12
NPM	3.72	±0.12
DMDS	2.00	±0.06
THT	6.44	±0.16

12.1.2 *Reproducibility (Different Operators, Apparatus, and Laboratories)*—Since reference samples stable over a long testing period, which are required for this determination, are not available at this time, reproducibility cannot be determined.

12.2 *Bias*—Since there is no accepted reference material for determining the bias, no statement on bias can be made.

13. Keywords

13.1 flame photometric detection; gas chromatography; sulfur compounds, odorants

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

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Standard Test Method for Determination of Gaseous Organic Compounds by Direct Interface Gas Chromatography-Mass Spectrometry[1]

This standard is issued under the fixed designation D 6420; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method employs a direct interface gas chromatograph/mass spectrometer (GCMS) to identify and quantify the 36 volatile organic compounds (or sub-set of these compounds) listed as follows. The individual Chemical Abstract Service (CAS) numbers are listed after each compound.

Benzene-71432
Bromodichloromethane-75274
Carbon disulfide-75150
Chloroform-67663
Methyl iso-Butyl ketone-108101
Styrene-100425
Tetrachloroethylene-127184
Toluene-108883
Bromoform-75252
Vinyl acetate-108054
Vinyl chloride-75014
Chloromethane-74873
cis-1,2-Dichloroethene-156592
Dibromochloromethane-124481
1,1-Dichloroethane-107062
1,2-Dichloropropane-78875
Ethylbenzene-100414
Ethyl chloride-75003
Methylene chloride-75092
1,1,2,2-Tetrachloroethane-79349
1,1,1-Trichloroethane-71556
1,1,2-Trichloroethane-79005
p-Xylene-106423
Bromomethane-74839
Carbon tetrachloride-56235
Chlorobenzene-108907
c-1,3-Dichloropropene-10061015
1,2-Dichloroethane-156592
1,1-Dichloroethene-75354
t-1,2-Dichloroethene-156605
Methyl ethyl ketone-78933
2-Hexanone-591786
t-1,3-Dichloropropene-542756
Trichloroethene-79016
m-Xylene-108383
o-Xylene-95476

1.2 The test method incorporates a performance-based approach, which validates each GCMS analysis by placing boundaries on the instrument response to gaseous internal standards and their specific mass spectral relative abundance. Using this approach, the test method may be extended to analyze other compounds.

1.3 The test method provides on-site analysis of extracted, unconditioned, and unsaturated (at the instrument) gas samples from stationary sources. Gas streams with high moisture content may require conditioning to prevent moisture condensation within the instrument. For these samples, quality assurance (QA) requirements are provided in the test method to validate the analysis of polar, water-soluble compounds.

1.4 The instrument range should be sufficient to measure the listed volatile organic compounds from 150 ppb(v) to 100 ppm(v), using a full scan operation (between 45 and 300 atomic mass units). The range may be extended to higher or lower concentrations using either of the following procedures:

1.4.1 The initial three-point calibration concentrations and the continuing calibration checks are adjusted to match the stack concentrations, or

1.4.2 The three-point calibration is extended to include additional concentrations to cover the measurement range.

1.5 The minimum quantification level is 50 % of the lowest calibration concentration. Responses below this level are considered to be estimated concentrations, unless a calibration standard check is conducted at a lower concentration to demonstrate linearity. The sensitivity of the GCMS measurement system for the individual target analytes depends upon:

1.5.1 The specific instrument response for each target analyte and the number of mass spectral quantification ions available.

1.5.2 The amount of instrument noise, and

1.5.3 The percent moisture content of the sample gas.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Additional safety precautions are described in Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres[2]

D 3195 Practice for Rotameter Calibration[2]

2.2 *EPA Test Methods:*

Method 1–Sample and Velocity Traverses for Stationary Sources[3]

Method 2–Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)[3]

Method 3–Gas Analysis for Carbon Dioxide, Oxygen, Excess Air, and Dry Molecular Weight[3]

Method 4–Determination of Moisture Content in Stack Gases[3]

Method 624–Purgables[4]

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[1] This test method is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.03 on Ambient Atmospheres and Source Emissions.

Current edition approved May 10, 1999. Published July 1999.

[2] *Annual Book of ASTM Standards*, Vol 11.03.

[3] Code of Federal Regulations 40 CFR Part 60, Appendix A, available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

[4] Code of Federal Regulations 40 CFR Part 136, Appendix A, available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

3. Terminology

3.1 See Terminology D 1356 for definition of terms used in this test method.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *blank analysis, n*—injecting zero air or nitrogen into the GCMS to determine background levels of the target analytes.

3.2.2 *CCC, n—continuing calibration check*—injecting calibration gas standards into the GCMS to verify the calibration status.

3.2.2.1 *Discussion*—The continuing calibration check is performed before each testing day, before resuming sampling after instrument shutdown or malfunction, and before resuming sampling after 12 h of continuous instrument operation.

3.2.3 *quantification ion, n*—a specific ion in the analytes mass spectrum that is used for quantification.

3.2.4 *system calibration, n*—calibration obtained by injecting the calibration standard(s) through the entire sampling system.

3.2.5 *system zero, n*—zero obtained by injecting dry nitrogen or zero gas through the entire sampling system to determine the system background levels of the target analytes.

4. Summary of Test Method

4.1 *Analysis*—Volatile Organic Hazardous Air Pollutants (VOHAP) are analyzed using gas chromatography (GC) to separate the individual compounds and mass spectrometry (MS) to identify the compounds. The MS scans a defined mass range (usually from 45 to 300 atomic mass units (amu) for combustion sources) to identify the specific fragments for each molecule. The target analytes are identified positively by: (*1*) comparing eluting analyte GC peak retention times in the total ion chromatograph (TIC) to those contained in a three-point calibration, and (*2*) examining the mass spectral pattern of the eluted peaks. Internal standards are used to correct for hardware-related errors such as different injection volumes, operational temperature fluctuations, and electron multiplier drift.

4.2 *Sampling*—Samples are extracted from the stack or duct at a constant rate, filtered, conditioned (if required), and transported to the GCMS for analysis. Calibration gases are introduced at the extractive probe outlet, upstream of the primary particulate filter. All sample extraction components are maintained at temperatures that prevent moisture condensation within the measurement system components.

5. Significance and Use

5.1 This field test method determines the mass concentration of VOHAPs (or any subset) listed in Section 1.

5.2 Multiplying the mass concentration by the effluent volumetric flow rate (see 2.2) yields mass emission rates.

5.3 This field test method employs the typical laboratory GCMS techniques and QA/QC procedures.

5.4 This field test method provides data with accuracy and precision similar to most laboratory GCMS instrumentation.

NOTE 1—Supporting data are available from ASTM Headquarters Request RR:_______.

6. Interferences

6.1 *Analytical Interferences*—Analytical interferences occur when chromatographic peak(s) and quantification ion(s) overlap to such an extent that quantification of specific target compounds is prohibited. The nature of the GCMS technique virtually eliminates these types of analytical interferences. However, compounds having very simple mass spectra (that is, only one or two mass fragments) may be difficult to identify positively.

6.2 *Sampling System Interferences*—Sampling system interferences occur when target analytes are not transported to the instrumentation or when compounds damage the measurement system components. Water, reactive particulate matter, adsorptive sites within the sampling system components, and reactive gases are examples of such potential sampling system interferences. Specific provisions and performance criteria are included in this test method to detect and prevent the presence of sampling system interferences.

7. Apparatus

7.1 *Analytical Instrumentation:*

7.1.1 *Gas Chromatograph/Mass Spectrometer (GCMS)*, capable of separating the analyte mixture and detecting compounds in the 45 to 300 atomic mass unit (amu) range.

7.1.2 *Personal Computer*, with compatible GCMS software for control of the GCMS and for data quantification.

7.2 *Sampling System:*

7.2.1 *Sampling Probe*, glass, stainless steel, or other appropriate material of sufficient length and physical integrity to sustain heating, prevent adsorption of analytes, and to reach the gas sampling point.

7.2.2 *Calibration Assembly*, typically fabricated by user, to introduce calibration standards into the sampling system at the probe outlet, upstream of the primary particulate filter, at the same pressure and temperature as that of the effluent samples, with provisions for monitoring the sample pressure and temperature during continuing calibrations and effluent sampling.

7.2.3 *Particulate Filters*, rated at 0.3 µm, placed immediately after the heated probe and after the sample condenser system.

7.2.4 *Pump*, leak-free, with heated head, capable of maintaining an adequate sample flow rate (at least 1.5 L/min).

7.2.5 *Sampling Line*, of suitable internal diameter, heated to prevent sample condensation, made of stainless steel, tetrafluorocarbon polymer, or other material that minimizes adsorption of analytes, of minimal length.

7.2.6 *Sample Condenser System*, a refrigeration unit capable of reducing and removing the moisture of the sample gas to a level acceptable for sample injection.

7.2.7 *Sample Flow Rotameters*, capable of withstanding sample gas conditions, calibrated according to Practice D 3195.

7.2.8 *Sample Transfer Line*, to transport sample from sample interface to GCMS, heated to prevent sample condensation and fabricated of stainless steel, tetrafluorocarbon polymer, or other material to minimize adsorption of analytes, of minimal length.

7.3 *Auxiliary Equipment*:

7.3.1 *Calibration Gas Manifold*, capable of delivering nitrogen or calibration gases through sampling system or directly to the instrumentation, with provisions to provide for accurate dilution of the calibration gases as necessary. See Fig. 1 for an example schematic.

7.3.2 *Mass Flow Meters or Controllers*, with a stated accuracy and calibrated range (±2 % of scale from 0 to 500 mL/min or 0 to 5 L/min).

7.3.3 *Digital Bubble Meter (or equivalent)*, having a NIST-traceable calibration and accuracy of ±2 % of reading, with an adequate range to calibrate mass flow meters or controllers and rotameters at the specific flow rates (within ±10 %) required to perform the test method.

7.3.4 *Tubing, tetrafluorocarbon polymer* (or other material), of suitable diameter and length to connect cylinder regulators and minimize the adsorption of analytes on the tubing surface.

7.3.5 *Tubing*, 316 stainless steel (or other material), of suitable diameter and length for heated connections.

7.3.6 *Gas Regulators*, appropriate for individual gas cylinders, constructed of materials that minimize adsorption of analytes.

8. Reagents and Materials

8.1 *Calibration Gases*, gas standards (in nitrogen balance or other inert gas) for those compounds identified in Section 1, certified by the manufacturer to be accurate to 5 % or better, used for the initial and continuing calibrations.

NOTE 2—The analytical accuracy of the calibration standards must be known. The analytical accuracy for gas mixtures may be concentration dependent.

8.2 *Internal Standards*, manufacturer-certified mixtures for co-injection with sample gas.

8.3 *High Purity (HP) Nitrogen or Zero Air*, for purging sample lines and sampling system components, dilutions, and blank runs.

9. Hazards

9.1 *Target Analytes*—Many of the compounds listed in Section 1 are toxic and carcinogenic. Therefore, avoid exposure to these chemicals. Calibration standards are contained in compressed gas cylinders; exercise appropriate safety precautions to avoid accidents in their transport and use.

9.2 *Sampling Location*—This test method may involve sampling locations with high positive or negative pressures, high temperatures, elevated heights, or high concentrations of hazardous or toxic pollutants.

9.3 *Mobile or Remote Laboratory*—To avoid exposure to hazardous pollutants and to protect personnel in the laboratory, perform a leak check of the sampling system and inspect the sample exhaust equipment before sampling the calibration standards or effluent. Properly vent the exhaust gases.

10. Calibration and Standardization

10.1 *Calibration Standards*—Because of the incompatibility of some target compounds, many gas blends at each concentration may be needed to construct a calibration curve for all of the 36 target analytes listed in 1.1. Obtain or generate calibration standards of each target compound at nominal concentrations of 300 ppb(v), 1 ppm(v), and 10 ppm(v) by either of the following options:

10.1.1 *Option 1*—Obtain calibration gas standards for the target compounds at the three specified nominal concentrations.

10.1.2 *Option 2*—Obtain 10 ppm(v) calibration standards for the target analytes. Then successively dilute the 10 ppm(v) standard with ultra-pure nitrogen using mass flow meters. Dilute the 10 ppm(v) standard to 1 ppm(v) and then the 1 ppm(v) to 300 ppb(v). If Option 2 is used, analyze the calibration check (see Table 5) compounds directly as a QA audit of the dilution technique. The audit result using the calibrated GCMS must be within ±20 % for the calibration to be valid.

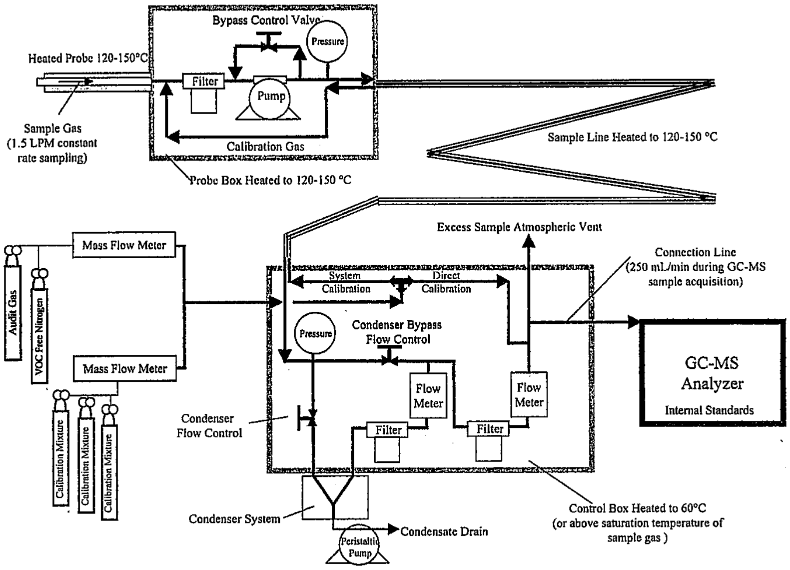

FIG. 1 Example Direct Interface GCMS Measurement System

TABLE 1 Calibration and Sampling QA/QC Criteria

Operational Mode	IS Recommended Requirements	GC Retention Time Requirements	Extracted Ion Chromatographic Criteria	Accuracy and Sensitivity
Mass Spectrometer Tune	See Table 3			
Three-Point Calibration	See Table 4	RTs for individual VOHAPS must be within ±6 % of each other from run to run.	% RSDs calculated from the individual RRFs at each calibration point must be ±20 % for each target analyte. The RFs of the internal standards must be within –50 - 100 % of the mean for the initial three-point calibration.	ARRFs must be > 0.25 to ensure proper instrumental response. A signal to noise of 10:1 (measured in accordance with the instrument manufacturer's procedures) is recommended for the low concentration level.
Surrogate Continuing Calibration Check (CCC)	See Table 4	RTs for individual VOHAPS must be within± 6 % of the initial three-point calibration.	The RFs of the internal standards must be within –50 - 100 % of their ARFs from the initial three-point calibration.	Surrogate continuing system calibration results must be within ±20 % of the manufacturers certified value from analysis by the three-point calibration for valid continuing calibration.
Sampling	See Table 4	RTs for detected VOHAPS must be within ±6 % of those in the initial three-point calibration.	The RFs of the internal standards must be within –50 - 100 % of their ARFs from the initial three-point calibration.	Spectral ions of greater than 10 % abundance in the identified compounds mass spectrum must also be contained in the reference calibration spectrum for that particular target analyte.

TABLE 2 Relative Ion Abundance Criteria for Bromofluorobenzene

Mass Fragment	Ion Abundance Criteria
50	15-40 %
75	30-60 %
95	Base peak
96	5-9 % of mass 95
173	<2 % of mass 174
174	>50 % of mass 95
175	5-9 % of mass 174
176	>95 % but <101 % of mass 174
177	5-9 % of mass 174

TABLE 3 Instrument Performance for Blended TRIS/BPFB Internal Standard Mixture in Tune

Mass Fragment	Recommended Percent Relative Low Abundance Criteria	Recommended Percent Relative High Abundance Criteria
50	5	8
55	5	8
69	33	36
93	39	45
117	100	100
167	50	65
248	30	99
263	20	50
282	10	30

10.1.3 Option 2 for preparation of the three-point calibration curve may also generate concentration levels above and below the suggested concentration range. It is necessary, when using this option, to audit the calibration curve using a certified calibration standard independent from those used to prepare the curve.

10.1.4 If the QA audit results are not within 20 % of the expected value, corrective action must be taken.

10.2 *Mass Flow Meters*—For Option 2, calibrate the mass flow meter using a digital bubble meter having a NIST-traceable calibration at the specific flow rates (±10 %) necessary for diluting the calibration standards.

TABLE 4 Instrument Performance for Separated Internal Standard Mixture

Internal Standard	Mass	Recommended Ion Abundance Criteria
TRIS	50	5-20 % of mass 69
	69	Base Peak
	75	40-60 % of mass 69
	213	50-90 % of mass 69
	263	75-95 % of mass 69
	282	30-70 % of mass 69
BPFB	93	20-50 % of mass 117
	117	Base Peak
	167	45-75 % of mass 117
	245	<2 % of mass 246
	246	>25 % of mass 117
	247	5-9 % of mass 246
	248	>25 % of mass 117
	249	5-9 % of mass 248

TABLE 5 Surrogate System Continuing Calibration Compounds

NOTE 1—The compounds listed in Table 5 represent certain class(s) of analytes known as surrogate mixture.

Compound	Class Representing	Molecular Weight	Quantification-Ion	Retention Time^A
Methylene Chloride	Chlorinated	84	84	2:41 min
Methyl Ethyl Ketone (MEK)	Polar	72	72	2:57 min
Carbon tetrachloride	Chlorinated	152	117	3:35 min
Toluene	Aromatic	92	91	5:08 min
Chlorobenzene	Chlorinated aromatic	112	112	7:22 min
O-xylene	Aromatic	91	91	9:44 min

^{A}Retention time based on 60°C isothermal separation on a SPB-1 GC column 30 m long and 0.32 mm inside diameter.

10.3 *MS Tune*—Follow the manufacturer's written instructions for the set-up, tune, operation, and calibration of the GCMS instrument.

10.3.1 If NIST library searchable mass spectra are needed to identify compounds not included in the three-point calibration or to facilitate comparison with other mass spectral analyses, the mass spectrometer tune must be able to produce mass spectra for bromofluorobenzene (BFB) that meet the relative abundance criteria in Table 2.

NOTE 3—BFB is a compound typically used to tune mass spectrometers for use in quantifying volatile organic compounds according to EPA Method 624.[4]

10.3.2 Table 3 presents a specific example of applicable MS tune limits for the mixture of two recommended gaseous internal standards—1,3,5 trifluoromethylbenzene (tris) and bromopentafluorobenzene (BPFB). These criteria have been used to demonstrate that the instrument produces an acceptable instrumental response for BFB. Table 4 presents a specific example of applicable ion abundance criteria for the two co-injected, GC separated gaseous internal standards. Meeting the QA criteria, in Tables 3 and 4 ensures proper and consistent GCMS response in each run, and thus allows meaningful searches of the NIST mass spectral library for data acquired under these conditions.

10.4 *Initial Three-Point Calibration*—Perform an initial three-point calibration for each target compound at each of the three nominal concentrations in 10.1 using duplicate analyses. Calculate relative response factors (RRFs) and average relative response factors (ARRFs) for each target compound at each of the three concentrations (Section 12, Eq 1 and 2). Calculate the percent RSD from these analyses for each target analyte. Verify that the QA/QC performance criteria in Table 1 are met.

10.5 *System Continuing Calibration Check (CCC)*:

10.5.1 Introduce the six calibration check compounds identified in Table 5 at the calibration assembly port, and determine their concentrations using the appropriate three-point calibration curves generated for the 36 analytes listed in 1.1. Allow sufficient time for the system to equilibrate. The system equilibration time should be no longer than the GCMS run time. (The compounds listed in 1.1, and any subset of these compounds, and the internal standards can be separated and detected in a 10 to 15 min isothermal GC run.) Calculate the percent differences from the respective certified values. Percent differences within ±20 % indicate that the calibration of the GCMS system for the analytes is still valid.

10.5.2 Perform analysis of the CCC each day before measuring the effluent, before resuming sampling after each instrument shut down for maintenance or corrective action, and before analyzing additional samples after twelve hours of continuous operation. Repeat procedure if difference is greater than 20 % of expected value for any target VOHAP.

NOTE 4—If only a subset of compounds identified in Section 1 are the target analytes for the test situation, then only those surrogates needed to verify the calibration status of the subset must be analyzed. (See Table 5 for the list of surrogates and compounds for which they represent.) Alternately, all of the target analytes can be used to verify continuing calibration status.

10.5.3 If a moisture condenser is used, conduct an additional CCC immediately after each test run (that is, while the potential for residual moisture in the sampling system is greatest) when analyzing for polar, water-soluble compounds. This QA check is to determine whether loss of water-soluble compounds in the system occurred. Take corrective action, if necessary, before repeating the sample run.

10.6 *Consistency During Runs*—Use the same instrument conditions to quantify effluent and QA samples that were used to establish the three-point calibration curve.

11. Procedure

11.1 *Pretest Preparations and Evaluations*:

11.1.1 *Flow Rate and Moisture Determination*—If the effluent volumetric flow rate is required, perform EPA Methods 1 through 3. Determine the moisture content to within ±2 % using EPA Method 4, wet-bulb dry-bulb measurements, saturation calculations, or other applicable means. If the moisture content of the flue gas is greater than the applicable instrument operating limit, remove the moisture (using a condenser system or other suitable apparatus) before introduction into the GCMS.

11.1.2 *Sample Interface Preparation*—Assemble the sampling system and allow the sample interface system components to reach operating temperatures. See Fig. 1 for an example schematic. Operate the sampling system at a constant sample flow rate during the entire test.

11.1.3 *Continuing Calibration Check*—Perform a CCC according to 10.5. Verify that the results agree to within ±20 % of the certified value before proceeding.

11.1.4 *System Zero Analysis*—After the CCC, perform a system zero (through the calibration assembly port) using nitrogen or zero air. Analyze nitrogen samples until the measurement system background levels are less than 50 ppb(v) for the target analytes of interest.

11.2 *Sampling and Analysis*:

11.2.1 Extract effluent sample gas for a period equal to the sample equilibration time (appropriate to the individual sampling system) before acquiring and analyzing the first sample. Co-inject the internal standards with each sample. Continuously extract the effluent between consecutive GCMS sample acquisitions to ensure constant sample equilibration within the sample interface system.

NOTE 5—The gaseous internal standards must be co-injected with the sample gas on each GCMS run. The concentration should be no greater than 10 times the expected value of the sample, but in practice this may not be possible for some sources.

11.2.2 Analyze the sample using prepared calibration files and quantification algorithms. Identify and determine the concentration of the target VOHAP according to 12.6. Verify that the QA/QC criteria listed in Table 1 are met for each run. Each sample analysis represents the emissions concentration over a period of approximately 15 min.

11.2.3 Typical test run durations are from 45 to 60 min. During each test run, analyze a minimum of three samples, unless otherwise specified.

11.3 *Data Storage*—Identify all samples with a unique file name. Store backup copies of data files.

12. Calculation

12.1 *Relative Response Factor (RRF)*—Calculate RRF as follows:

$$RRF = \frac{A_X}{A_{IS}} \frac{C_{IS}}{C_X} \quad (1)$$

where

RRF = relative response factor,

A_X = peak area of selected target VOHAP quantion,
A_{IS} = peak area of corresponding selected internal standard quantion,
C_{IS} = concentration of corresponding selected internal standard, and
C_X = concentration of target VOHAP.

12.2 *Average Relative Response Factor (ARRF)*—Calculate ARRF as follows:

$$ARRF = \frac{1}{n}\sum_{i=1}^{n} RRF_i \tag{2}$$

where:
$ARRF$ = average relative response factor,
RRF = individual RRFs calculated from calibration run (12.1).
n = 6, sample size. (Duplicate samples at each of three concentration levels.)

12.3 *Standard Deviation(s)*—Calculate as follows:

$$s = \sqrt{\frac{\sum_{i=1}^{n} (RRF_I - ARRF)^2}{n-1}} \tag{3}$$

where:
s = standard deviation,
RRF_I = individual relative response factor,
$ARRF$ = average relative response factor, and
n = 6, sample size.

12.4 *Percent Relative Standard Deviation (%RSD).* Calculate % RSD as follows:

$$\%\,RSD = \frac{s}{ARRF} \times 100 \tag{4}$$

where:
$\%\,RSD$ = percent relative standard deviation.

12.5 *Continuing Calibration Check (CCC) Percent Difference (%D).* Calculate % D as follows:

$$\%\,D = \frac{(ARRF - RRF_{CCC})}{ARRF} \times 100 \tag{5}$$

where:
$\%\,D$ = percent difference between RRF_{CCC} and ARRF,
RRF_{CCC} = target VOHAP RRF obtained from the continuing (system) calibration check, and
$ARRF$ = average relative response factor from three-point calibration.

12.6 *Quantification of Results*—Calculate the VOHAP concentrations in gas samples as follows:

$$Target\ VOHAP,\ \text{ppm(v)} = \frac{A_X}{ARRF}\,\frac{C_{IS}}{A_{IS}} \tag{6}$$

where:
A_X = peak area of target VOHAP quantion,
A_{IS} = peak area of corresponding selected internal standard quantion,
C_{IS} = concentration of corresponding selected internal standard, and
$ARRF$ = average relative response factor of target VOHAP calculated from three-point calibration.

12.7 *Alternative Quantification Calculations*—Alternative quantification algorithms, such as regression analyses, may be used to develop calibration files and sample analysis quantification. In some cases, calculation procedures allowing a non-zero y-axis intercept improve the accuracy and RSDs of measurement results. If the alternative is used, apply the same numerical procedure for the three-point calibration and all sample analyses for the test series.

13. Report

13.1 Report the results for the individual GCMS analyses, and the mean of all samples for each target analyte for each run.

13.2 Include copies of the three-point calibration including % RSD, RRFs and ARRFs, calibration check continuing system calibration(s) results and other test method QA/QC activities in the test report.

13.3 Store and include records of the manufacturer's certificates of calibration standards and internal standards.

14. Precision and Bias

14.1 *Data Quality Objectives*—The overall data quality objectives are within ±20 % of the expected value. A precision value of 10 % for each measurement value has been achieved using this method as documented in the research report. Achieving the performance criteria listed in Tables 1-4 has demonstrated that these objectives can be met.[5]

15. Keywords

15.1 gas chromatography; mass spectrometry; stack gas analysis; stationary source; volatile organic hazardous air pollutant

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[5] "Evaluation of HAPSITE and a Direct Interface GCMS Test Method for Measurement of Volatile Organic Compounds in Stationary Source Effluent," Vols I and II, July 1997. Available from ASTM Headquarters. Request RR: D22-1028 .

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Standard Test Method for Enterococci in Water Using Enterolert™[1]

This standard is issued under the fixed designation D 6503; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes a simple procedure for the detection of enterococci in water and wastewater. It is based on IDEXX's patented Defined Substrate Technology® (DST™). This product, Enterolert, utilizes a nutrient indicator that fluoresces when metabolized. It can detect these bacteria at one colony forming unit (CFU)/100 mL within 24 h. The presence of this microorganism in water is an indication of fecal contamination and the possible presence of enteric pathogens.

1.2 This test method can be used successfully with drinking water, source water, recreational (fresh and marine) water, and bottled water. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water[2]

D 1193 Specification for Reagent Water[2]

D 3370 Practices for Sampling Water from Closed Conduits[2]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *enterococci, n*—a gram positive bacteria possessing the enzyme β-D-glucosidase, which cleaves the nutrient indicator and produces fluorescence under a long wave length (366 nm) ultraviolet (UV) light.

3.2.2 *most probable number (MPN), n*—a statistical method for determining bacterial density based on the Poisson distribution.

3.2.3 *presence-absence, n*—a term used to indicate if enterococci is present in a water sample. It is a qualitative value, "yes" or "no" for reporting results.

3.2.4 *quanti-tray*™, *n*—a system for the quantification of enterococci. It consists of a sealer and trays which have multi-wells and can enumerate up to 2000 CFU/100 mL without dilution.

3.2.5 *snap pack, n*—a package containing Enterolert reagent for testing 100-mL sample either in the P/A format or quantitatively, that is, Quanti-Tray™ system).

4. Summary of Test Method [3]

4.1 This test method is used for the detection of enterococci, such as *E. faecium, E. faecalis* in drinking water, source water, recreational waters (marine water and fresh), and bottled water. When the reagent is added to the sample and incubated at 41 ± 0.5°C for 24 h, Enterolert can detect these bacteria at 1 CFU/100 mL. Fluorescence is produced when enterococci metabolizes the nutrient indicator. Enterolert can be used as a presence-absence test or for quantification (5-tube, 10-tube MPN, 15-tube serial dilution or the Quanti-Tray system).

5. Significance and Use

5.1 This test provides an easy and reliable method for the detection of enterococci in water within 24 h. For recreational water (fresh and marine) testing is performed to insure areas are safe for swimming. Enterolert also can be used for testing bottled water and drinking water.

6. Interferences

6.1 The presence of Bacillus spp. can interfere with the testing of marine water samples. To eliminate interference, a 1:10 dilution is required with sterile water (deionized or distilled).

7. Apparatus

7.1 *Ultraviolet Lamp*, 6-watt long wavelength (366 nm).

7.2 *41°C Incubator (± 0.5°C)*, air or water bath.

7.3 *Vessels*, sterile, nonfluorescent.

7.4 *Quanti-Tray Sealer*[4].

7.5 Quanti-Tray or Quanti-Tray 2000[4].

8. Reagents and Materials

8.1 *Purity of Water*—Unless otherwise indicated, references

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[1] This test method is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.24 on Microbiology.

Current edition approved Dec. 10, 1999. Published April 2000.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] This test method is based on Enterolert, a product of IDEXX Laboratories, Westbrook, ME 04092.

[4] Available from IDEXX Laboratories, One Idexx Dr., Westbrook, ME 04092.

to water shall be understood to mean reagent water conforming to Specification D 1193, Type IV. Sterilize the water by either autoclaving or by sterile filtration (0.22 micron-filtered water).

8.2 *Enterolert Test Kit*[4].

9. Precautions

9.1 The analyst must observe the normal good laboratory practices and safety procedures required in a microbiology laboratory while preparing, using, and disposing of cultures, reagents and materials and while operating sterilization equipment and other equipment.

10. Sampling

10.1 Collect the sample as described in detail in the USEPA microbiological methods manual[5] and in accordance with Practices D 3370.

10.2 *Sample Storage Temperature and Handling Conditions*—Ice or refrigerate water samples at a temperature of 2 to 8°C during transit to the laboratory. Use insulated containers to ensure proper maintenance of storage temperatures. Take care that sample bottles are not totally immersed in water during transit or storage.

10.3 *Holding Time Limitations*—Examine samples, as soon as possible, after collection. Do not hold samples longer than 6 h between collection and initiation of analyses.

11. Quality Control Check

11.1 Check and record temperatures in incubators daily to insure temperature is within stated limits.

11.2 Quality control should be conducted on each new lot of Enterolert. See package insert for the recommended quality control procedure, which consists of the following protocol:

11.2.1 For each type of the American Type Culture Collection (ATCC) bacterial strain listed below, streak the culture onto labeled TSA or blood agar plates and incubate at 35°C for 18–24 h.

11.2.2 For each bacterial strain, touch a 1-µl loop to a colony and use it to inoculate a labeled test tube containing 5 mL of sterile deionized water. Close cap and shake thoroughly.

11.2.3 For each bacterial strain, take a 1-µl loop from the test tube and use it to inoculate a labeled vessel containing 100 mL.

11.2.4 Follow the Enterolert presence/absence steps listed above to test these controls. Compare the test results to the following expected results:

Control	ATTC No.	Expected Result
Enterococcus faecium	335667	Fluorescence
Serratia marcescens (g, –)	43862	No fluorescence
Aerococcus viridians (g, +)	10400	No fluorescence

12. Procedure

12.1 *Presence/Absence*—See package insert.

12.1.1 Samples should be brought to room temperature (18–30°C).

12.1.2 Carefully separate one snap pack from the strip.

12.1.3 Tap the snap pack to insure that all of the powder is towards the bottom of the pack.

12.1.4 Open the pack by snapping back the top of the score line. Do not touch the opening of pack.

12.1.5 Add the reagent to a 100-mL water sample, which is in a sterile, transparent, nonfluorescent vessel.

12.1.6 Aseptically cap and seal the vessel.

12.1.7 Shake until dissolved.

12.1.8 Incubate Enterolert for 24 h at 41 ± 0.5°C,

12.1.9 Read results at 24 h. If the sample is inadvertently incubated over 28 h without observation, the following guidelines apply: Lack of fluorescence after 28 h is a valid negative test. Fluorescence after 28 h is an invalid result.

12.1.10 Check for fluorescence by placing a 6-W 366-nm UV light within 5 in. of the sample in a dark environment. Be sure the light is facing away from your eyes and towards the vessel. If fluorescence is observed, the presence of enterococci is confirmed.

12.2 *MPN*—Quanti-tray enumeration test procedure for 100-mL sample (see package insert).

12.2.1 Follow steps 12.1.1-12.1.7.

12.2.2 Pour the reagent sample into the Quanti-Tray avoiding contact with the foil tab and seal the tray according to the Quanti-Tray package insert.

12.2.3 Incubate for 24 h at 41 ± 0.5°C.

12.2.4 Follow the same interpretation instructions from 12.1.9 through 12.1.10, and count the number of positive wells. Refer to the MPN table (see Table 1) provided with the Quanti-Tray to determine the CFU/100 mL.

12.3 *MPN*—5-tube × 20 mL, 10-tube × 10 mL and 15-tube serial dilution.

12.3.1 Follow 12.1.1-12.1.7.

12.3.2 sterile nonfluorescent tubes or transfer 20 mL of the reagent sample into five sterile nonfluorescent tubes.

12.3.3 Incubate for 24 h at 41 ± 0.5°C.

12.3.4 Follow 12.1.9 and 12.1.10 for interpretation.

12.3.5 Refer to the MPN tables (see Tables 2-4) to determine the CFU/100 mL.

13. Calculation

13.1 For P/A, there are no calculations. For quantification, refer to Quanti-Tray MPN tables and for the 5, 10, and 15 tube test results refer to the respective MPN tables.[6]

14. Report

14.1 Report as positive or negative for presence/absence testing.

14.2 Reporting of results is based on calculation of enterococci density determined from the appropriate MPN tables.

15. Precision and Bias [7]

15.1 *Precision*—A limited collaborative study was conducted. Nine technicians from three laboratories tested three different matrixes at three levels following Practice D 2777. Outliers were rejected in accordance with the statistical tests outlined in Practice D 2777. All data from one technician was

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[5] Bordner, R.H., Winter, J.A., and Scarpino, P.V., Eds., *Microbiological Methods for Monitoring the Environment, Water, and Wastes*, EPA-600/8-78-017.

[6] *Standard Methods for the Examination of Water and Waste Water*, 19th Edition.

[7] Supporting data for this test method are available from ASTM Headquarters. Request RR: D 19-1167.

rejected for recreational water-marine and single values were rejected for both recreational water-fresh at the low level and for recreational water-marine at the low level. The mean count, the overall standard deviation (St), and the single operator standard deviation (so), are indicated in Table 5.

15.2 *Bias*—The mean value obtained for the samples (drinking water, recreational water fresh and marine) from the nine technicians for the low-, mid- and high-spiked samples all fall within the 95 % confidence interval (poisson distribution) of the actual values obtained from plating on blood agar.

15.3 Results of this collaborative study may not be typical of results for matrices other than those studied.

16. Keywords

16.1 bottled water; drinking water; enterococci; Enterolert; most probable number; presence-absence; Quanti-Tray; recreational water; source water

TABLE 1 51-Well Quanti-Tray® MPN Table

No. of Wells Giving Positive Reaction	MPN/100-mL Sample	95 % Confidence Limits	
		Lower	Upper
0	<1	0.0	3.7
1	1.0	0.3	5.6
2	2.0	0.6	7.3
3	3.1	1.1	9.0
4	4.2	1.7	10.7
5	5.3	2.3	12.3
6	6.4	3.0	13.9
7	7.5	3.7	15.5
8	8.7	4.5	17.1
9	9.9	5.3	18.8
10	11.1	6.1	20.5
11	12.4	7.0	22.1
12	13.7	7.9	23.9
13	15.0	8.8	25.7
14	16.4	9.8	27.5
15	17.8	10.8	29.4
16	19.2	11.9	31.3
17	20.7	13.0	33.3
18	22.2	14.1	35.2
19	23.8	15.3	37.3
20	25.4	16.5	39.4
21	27.1	17.7	41.6
22	28.8	19.0	43.9
23	30.6	20.4	46.3
24	32.4	21.8	48.7
25	34.4	23.3	51.2
26	36.4	24.7	53.9
27	38.4	26.4	56.6
28	40.6	28.0	59.5
29	42.9	29.7	62.5
30	45.3	31.5	65.6
31	47.8	33.4	69.0
32	50.4	35.4	72.5
33	53.1	37.5	76.2
34	56.0	39.7	80.1
35	59.1	42.0	84.4
36	62.4	44.6	88.8
37	65.9	47.2	93.7
38	69.7	50.0	99.0
39	73.8	53.1	104.8
40	78.2	56.4	111.2
41	83.1	59.9	118.3
42	88.5	63.9	126.2
43	94.5	68.2	135.4
44	101.3	73.1	146.0
45	109.1	78.6	158.7
46	118.4	85.0	174.5
47	129.8	92.7	195.0
48	144.5	102.3	224.1
49	165.2	115.2	272.2
50	200.5	135.8	387.6
51	>200.5	146.1	infinite

TABLE 2 IDEXX Quanti-Tray/2000 MPN Table (cfu/100 mL)

No. Large Wells Positive	No. Small Wells Positive																							
	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23
0	<1	1.0	2.0	3.0	4.0	5.0	6.0	7.0	8.0	9.0	10.0	11.0	12.0	13.0	14.0	15.1	16.1	17.1	18.1	19.1	20.2	21.2	22.2	23.2
1	1.0	2.0	3.0	4.0	5.0	6.0	7.0	8.1	9.1	10.1	11.1	12.1	13.2	14.2	15.2	16.2	17.3	18.3	19.3	20.4	21.4	22.4	23.5	24.5
2	2.0	3.0	4.1	5.1	6.1	7.1	8.1	9.2	10.2	11.2	12.2	13.3	14.3	15.3	16.4	17.4	18.5	19.5	20.6	21.6	22.6	23.7	24.8	25.8
3	3.1	4.1	5.1	6.1	7.2	8.2	9.2	10.3	11.3	12.4	13.4	14.4	15.5	16.5	17.6	18.6	19.7	20.8	21.8	22.9	23.9	25.0	26.1	27.1
4	4.1	5.2	6.2	7.2	8.3	9.3	10.4	11.4	12.5	13.5	14.6	15.6	16.7	17.8	18.8	19.9	21.0	22.0	23.1	24.2	25.2	26.3	27.4	28.5
5	5.2	6.3	7.3	8.4	9.4	10.5	11.5	12.6	13.7	14.7	15.8	16.9	17.9	19.0	20.1	21.2	22.2	23.3	24.4	25.5	26.6	27.7	28.8	29.9
6	6.3	7.4	8.4	9.5	10.6	11.6	12.7	13.8	14.9	15.9	17.0	18.1	19.2	20.3	21.4	22.5	23.6	24.7	25.8	26.9	28.0	29.1	30.2	31.3
7	7.4	8.5	9.6	10.7	11.8	12.8	13.9	15.0	16.1	17.2	18.3	19.4	20.5	21.6	22.7	23.8	24.9	26.0	27.1	28.3	29.4	30.5	31.6	32.8
8	8.6	9.7	10.8	11.9	13.0	14.1	15.2	16.3	17.4	18.5	19.6	20.7	21.8	22.9	24.1	25.2	26.3	27.4	28.6	29.7	30.8	32.0	33.1	34.3
9	9.8	10.9	12.0	13.1	14.2	15.3	16.4	17.5	18.7	19.8	20.9	22.0	23.2	24.3	25.4	26.6	27.7	28.9	30.0	31.2	32.3	33.5	34.6	35.8
10	11.0	12.1	13.2	14.3	15.5	16.6	17.7	18.9	20.0	21.1	22.3	23.4	24.6	25.7	26.9	28.0	29.2	30.3	31.5	32.7	33.8	35.0	36.2	37.4
11	12.2	13.4	14.5	15.6	16.8	17.9	19.1	20.2	21.4	22.5	23.7	24.8	26.0	27.2	28.3	29.5	30.7	31.9	33.0	34.2	35.4	36.6	37.8	39.0
12	13.5	14.6	15.8	16.9	18.1	19.3	20.4	21.6	22.7	23.9	25.1	26.3	27.5	28.6	29.8	31.0	32.2	33.4	34.6	35.8	37.0	38.2	39.4	40.7
13	14.8	16.0	17.1	18.3	19.5	20.6	21.8	23.0	24.2	25.4	26.6	27.8	29.0	30.2	31.4	32.6	33.8	35.0	36.2	37.5	38.7	39.9	41.1	42.4
14	16.1	17.3	18.5	19.7	20.9	22.1	23.3	24.4	25.7	26.9	28.1	29.3	30.5	31.7	33.0	34.2	35.4	36.7	37.9	39.1	40.4	41.6	42.9	44.2
15	17.5	18.7	19.9	21.1	22.3	23.5	24.7	25.9	27.2	28.4	29.6	30.9	32.1	33.3	34.6	35.8	37.1	38.4	39.6	40.9	42.2	43.4	44.7	46.0
16	18.9	20.1	21.3	22.6	23.8	25.0	26.2	27.5	28.7	30.0	31.2	32.5	33.7	35.0	36.3	37.5	38.8	40.1	41.4	42.7	44.0	45.3	46.6	47.9
17	20.3	21.6	22.8	24.0	25.3	26.5	27.8	29.1	30.3	31.6	32.9	34.1	35.4	36.7	38.0	39.3	40.6	41.9	43.2	44.5	45.9	47.2	48.5	49.8
18	21.8	23.1	24.3	25.6	26.9	28.1	29.4	30.7	32.0	33.3	34.6	35.9	37.2	38.5	39.8	41.1	42.4	43.8	45.1	46.4	47.8	49.1	50.5	51.9
19	23.3	24.6	25.9	27.2	28.5	29.8	31.1	32.4	33.7	35.0	36.3	37.6	39.0	40.3	41.6	43.0	44.3	45.7	47.1	48.4	49.8	51.2	52.6	54.0
20	24.9	26.2	27.5	28.8	30.1	31.4	32.8	34.1	35.4	36.8	38.1	39.5	40.8	42.2	43.6	44.9	46.3	47.7	49.1	50.5	51.9	53.3	54.7	56.1
21	26.5	27.8	29.2	30.5	31.8	33.2	34.5	35.9	37.3	38.6	40.0	41.4	42.8	44.1	45.5	46.9	48.4	49.8	51.2	52.6	54.1	55.5	56.9	58.4
22	28.2	29.5	30.9	32.3	33.6	35.0	36.4	37.7	39.1	40.5	41.9	43.3	44.7	46.2	47.6	49.0	50.5	51.9	53.4	54.8	56.3	57.8	59.3	60.7
23	29.9	31.3	32.7	34.1	35.4	36.8	38.2	39.7	41.1	42.5	43.9	45.4	46.8	48.3	49.7	51.2	52.7	54.2	55.6	57.1	58.6	60.2	61.7	63.2
24	31.7	33.1	34.5	35.9	37.3	38.8	40.2	41.6	43.1	44.6	46.0	47.5	49.0	50.5	51.9	53.4	55.0	56.5	58.0	59.5	61.1	62.6	64.2	65.8
25	33.5	35.0	36.4	37.9	39.3	40.8	42.2	43.7	45.2	46.7	48.2	49.7	51.2	52.7	54.3	55.8	57.3	58.9	60.5	62.0	63.6	65.2	66.8	68.4
26	35.5	36.9	38.4	39.9	41.3	42.8	44.3	45.9	47.4	48.9	50.4	52.0	53.5	55.1	56.7	58.2	59.8	61.4	63.0	64.7	66.3	67.9	69.6	71.2
27	37.4	38.9	40.4	41.9	43.5	45.0	46.5	48.1	49.6	51.2	52.8	54.4	56.0	57.6	59.2	60.8	62.4	64.1	65.7	67.4	69.1	70.8	72.5	74.2
28	39.5	41.0	42.6	44.1	45.7	47.2	48.8	50.4	52.0	53.6	55.2	56.9	58.5	60.1	61.8	63.5	65.2	66.9	68.6	70.3	72.0	73.7	75.5	77.3
29	41.6	43.2	44.8	46.4	48.0	49.6	51.2	52.8	54.5	56.1	57.8	59.5	61.2	62.9	64.6	68.3	68.0	69.8	71.5	73.3	75.1	76.9	78.7	80.5
30	43.9	45.5	47.1	48.7	50.4	52.0	53.7	55.4	57.1	58.8	60.5	62.2	64.0	65.7	67.5	69.3	71.0	72.8	74.7	76.5	78.3	80.2	82.1	84.0
31	46.2	47.9	49.5	51.2	52.9	54.6	56.3	58.1	59.8	61.6	63.3	65.1	66.9	68.7	70.5	72.4	74.2	76.1	78.0	79.9	81.8	83.7	85.7	87.6
32	48.7	50.4	52.1	53.8	55.6	57.3	59.1	60.9	62.7	64.5	66.3	68.1	70.0	71.9	73.8	75.7	77.6	79.5	81.5	83.5	85.4	87.5	89.5	91.5
33	51.2	53.0	54.7	56.5	58.3	60.1	62.0	63.8	65.7	67.6	69.5	71.4	73.3	75.2	77.2	79.2	81.2	83.2	85.2	87.3	89.3	91.4	93.5	95.7
34	53.9	55.7	57.6	59.4	61.3	63.1	65.0	66.9	68.9	70.8	72.8	74.8	76.8	78.8	80.8	82.9	85.0	87.1	89.2	91.4	93.5	95.7	97.9	100.1
35	56.8	58.6	60.5	62.4	64.4	66.3	68.3	70.3	72.3	74.3	76.3	78.4	80.5	82.6	84.7	86.9	89.1	91.3	93.5	95.7	98.0	100.3	102.6	105.0
36	59.8	61.7	63.7	65.7	67.7	69.7	71.7	73.8	75.9	78.0	80.1	82.3	84.5	86.7	88.9	91.2	93.5	95.8	98.1	100.5	102.9	105.3	107.7	110.2
37	62.9	65.0	67.0	69.1	71.2	73.3	75.4	77.6	79.8	82.0	84.2	86.5	88.8	91.1	93.4	95.8	98.2	100.6	103.1	105.6	108.1	110.7	113.3	115.9
38	66.3	68.4	70.6	72.7	74.9	77.1	79.4	81.6	83.9	86.2	88.6	91.0	93.4	95.8	98.3	100.8	103.4	105.9	108.6	111.2	113.9	116.6	119.4	122.2
39	69.9	72.2	74.4	76.6	78.9	81.3	83.6	86.0	88.4	90.9	93.3	95.9	98.4	101.0	103.6	106.3	109.0	111.8	114.5	117.4	120.3	123.2	126.1	129.2
40	73.8	76.2	78.5	80.9	83.3	85.7	88.2	90.7	93.3	95.9	98.5	101.2	103.9	106.7	109.5	112.4	115.3	118.2	121.2	124.2	127.3	130.5	133.7	137.0
41	78.0	80.5	83.0	85.5	88.0	90.6	93.3	95.9	98.7	101.4	104.3	107.1	110.0	113.0	116.0	119.1	122.2	125.4	128.7	132.0	135.3	138.8	142.3	145.9
42	82.6	85.2	87.8	90.5	93.2	96.0	98.8	101.7	104.6	107.6	110.6	113.7	116.9	120.1	123.3	126.7	130.1	133.6	137.1	140.8	144.5	148.3	152.2	156.1
43	87.6	90.4	93.2	96.0	99.0	101.9	105.0	108.1	111.2	114.5	117.8	121.1	124.6	128.1	131.7	135.4	139.1	143.0	147.0	151.0	155.1	159.4	163.8	168.2
44	93.1	96.1	99.1	102.2	105.4	108.6	111.9	115.3	118.7	122.3	125.9	129.6	133.4	137.4	141.4	145.5	149.7	154.1	158.5	163.1	167.8	172.7	177.7	182.9
45	99.3	102.5	105.8	109.2	112.6	116.2	119.8	123.6	127.4	131.3	135.4	139.6	143.9	148.3	152.9	157.6	162.4	167.4	172.6	177.9	183.5	189.2	195.1	201.2
46	106.3	109.8	113.4	117.2	121.0	125.0	129.1	133.3	137.6	142.1	146.7	151.5	156.5	161.6	166.9	172.5	178.2	184.2	190.4	196.8	203.5	210.5	217.8	225.4
47	114.3	118.3	122.4	126.6	130.9	135.4	140.1	145.0	150.0	155.3	160.7	166.4	172.3	178.5	185.0	191.8	198.9	206.3	214.2	222.4	231.0	240.0	249.5	259.5
48	123.9	128.4	133.1	137.9	143.0	148.3	153.9	159.7	165.8	172.2	178.9	186.0	193.5	201.4	209.8	218.7	228.2	238.2	248.9	260.2	272.3	285.1	298.7	313.0
49	135.5	140.8	146.4	152.3	158.5	165.0	172.0	179.3	187.2	195.6	204.6	214.3	224.7	235.9	248.1	261.3	275.5	290.9	307.6	325.5	344.8	365.4	387.3	410.6

TABLE 3 IDEXX Quanti-Tray/2000 MPN Table (cfu/100 mL)

	No. Small Wells Positive																							
No.	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48
	25.3	26.3	27.4	28.4	29.5	30.5	31.5	32.6	33.6	34.7	35.7	36.8	37.8	38.9	39.9	41.0	42.1	43.1	44.2	45.3	46.3	47.4	48.5	49.5
	26.6	27.6	28.7	29.7	30.8	31.9	32.9	34.0	35.0	36.1	37.2	38.2	39.3	40.4	41.4	42.5	43.6	44.7	45.7	46.8	47.9	49.0	50.1	51.2
	27.9	29.0	30.0	31.1	32.2	33.2	34.3	35.4	36.5	37.5	38.6	39.7	40.8	41.9	42.9	44.0	45.1	46.2	47.3	48.4	49.5	50.6	51.7	52.8
	29.3	30.3	31.4	32.5	33.6	34.7	35.7	36.8	37.9	39.0	40.1	41.2	42.3	43.4	44.5	45.6	46.7	47.8	48.9	50.0	51.2	52.3	53.4	54.5
	30.7	31.7	32.8	33.9	35.0	36.1	37.2	38.3	39.4	40.5	41.6	42.8	43.9	45.0	46.1	47.2	48.3	49.5	50.6	51.7	52.9	54.0	55.1	56.3
	32.1	33.2	34.3	35.4	36.5	37.6	38.7	39.8	41.0	42.1	43.2	44.3	45.5	46.6	47.7	48.9	50.0	51.2	52.3	53.5	54.6	55.8	56.9	58.1
	33.5	34.6	35.8	36.9	38.0	39.1	40.3	41.4	42.6	43.7	44.8	46.0	47.1	48.3	49.4	50.6	51.7	52.9	54.1	55.2	56.4	57.6	58.7	59.9
	35.0	36.2	37.3	38.4	39.6	40.7	41.9	43.0	44.2	45.3	46.5	47.7	48.8	50.0	51.2	52.3	53.5	54.7	55.9	57.1	58.2	59.4	60.6	61.8
	36.5	37.7	38.9	40.0	41.2	42.3	43.5	44.7	45.9	47.0	48.2	49.4	50.6	51.8	53.0	54.1	55.3	56.5	57.7	58.9	60.2	61.4	62.6	63.8
	38.1	39.3	40.5	41.6	42.8	44.0	45.2	46.4	47.6	48.8	50.0	51.2	52.4	53.6	54.8	56.0	57.2	58.4	59.7	60.9	62.1	63.4	64.6	65.8
	39.7	40.9	42.1	43.3	44.5	45.7	46.9	48.1	49.3	50.6	51.8	53.0	54.2	55.5	56.7	57.9	59.2	60.4	61.6	62.9	64.2	65.4	66.7	67.9
	41.4	42.6	43.8	45.0	46.3	47.5	48.7	49.9	51.2	52.4	53.6	54.9	56.1	57.4	58.6	59.9	61.2	62.4	63.7	65.0	66.2	67.5	68.8	70.1
	43.1	44.3	45.6	46.8	48.1	49.3	50.5	51.8	53.1	54.3	55.6	56.8	58.1	59.4	60.7	61.9	63.2	64.5	65.8	67.1	68.4	69.7	71.0	72.3
	44.9	46.1	47.4	48.6	49.9	51.2	52.4	53.7	55.0	56.3	57.6	58.9	60.2	61.5	62.8	64.1	65.4	66.7	68.0	69.3	70.7	72.0	73.3	74.7
	46.7	48.0	49.3	50.5	51.8	53.1	54.4	55.7	57.0	58.3	59.6	60.9	62.3	63.6	64.9	66.3	67.6	68.9	70.3	71.6	73.0	74.4	75.7	77.1
	48.6	49.9	51.2	52.5	53.8	55.1	56.4	57.8	59.1	60.4	61.8	63.1	64.5	65.8	67.2	68.5	69.9	71.3	72.6	74.0	75.4	76.8	78.2	79.6
	50.5	51.8	53.2	54.5	55.8	57.2	58.5	59.9	61.2	62.6	64.0	65.3	66.7	68.1	69.5	70.9	72.3	73.7	75.1	76.5	77.9	79.3	80.8	82.2
	52.5	53.9	55.2	56.6	58.0	59.3	60.7	62.1	63.5	64.9	66.3	67.7	69.1	70.5	71.9	73.3	74.8	76.2	77.6	79.1	80.5	82.0	83.5	84.9
	54.6	56.0	57.4	58.8	60.2	61.6	63.0	64.4	65.8	67.2	68.6	70.1	71.5	73.0	74.4	75.9	77.3	78.8	80.3	81.8	83.3	84.8	86.3	87.8
	56.8	58.2	59.6	61.0	62.4	63.9	65.3	66.7	68.2	69.7	71.1	72.6	74.1	75.5	77.0	78.5	80.0	81.5	83.1	84.6	86.1	87.6	89.2	90.7
	59.0	60.4	61.9	63.3	64.8	66.3	67.7	69.2	70.7	72.2	73.7	75.2	76.7	78.2	79.8	81.3	82.8	84.4	85.9	87.5	89.1	90.6	92.2	93.8
	61.3	62.8	64.3	65.8	67.3	68.8	70.3	71.8	73.3	74.9	76.4	77.9	79.5	81.0	82.6	84.2	85.8	87.4	89.0	90.6	92.2	93.8	95.4	97.1
	63.7	65.3	66.8	68.3	69.8	71.4	72.9	74.5	76.1	77.6	79.2	80.8	82.4	84.0	85.6	87.2	88.9	90.5	92.1	93.8	95.5	97.1	98.8	100.5
	66.3	67.8	69.4	71.0	72.5	74.1	75.7	77.3	78.9	80.5	82.1	83.8	85.4	87.1	88.7	90.4	92.1	93.8	95.5	97.2	98.9	100.6	102.3	104.1
	68.9	70.5	72.1	73.7	75.3	77.0	78.6	80.2	81.9	83.6	85.2	86.9	88.6	90.3	92.0	93.8	95.5	97.2	99.0	100.7	102.5	104.3	106.1	107.9
	71.7	73.3	75.0	76.6	78.3	80.0	81.6	83.3	85.0	86.8	88.5	90.2	92.0	93.7	95.5	97.3	99.1	100.9	102.7	104.5	106.3	108.2	110.0	111.9
	74.6	76.3	78.0	79.7	81.4	83.1	84.8	86.6	88.4	90.1	91.9	93.7	95.5	97.3	99.2	101.0	102.9	104.7	106.6	108.5	110.4	112.3	114.2	116.2
	77.6	79.4	81.1	82.9	84.6	86.4	88.2	90.0	91.9	93.7	95.5	97.4	99.3	101.2	103.1	105.0	106.9	108.8	110.8	112.7	114.7	116.7	118.7	120.7
	80.8	82.6	84.4	86.2	88.1	89.9	91.8	93.7	95.6	97.5	99.4	101.3	103.3	105.2	107.2	109.2	111.2	113.2	115.2	117.3	119.3	121.4	123.5	125.6
	84.2	86.1	87.9	89.8	91.7	93.6	95.6	97.5	99.5	101.5	103.5	105.5	107.5	109.5	111.6	113.7	115.7	117.8	120.0	122.1	124.2	126.4	128.6	130.8
	87.8	89.7	91.7	93.6	95.6	97.6	99.6	101.6	103.7	105.7	107.8	109.9	112.0	114.1	116.3	118.5	120.6	122.8	125.1	127.3	129.5	131.8	134.1	136.4
	91.6	93.6	95.6	97.7	99.7	101.8	103.9	106.0	108.2	110.3	112.5	114.7	116.9	119.1	121.4	123.6	125.9	128.2	130.5	132.9	135.3	137.7	140.1	142.5
	95.7	97.7	99.9	102.0	104.2	106.3	108.5	110.7	113.0	115.2	117.5	119.8	122.1	124.5	126.8	129.2	131.6	134.0	136.5	139.0	141.5	144.0	146.6	149.1
	100.0	102.2	104.4	106.6	108.9	111.2	113.5	115.8	118.2	120.5	122.9	125.3	127.8	130.3	132.8	135.3	137.8	140.4	143.0	145.6	148.3	150.9	153.6	156.4
	104.7	107.0	109.3	111.7	114.0	116.4	118.9	121.3	123.8	126.3	128.8	131.4	134.0	136.6	139.2	141.9	144.6	147.3	150.1	152.9	155.7	158.6	161.5	164.4
	109.7	112.2	114.6	117.1	119.6	122.1	124.7	127.3	129.9	132.6	135.3	138.0	140.8	143.6	146.4	149.2	152.1	155.0	158.0	161.0	164.0	167.1	170.2	173.3
	115.2	117.8	120.4	123.0	125.7	128.4	131.1	133.9	136.7	139.5	142.4	145.3	148.3	151.3	154.3	157.3	160.4	163.6	166.8	170.0	173.3	176.6	179.9	183.3
	121.3	124.0	126.8	129.6	132.4	135.3	138.2	141.2	144.2	147.2	150.3	153.5	156.6	159.9	163.1	166.4	169.8	173.2	176.7	180.2	183.7	187.3	191.0	194.7
	127.9	130.8	133.8	136.8	139.9	143.0	146.1	149.3	152.6	155.9	159.2	162.6	166.1	169.6	173.2	176.8	180.4	184.2	188.0	191.8	195.7	199.6	203.7	207.7
	135.3	138.5	141.7	145.0	148.3	151.7	155.1	158.6	162.1	165.7	169.4	173.1	176.9	180.7	184.7	188.6	192.7	196.8	201.0	205.3	209.6	214.0	218.5	223.0
	143.7	147.1	150.6	154.2	157.8	161.5	165.3	169.1	173.0	177.0	181.1	185.2	189.4	193.7	198.1	202.5	207.0	211.7	216.4	221.1	226.0	231.0	236.0	241.1
	153.2	157.0	160.9	164.8	168.9	173.0	177.2	181.4	185.8	190.3	194.8	199.5	204.2	209.1	214.0	219.0	224.2	229.4	234.8	240.2	245.8	251.5	257.2	263.1
	164.3	168.6	172.9	177.3	181.9	186.5	191.3	196.1	201.1	206.2	211.4	216.7	222.1	227.7	233.4	239.2	245.2	251.3	257.5	263.8	270.3	276.9	283.6	290.5
	177.5	182.3	187.3	192.4	197.6	202.9	208.4	214.0	219.8	225.8	231.8	238.1	244.5	251.0	257.7	264.6	271.7	278.9	286.3	293.8	301.5	309.4	317.4	325.6
	193.6	199.3	205.0	211.0	217.2	223.5	230.0	236.7	243.6	250.7	258.1	265.6	273.3	281.2	289.4	297.8	306.3	315.1	324.1	333.3	342.8	352.4	362.3	372.4
	214.1	220.9	227.9	235.1	242.7	250.4	258.4	266.7	275.3	284.1	293.2	302.6	312.3	322.3	332.5	343.0	353.8	364.9	376.2	387.9	399.8	412.0	424.5	437.4
	241.5	250.0	258.9	268.2	277.8	287.7	298.1	308.8	319.9	331.4	343.3	355.5	368.1	381.1	394.5	408.3	422.5	437.0	452.0	467.4	483.3	499.5	516.3	533.5
	280.9	292.4	304.4	316.9	330.0	343.6	357.8	372.5	387.7	403.4	419.8	436.6	454.1	472.1	490.7	509.9	529.8	550.4	571.7	593.8	616.7	640.5	665.3	691.0
	344.1	360.9	378.4	396.8	416.0	436.0	456.9	478.6	501.2	524.7	549.2	574.8	601.5	629.4	658.6	689.3	721.5	755.5	791.5	829.7	870.4	913.9	960.6	1011.1
	461.1	488.4	517.2	547.5	579.4	613.1	648.8	686.7	727.0	770.1	816.4	866.4	920.8	980.4	1046.2	1119.9	1203.3	1299.7	1413.6	1553.1	1732.9	1986.3	2419.2	>2419.2

TABLE 4 MPN Index and 95 % Confidence Limits for Various Combinations of Positive Results When Five Tubes are Used/Dilution (10 mL, 1.0 mL, 0.1 mL)[A]

Combination of Positives	MPN Index/100 mL	95 % Confidence Limits	
		Lower	Upper
0-0-0	<2	—	—
0-0-1	2	1.0	10
0-1-0	2	1.0	10
0-2-0	4	1.0	13
1-0-0	2	1.0	11
1-0-1	4	1.0	15
1-1-0	4	1.0	15
1-1-1	6	2.0	18
1-2-0	6	2.0	18
2-0-0	4	1.0	17
2-0-1	7	2.0	20
2-1-0	7	2.0	21
2-1-1	9	3.0	24
2-2-0	9	3.0	25
2-3-0	12	5.0	29
3-0-0	8	3.0	24
3-0-1	11	4.0	29
3-1-0	11	4.0	29
3-1-1	14	6.0	35
3-2-0	14	6.0	35
3-2-1	17	7.0	40
4-0-0	13	5.0	38
4-0-1	17	7.0	45
4-1-0	17	7.0	46
4-1-1	21	9.0	55
4-1-2	26	12	63
4-2-0	22	9.0	56
4-2-1	26	12	65
4-3-0	27	12	67
4-3-1	33	15	77
4-4-0	34	16	80
5-0-0	23	9.0	86
5-0-1	30	10	110
5-0-2	40	20	140
5-1-0	30	10	120
5-1-1	50	20	150
5-1-2	60	30	180
5-2-0	50	20	170
5-2-1	70	30	210
5-2-2	90	40	250
5-3-0	80	30	250
5-3-1	110	40	300
5-3-2	140	60	360
5-3-3	170	80	410
5-4-0	130	50	390
5-4-1	170	70	480
5-4-2	220	100	580
5-4-3	280	120	690
5-4-4	350	160	820
5-5-0	240	100	940
5-5-1	300	100	1300
5-5-2	500	200	2000
5-5-3	900	300	2900
5-5-4	1600	600	5300
5-5-5	≥1600	—	—

[A] Based on *Standard Methods for the Examination of Water and Wastewater*, 19th ed.

TABLE 5 Mean Count, Overall Standard Deviation and Single Operator Standard Deviation

NOTE 1—All calculations were made from the statistical summary given as Table One in the study file

Summary Table	Low Level				Middle Level				High Level			
Background	n	X MPN/100 mL	St %	So %	n	X MPN/100 mL	St %	So %	n	X MPN/100 mL	St %	So %
Matrix												
Drinking water	9	9.1	27.7	24.2	9	26.2	15.1	15	9	61.0	10.2	9.5
Rec. water Fresh	9	9.4[A]	30.2[A]	23.4[A]	9	29.1	14.1	6.9	9	64.3	10.6	11
Rec. water Marine	8	7.9[A]	25.2[A]	17.7[A]	8	29.4	17.4	13	8	67.5	8.0	7.6

[A] One value rejected to make this estimate.

CERTIFICATE

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An American National Standard

Standard Specification for Wire Cloth and Sieves for Testing Purposes[1]

This standard is issued under the fixed designation E 11; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers the requirements for design and construction of testing sieves using a medium of woven wire cloth mounted in a frame for use in testing for the classification of materials according to designated particle size (See Notes 1 and 2), and wire cloth, meeting the specifications of Table 1, to be designated test grade wire cloth. All subsequent references to wire cloth shall mean test grade wire cloth. Methods for checking testing sieves and wire cloth for conformance to this specification are included in the annex.

NOTE 1—Complete instructions and procedures on the use and calibration of testing sieves are contained in *ASTM STP447B*.[2] Note that sieve analysis results from two testing sieves of the same sieve designation may not be the same because of the variances in sieve opening permitted by this specification. To minimize the differences in sieve analysis results, the use of testing sieves matched on a performance basis is suggested. *ASTM STP447B*[2] also contains a list of all published ASTM standards on sieve analysis procedures for specific materials or industries. This list may be referenced to obtain statements of precision and bias for sieve analysis of specific materials.

NOTE 2—For other types of sieves, see Specification E 323 and Specification E 161.

1.2 The values stated in SI units shall be considered standard for the dimensions of the wire cloth openings and the diameter of the wires used in the wire cloth. The values stated in inch-pound units shall be considered standard with regard to the sieve frames.

1.3 The following precautionary statement refers only to the test method portion, Annex A1, of this specification: *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 430 Test Method for Fineness of Hydraulic Cement by the 45-µm No. 325 Sieve[3]

E 161 Specification for Precision Electroformed Sieves (Square-Opening Series)[4]

E 323 Specification for Perforated-Plate Sieves for Testing Purposes[4]

E 437 Specifications for Industrial Wire Cloth and Screens (Square Opening Series)[4]

2.2 *Federal Standard:*

Fed. Std. No. 123 Marking for Shipment (Civil Agencies)[5]

2.3 *Military Standard:*

MIL-STD-129 Marking for Shipment and Storage[5]

3. Ordering Information

3.1 Orders for items under this specification include the following information as necessary:

3.1.1 Name of material (U.S.A. Standard Testing Sieves or U.S.A. Standard sieve cloth),

3.1.2 ASTM designation and year of issue (ASTM E 11 – 95),

3.1.3 Quantity of each item,

3.1.4 Standard sieve designation (see Table 1, Column 1),

3.1.5 Alternative sieve designation if needed (see Table 1, Column 2),

3.1.6 For testing sieves in standard circular frames:

3.1.6.1 Nominal sieve frame diameter (see 5.2 and 5.3),

3.1.6.2 Nominal sieve frame height (see Table 2),

3.1.7 For sieve cloth not in frames or in nonstandard frames:

3.1.7.1 Lateral dimensions of sieve cloth,

3.1.7.2 Description of nonstandard frame,

3.1.8 For U.S. Government purchases, if supplementary requirements apply,

3.1.9 Compatible sieve pans and covers, and

3.1.10 Special requirements (specific type of metal for sieve cloth and frames, matched sieves, for example).

4. Sieve Cloth Requirements

4.1 Wire cloth used in U.S.A. standard testing sieves meeting the specifications shown in Table 1 shall be designated "test grade". Test grade sieve cloth shall be woven from stainless steel, brass, bronze, or other suitable wire with a plain weave, except that cloth with openings of 63 µm (No. 230) and finer may be woven with a twill weave. For definitions of "plain" and "twill" weave, refer to Specification E 437. The wire shall not be coated or plated.

4.2 The openings of the sieve cloth of successive sieves progress from a base of 1 mm in the ratio of approximately $\sqrt[4]{2}$:1.

4.3 All measurements of openings and wire diameters

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[1] This specification is under the jurisdiction of ASTM Committee E-29 on Particle Size Measurement and is the direct responsibility of Subcommittee E29.01 on Sieves, Sieving Methods, and Screening Media.

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[2] *Manual on Testing Sieving Methods, ASTM STP 447B*. Available from ASTM Headquarters.

[3] *Annual Book of ASTM Standards*, Vol 04.01.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

TABLE 1 Nominal Dimensions, Permissible Variations for Wire Cloth of Standard Test Sieves (U.S.A.) Standard Series

Sieve Designation		Nominal Sieve Opening, in.[B]	Permissible Variation of Average Opening from the Standard Sieve Designation	Opening Dimension Exceeded By Not More Than 5 % of the Openings	Maximum Individual Opening	Nominal Wire Diameter, mm[C]
Standard[A]	Alternative					
(1)	(2)	(3)	(4)	(5)	(6)	(7)
125 mm	5 in.	5	±3.70 mm	130.0 mm	130.9 mm	8.00
106 mm	4.24 in.	4.24	±3.20 mm	110.2 mm	111.1 mm	6.30
100 mm[D]	4 in.[D]	4	±3.00 mm	104.0 mm	104.8 mm	6.30
90 mm	3½ in.	3.5	±2.70 mm	93.6 mm	94.4 mm	6.30
75 mm	3 in.	3	±2.20 mm	78.1 mm	78.7 mm	6.30
63 mm	2½ in.	2.5	±1.90 mm	65.6 mm	66.2 mm	5.60
53 mm	2.12 in.	2.12	±1.60 mm	55.2 mm	55.7 mm	5.00
50 mm[D]	2 in.[D]	2	±1.50 mm	52.1 mm	52.6 mm	5.00
45 mm	1¾ in.	1.75	±1.40 mm	46.9 mm	47.4 mm	4.50
37.5 mm	1½ in.	1.5	±1.10 mm	39.1 mm	39.5 mm	4.50
31.5 mm	1¼ in.	1.25	±1.00 mm	32.9 mm	33.2 mm	4.00
26.5 mm	1.06 in.	1.06	±.800 mm	27.7 mm	28.0 mm	3.55
25.0 mm[D]	1.00 in.[D]	1	±.800 mm	26.1 mm	26.4 mm	3.55
22.4 mm	⅞ in.	0.875	±.700 mm	23.4 mm	23.7 mm	3.55
19.0 mm	¾ in.	0.750	±.600 mm	19.9 mm	20.1 mm	3.15
16.0 mm	⅝ in.	0.625	±.500 mm	16.7 mm	17.0 mm	3.15
13.2 mm	0.530 in.	0.530	±.410 mm	13.83 mm	14.05 mm	2.80
12.5 mm[D]	½ in.[D]	0.500	±.390 mm	13.10 mm	13.31 mm	2.50
11.2 mm	7⁄16 in.	0.438	±.350 mm	11.75 mm	11.94 mm	2.50
9.5 mm	⅜ in.	0.375	±.300 mm	9.97 mm	10.16 mm	2.24
8.0 mm	5⁄16 in.	0.312	±.250 mm	8.41 mm	8.58 mm	2.00
6.7 mm	0.265 in.	0.265	±.210 mm	7.05 mm	7.20 mm	1.80
6.3 mm[D]	¼ in.[D]	0.250	±.200 mm	6.64 mm	6.78 mm	1.80
5.6 mm	No. 3½[E]	0.223	±.180 mm	5.90 mm	6.04 mm	1.60
4.75 mm	No. 4	0.187	±.150 mm	5.02 mm	5.14 mm	1.60
4.00 mm	No. 5	0.157	±.130 mm	4.23 mm	4.35 mm	1.40
3.35 mm	No. 6	0.132	±.110 mm	3.55 mm	3.66 mm	1.25
2.80 mm	No. 7	0.110	±.095 mm	2.975 mm	3.070 mm	1.12
2.36 mm	No. 8	0.0937	±.080 mm	2.515 mm	2.600 mm	1.00
2.00 mm	No. 10	0.0787	±.070 mm	2.135 mm	2.215 mm	0.900
1.7 mm	No. 12	0.0661	±.060 mm	1.820 mm	1.890 mm	0.800
1.4 mm	No. 14	0.0555	±.050 mm	1.505 mm	1.565 mm	0.710
1.18 mm	No. 16	0.0469	±.045 mm	1.270 mm	1.330 mm	0.630
1.00 mm	No. 18	0.0394	±.040 mm	1.080 mm	1.135 mm	0.560
850 µm[F]	No. 20	0.0331	±35 µm	925 µm	970 µm	0.500
710 µm	No. 25	0.0278	±30 µm	775 µm	815 µm	0.450
600 µm	No. 30	0.0234	±25 µm	660 µm	695 µm	0.400
500 µm	No. 35	0.0197	±20 µm	550 µm	585 µm	0.315
425 µm	No. 40	0.0165	±19 µm	471 µm	502 µm	0.280
355 µm	No. 45	0.0139	±16 µm	396 µm	426 µm	0.224
300 µm	No. 50	0.0117	±14 µm	337 µm	363 µm	0.200
250 µm	No. 60	0.0098	±12 µm	283 µm	306 µm	0.160
212 µm	No. 70	0.0083	±10 µm	242 µm	263 µm	0.140
180 µm	No. 80	0.0070	±9 µm	207 µm	227 µm	0.125
150 µm	No. 100	0.0059	±8 µm	174 µm	192 µm	0.100
125 µm	No. 120	0.0049	±7 µm	147 µm	163 µm	0.090
106 µm	No. 140	0.0041	±6 µm	126 µm	141 µm	0.071
90 µm	No. 170	0.0035	±5 µm	108 µm	122 µm	0.063
75 µm	No. 200	0.0029	±5 µm	91 µm	103 µm	0.050
63 µm	No. 230	0.0025	±4 µm	77 µm	89 µm	0.045
53 µm	No. 270	0.0021	±4 µm	66 µm	76 µm	0.036
45 µm	No. 325	0.0017	±3 µm	57 µm	66 µm	0.032
38 µm	No. 400	0.0015	±3 µm	48 µm	57 µm	0.030
32 µm	No. 450	0.0012	±3 µm	42 µm	50 µm	0.028
25 µm[D]	No. 500	0.0010	±3 µm	34 µm	41 µm	0.025
20 µm[D]	No. 635	0.0008	±3 µm	29 µm	35 µm	0.020

[A] These standard designations correspond to the values for test sieve openings recommended by the International Standards Organization, Geneva, Switzerland, except where noted.

[B] Only approximately equivalent to the metric values in Column 1.

[C] The average diameter of the wires in the x and y direction, measured separately, of any wire cloth shall not deviate from the nominal values by more than ±15 %.

[D] These sieves are not in the standard series but they have been included because they are in common usage.

[E] These numbers (3½ to 635) are the approximate number of openings per linear in. but it is preferred that the sieve be identified by the standard designation in millimetres or micrometres.

[F] 1000 µm—1 mm.

shall be made along the midpoints of the opening as shown in Fig. 1.

4.4 Sieve cloth shall conform to the dimensional requirements of Table 1. The average opening (distance between parallel wires measured at the center of the opening), in the *x* (horizontal) and *y* (vertical) directions measured separately, shall conform to the values in Column 1, within the permissible variation in average opening size shown in Column 4. Not more than 5 % of the openings shall exceed the value shown in Column 5. The maximum individual

TABLE 2 Dimensions of Standard Frames

Nominal Diameter	Mean Diameter, in. (mm)		Typical Frame[A]
in.	Inside at Top[B]	Outside on Skirt	Nominal Height[C] in. (mm)
3	3.000 + 0.030/−0.000	3.000 + 0.000/−0.030	1¼ (32) FH[D]
	(76 + 0.76/ −0.00)	(76 + 0.00/ −0.76)	⅝ (16) HH
6	6.000 + 0.030/−0.000	6.000 + 0.000/−0.030	1¾ (45) FH
	(152 + 0.76/ −0.00)	(152 + 0.00/ −0.76)	1 (25) HH
8	8.000 + 0.030/−0.000	8.000 + 0.000/−0.030)	2 (50) FH
	(203 + 0.76/ −0.00	(203 + 0.00/ −0.76)	1 (25) HH
10	10.000 + 0.030/−0.000	10.000 + 0.000/−0.030	3 (76) FH
	(254 + 0.76/ −0.00)	(254 + 0.00/ −0.76)	1½ (38) HH
12	12.000 + 0.030/−0.000	12.000 + 0.000/−0.030	3¼ (83) FH
	(305 + 0.76/ −0.00)	(305 + 0.00/ −0.76)	2 (50) IH
			1⅝ (41) HH

[A] Other frame heights are not precluded.

[B] Measured 0.2 in. (5 mm) below the top of the frame.

[C] Distance from the top of the frame to the sieve cloth surface.

[D] FH = full height; HH = half height; IH = intermediate height.

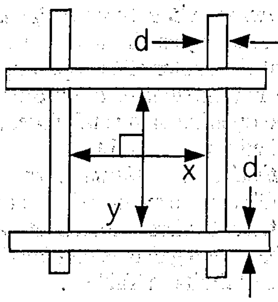

FIG. 1 Proper Dimensioning of Wire Cloth Mesh

opening size shall not exceed the value shown in Column 6.

4.4.1 The average diameter of the x (horizontal) and y (vertical) wires, measured separately, shall conform to the diameter in Column 7 within the tolerances in Footnote A of Table 1.

4.5 Wires shall be crimped in such a manner that they will be rigid when in use.

4.6 There shall be no punctures or obvious defects in the cloth.

5. Test Sieve Frames

5.1 *General Requirements*—Frames for wire cloth sieves shall be constructed in such a manner as to be rigid. The wire cloth shall be mounted on the frame without distortion, looseness, or waviness. To prevent the material being sieved from catching in the joint between the wire cloth and the frame, the joint shall be filled smoothly or constructed so that the material will not be trapped.

5.2 *Standard Frames*—Sieve frames shall be circular with nominal diameters of 3, 6, 8, 10, or 12 in. (76, 152, 203, 254, or 305 mm) as may be specified. The dimensions shall conform to the requirements in Table 2. Frames shall be made from noncorrosive material such as brass or stainless steel and be of seamless construction.

5.2.1 The bottom of the frame shall be constructed so as to provide an easy sliding fit with any sieve frame of the same nominal diameter conforming to the specified dimensions.

5.2.2 The joint or fillet at the connection of the sieve cloth to the frame will provide a minimum clear sieving surface with a diameter equal to the nominal diameter less 0.5 in. (13 mm).

NOTE 3—Attention is called to Test Method C 430, which contains requirements for 2 in. (51 mm) diameter sieves used in the mineral industry, especially the cement group.

5.3 *Nonstandard Frames*—Other sieve frames may be either square, rectangular, or circular. The frame may have the sieve cloth permanently installed, or may be designed to permit replacement. The provisions of 5.1 apply.

NOTE 4—While there are no requirements for nesting of nonstandard sieve frames, care should be applied in use to prevent loss of material during analysis.

5.4 *Pans and Covers*—Pans and covers for use with sieves shall be made so as to nest with the sieves. Pans with extended rims ("stacking skirts") shall be furnished when specified. The pans and covers shall conform to the dimensions in Table 2.

6. Product Marking

6.1 Each test sieve shall bear a label marked with the following information:

6.1.1 U.S.A. standard testing sieve,

6.1.2 This designation (ASTM E-11),

6.1.3 Standard sieve designation (from Table 1, Column 1),

6.1.4 Name of manufacturer or distributor, and

6.1.5 Alternative sieve designation (from Table 1, Column 2) (optional).

7. Keywords

7.1 opening; particle size; sieve; sieve analysis; sieve cloth; sieve designation; test grade wire cloth; test sieve

SUPPLEMENTARY REQUIREMENTS

The following supplementary requirements shall apply only when specified by the purchaser in the contract or order.

S1. Responsibility for Inspection

S1.1 Unless otherwise specified in the contract or purchase order, the producer is responsible for the performance of all inspection and test requirements specified herein. Except as otherwise specified in the contract or order, the producer may use his own or any other suitable facilities for the performance of the inspection and test requirements specified herein, unless disapproved by the purchaser. The purchaser shall have the right to perform any of the inspections and tests set forth in this specification where such inspections are deemed necessary to ensure that materials meet the specification.

S2. Government Procurement

S2.1 Unless otherwise specified in the contract, the materials shall be packaged in accordance with the suppliers' standard practice that will be acceptable to the carrier at lowest rates. Containers and packing shall comply with the Uniform Freight Classification rules or National Motor Freight Classification rules. Marking for shipment of such materials shall be in accordance with Fed. Std. No. 123 for civil agencies, and MIL-STD-129 for military agencies.

ANNEX

(Mandatory Information)

A1. TEST METHODS FOR CHECKING WIRE CLOTH AND TESTING SIEVES TO DETERMINE WHETHER THEY CONFORM TO SPECIFICATION

A1.1 Every opening in the metal wire cloth in a test sieve shall be eligible for inspection for compliance with the requirements listed in Table 1.

A1.1.1 When a sieve has 30 openings or less, measure all openings. In other cases the examination shall proceed in stages from a survey of general condition, to a methodical scrutiny of individual openings, and finally to measurement of opening size for compliance with the tolerances.

A1.1.2 Measure opening size, as described in Test Methods Two, Three, and Four, on equipment with a precision of at least 2.5 µm or 10 % of the value in Column 4 for the specific mesh designation, whichever is greater.

A1.2 *Test Method One—Examination of General Condition of the Wire Cloth*—For this purpose, view the sieve cloth against a uniformly illuminated background. If obvious deviations, for example, weaving defects, creases, wrinkles, foreign matter in the cloth, are found, the wire cloth is unacceptable.

A1.3 *Test Method Two—Examination for Maximum Individual Opening*—The observer shall carefully and methodically examine the appearance of all the openings, in order to detect oversize openings. Openings whose width deviates by about 10 % of the average value are apparent to the unaided eye of a skilled observer. By this test method, known as the "handicap method", it is probable that all oversize openings exceeding the average value by about 10 % or more will be detected. At the same time it is easily possible to detect sequences of large openings, and local irregularities in the weaving, appearing as distortions in the openings. If an opening is found to be larger than that permissible in accordance with Column 6 of Table 1, the wire cloth is unacceptable.

A1.4 *Test Method Three—Determination of the Size Distribution of Wire Cloth Openings*—To establish the size distribution of sieve openings, determine the frequency of opening size measurements using the following procedures:

A1.4.1 For samples (testing sieves or wire cloth) with 30 or less openings, measure all full openings. For samples with over 30 openings, measure a minimum of 30 full openings.

A1.4.2 Select openings in a line or lines diagonal to the direction of the wires according to Fig. A1.1, and measure ten adjacent openings along each line. When greater numbers of openings are available, choose the fields in such a manner that none of the openings being measured overlap.

A1.4.3 *Measurement of the Average Opening Size*—Measure the average opening as the distance between parallel wires (measured at the center of the opening—see Fig. 1) in both directions, being sure to keep the x and y measurements separate. Once the opening data is tabulated, check the data versus the prescribed limits in Table 1.

A1.5 *Test Method Four—Measurement of the Average Wire Diameter*—Obtain the average diameter of the wires by measuring 30 different wires selected at random in each direction. Once the opening data is tabulated, check the data versus the prescribed limits in Table 1.

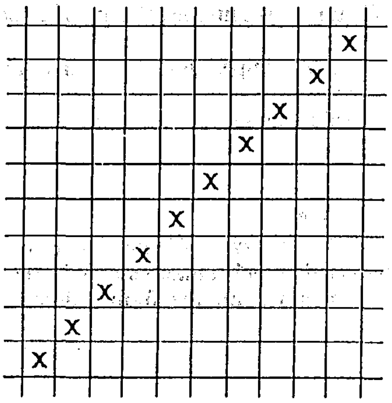

FIG. A1.1 Orientation of Openings to be Measured in Each Field

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Standard Methods for
NOTCHED BAR IMPACT TESTING OF METALLIC MATERIALS[1]

This standard is issued under the fixed designation E 23; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

These methods have been approved for use by agencies of the Department of Defense to replace method 221.1 of Federal Test Method Standard No. 151b and for listing in the DoD Index of Specifications and Standards.

NOTE—Figures 2, 3, 4, 5, 6, 7, 11, 12, 13, 14, 15, and 16 were editorially corrected, and the designation date was changed March 5, 1982.

1. Scope

1.1 These methods describe notched-bar impact testing of metallic materials by the Charpy (simple-beam) apparatus and the Izod (cantilever-beam) apparatus. They give: (*a*) a description of apparatus, (*b*) requirements for inspection and calibration, (*c*) safety precautions, (*d*) sampling, (*e*) dimensions and preparation of specimens, (*f*) testing procedures, (*g*) precision and accuracy, and (*h*) appended notes on the significance of notched-bar impact testing. These methods will in most cases also apply to tests on unnotched specimens.

1.2 The values stated in SI units are to be regarded as the standard.

2. Summary of Methods

2.1 The essential features of an impact test are: (*a*) a suitable specimen (specimens of several different types are recognized), (*b*) an anvil or support on which the test specimen is placed to receive the blow of the moving mass, (*c*) a moving mass of known kinetic energy which must be great enough to break the test specimen placed in its path, and (*d*) a device for measuring the energy absorbed by the broken specimen.

3. Significance

3.1 These methods of impact testing relate specifically to the behavior of metal when subjected to a single application of a load resulting in multiaxial stresses associated with a notch, coupled with high rates of loading and in some cases with high or low temperatures. For some materials and temperatures, impact tests on notched specimens have been found to predict the likelihood of brittle fracture better than tension tests or other tests used in material specifications. Further information on significance appears in the Appendix.

4. Apparatus

4.1 *General Requirements:*

4.1.1 The testing machine shall be a pendulum type of rigid construction and of capacity more than sufficient to break the specimen in one blow.

4.1.2 The machine frame shall be equipped with a bubble level or a machined surface suitable for establishing levelness. The machine shall be level to within 3:1000 and securely bolted to a concrete floor not less than 150 mm (6 in.) thick or, when this is not practical, the machine shall be bolted to a foundation having a mass not less than 40 times that of the pendulum. The bolts shall be tightened as specified by the machine manufacturer.

4.1.3 The machine shall be furnished with scales graduated either in degrees or directly in energy on which readings can be estimated in increments of 0.25 % of the energy range or less. The scales may be compensated for wind-

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[1] These methods are under the jurisdiction of ASTM Committee E-28 on Mechanical Testing and are the direct responsibility of Subcommittee E28.07 on Impact Testing.

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age and pendulum friction. The error in the scale reading at any point shall not exceed 0.2 % of the range or 0.4 % of the reading, whichever is larger. (See 5.2.6.2 and 5.2.7.)

4.1.4 The total friction and windage losses of the machine during the swing in the striking direction shall not exceed 0.75 % of the scale range capacity, and pendulum energy loss from friction in the indicating mechanism shall not exceed 0.25 % of scale range capacity.

4.1.5 The dimensions of the pendulum shall be such that the center of percussion of the pendulum is at the center of strike within 1 % of the distance from the axis of rotation to the center of strike. When hanging free, the pendulum shall hang so that the striking edge is within 2.5 mm (0.10 in.) of the position where it would just touch the test specimen. When the indicator has been positioned to read zero energy in a free swing, it shall read within 0.2 % of scale range when the striking edge of the pendulum is held against the test specimen. The plane of swing of the pendulum shall be perpendicular to the transverse axis of the Charpy specimen anvils or Izod vise within 3: 1000.

4.1.6 Transverse play of the pendulum at the striker shall not exceed 0.75 mm (0.030 in.) under a transverse force of 4 % of the effective weight of the pendulum applied at the center of strike. Radial play of the pendulum bearings shall not exceed 0.075 mm (0.003 in.). The tangential velocity (the impact velocity) of the pendulum at the center of the strike shall not be less than 3 nor more than 6 m/s (not less than 10 nor more than 20 ft/s).

4.1.7 Before release, the height of the center of strike above its free hanging position shall be within 0.4 % of the range capacity divided by the pendulum weight, measured as described in 5.2.3.3. If windage and friction are compensated for by increasing the height of drop, the height of drop may be increased by not more than 1 %.

4.1.8 The mechanism for releasing the pendulum from its initial position shall operate freely and permit release of the pendulum without initial impulse, retardation, or side vibration. If the same lever that is used to release the pendulum is also used to engage the brake, means shall be provided for preventing the brake from being accidentally engaged.

4.2 *Specimen Clearance*—To ensure satisfactory results when testing materials of different strengths and compositions, the test specimen shall be free to leave the machine with a minimum of interference and shall not rebound into the pendulum before the pendulum completes its swing. Pendulums used on Charpy machines are of two basic designs, as shown in Fig. 1. When using a C-type pendulum, the broken specimen will not rebound into the pendulum and slow it down if the clearance at the end of the specimen is at least 13 mm (0.5 in.) or if the specimen is deflected out of the machine by some arrangement as is shown in Fig. 1. When using the U-type pendulum, means shall be provided to prevent the broken specimen from rebounding against the pendulum (Fig. 1). In most U-type pendulum machines, the shrouds should be designed and installed to the following requirements: (*a*) have a thickness of approximately 1.5 mm (0.06 in.), (*b*) have a minimum hardness of 45 HRC, (*c*) have a radius of less than 1.5 mm (0.06 in.) at the underside corners, and (*d*) be so positioned that the clearance between them and the pendulum overhang (both top and sides) does not exceed 1.5 mm (0.06 in.).

NOTE 1—In machines where the opening within the pendulum permits clearance between the ends of a specimen (resting on the anvil supports) and the shrouds, and this clearance is at least 13 mm (0.5 in.) requirements (*a*) and (*d*) need not apply.

4.3 *Charpy Apparatus:*

4.3.1 Means shall be provided (Fig. 2) to locate and support the test specimen against two anvil blocks in such a position that the center of the notch can be located within 0.25 mm (0.010 in.) of the midpoint between the anvils (see 11.2.1.2).

4.3.2 The supports and striking edge shall be of the forms and dimensions shown in Fig. 2. Other dimensions of the pendulum and supports should be such as to minimize interference between the pendulum and broken specimens.

4.3.3 The center line of the striking edge shall advance in the plane that is within 0.40 mm (0.016 in.) of the midpoint between the supporting edges of the specimen anvils. The striking edge shall be perpendicular to the longitudinal axis of the specimen within 5:1000. The striking edge shall be parallel within 1: 1000 to the face of a perfectly square test specimen held against the anvil.

4.3.4 Specimen supports shall be square with anvil faces within 2.5:1000. Specimen supports shall be coplanar within 0.125 mm (0.005 in.) and parallel within 2:1000.

4.4 *Izod Apparatus:*

4.4.1 Means shall be provided (Fig. 3) for clamping the specimen in such a position that the face of the specimen is parallel to the striking edge within 1:1000. The edges of the clamping surfaces shall be sharp angles of 90 ± 1° with radii less than 0.40 mm (0.016 in.). The clamping surfaces shall be smooth with a 2-μm (63-μin.) finish or better, and shall clamp the specimen firmly at the notch with the clamping force applied in the direction of impact. For rectangular specimens, the clamping surfaces shall be flat and parallel within 0.025 mm (0.001 in.). For cylindral specimens, the clamping surfaces shall be contoured to match the specimen and each surface shall contact a minimum of $\pi/2$ rad (90°) of the specimen circumference.

4.4.2 The dimensions of the striking edge and its position relative to the specimen clamps shall be as shown in Fig. 3.

4.5 *Energy Range*—Energy values above 80 % of the scale range are inaccurate and shall be reported as approximate. Ideally an impact test would be conducted at a constant impact velocity. In a pendulum-type test, the velocity decreases as the fracture progresses. For specimens that have impact energies approaching the capacity of the pendulum, the velocity of the pendulum decreases during fracture to the point that accurate impact energies are no longer obtained.

5. Inspection

5.1 *Critical Parts:*

5.1.1 *Specimen Anvils and Supports or Vise*—These shall conform to the dimensions shown in Fig. 2 or 3. To ensure a minimum of energy loss through absorption, bolts shall be tightened as specified by the machine manufacturer.

NOTE 2—The impact machine will be inaccurate to the extent that some energy is used in deformation or movement of its component parts or of the machine as a whole; this energy will be registered as used in fracturing the specimen.

5.1.2 *Pendulum Striking Edge*—The striking edge (tup) of the pendulum shall conform to the dimensions shown in Figs. 2 or 3. To ensure a minimum of energy loss through absorption, the striking edge bolts shall be tightened as specified by the machine manufacturer. The pendulum striking edge (tup) shall comply with 4.3.3 (for Charpy tests) or 4.4.1 (for Izod tests) by bringing it into contact with a standard Charpy or Izod specimen.

5.2 *Pendulum Operation:*

5.2.1 *Pendulum Release Mechanism*—The mechanism for releasing the pendulum from its initial position shall comply with 4.1.8.

5.2.2 *Pendulum Alignment*—The pendulum shall comply with 4.1.5 and 4.1.6. If the side play in the pendulum or the radial plays in the bearings exceeds the specified limits, adjust or replace the bearings.

5.2.3 *Potential Energy*—Determine the initial potential energy using the following procedure when the center of strike of the pendulum is coincident with the line from the center of rotation through the center of percussion. If the center of strike is more than 2.5 mm (0.1 in.) from this line, suitable corrections in elevation of the center of strike must be made in 5.2.3.2, 5.2.3.3, 5.2.6.1, and 5.2.7, so that elevations set or measured correspond to what they would be if the center of strike were on this line.

5.2.3.1 For Charpy machines place a half-width specimen (see Fig. 4) 10 by 5 mm (0.394 by 0.197 in.) in test position. With the striking edge in contact with the specimen, a line scribed from the top edge of the specimen to the striking edge will indicate the center of strike on the striking edge.

5.2.3.2 For Izod machines, the center of strike may be considered to be the contact line when the pendulum is brought into contact with a specimen in the normal testing position.

NOTE 3—A method of accurately determining the centers of strike of Izod machines is to place a specimen, so machined that the distance from the center of the notch to the top of the specimen is 22.66 mm (0.892 in.), in test position. With the striking edge in contact with the specimen, a line scribed from the top edge of the specimen to the striking edge will indicate the center of strike on the striking edge.

5.2.3.3 Support the pendulum horizontally to within 15:1000 with two supports, one at the bearings (or center of rotation) and the other at the center of strike on the striking edge (see Fig. 5). Arrange the support at the striking edge to react upon some suitable weighing device such as a platform scale or balance, and determine the weight to within 0.4 %. Take care to minimize friction at either point of support.

Make contact with the striking edge through a round rod crossing the edge at a 90° angle. The weight of the pendulum is the scale reading minus the weights of the supporting rod and any shims that may be used to maintain the pendulum in a horizontal position.

5.2.3.4 Measure the height of pendulum drop for compliance with the requirement of 4.1.7. On Charpy machines measure the height from the top edge of a half-width (or center of a full-width) specimen to the elevated position of the center of strike to 0.1 %. On Izod machines measure the height from a distance 22.66 mm (0.892 in.) above the vise to the release position of the center of strike to 0.1 %.

5.2.3.5 The potential energy of the system is equal to the height from which the pendulum falls, as determined in 5.2.3.4, times the weight of the pendulum, as determined in 5.2.3.3.

5.2.4 *Impact Velocity*—Determine the impact velocity, v, of the machine, neglecting friction, by means of the following equation:

$$v = \sqrt{2gh}$$

where:

v = velocity, m/s (or ft/s),

g = acceleration of gravity, m/s^2 (or ft/s^2), and

h = initial elevation of the striking edge, m (or ft).

5.2.5 *Center of Percussion*—To ensure that minimum force is transmitted to the point of rotation, the center of percussion shall be at a point within 1 % of the distance from the axis of rotation to the center of strike in the specimen. Determine the location of the center of percussion as follows:

5.2.5.1 Using a stop watch or some other suitable time-measuring device, capable of measuring time to within 0.2 s, swing the pendulum through a total angle not greater than 15° and record the time for 100 complete cycles (to and fro).

5.2.5.2 Determine the center of percussion by means of the following equation:

$l = 0.2484p^2$, to determine l in metres

$l = 0.815p^2$, to determine l in feet

where:

l = distance from the axis to the center of percussion, m (or ft), and

p = time of a complete cycle (to and fro) of the pendulum, s.

5.2.6 *Friction*—The energy loss from friction and windage of the pendulum and friction in the recording mechanism, if not corrected, will be included in the energy loss attributed to breaking the specimen and can result in erroneously high impact values. In machines recording in degrees, normal frictional losses are usually not compensated for by the machine manufacturer, whereas they are usually compensated for in machines recording directly in energy by increasing the starting height of the pendulum. Determine energy losses from friction as follows:

5.2.6.1 Without a specimen in the machine, and with the indicator at the maximum energy reading, release the pendulum from its starting position and record the energy value indicated. This value should indicate zero energy if frictional losses have been corrected by the manufacturer. Raise the pendulum so it just contacts the pointer at the value obtained in the free swing. Secure the pendulum at this height and determine the vertical distance from the center of strike to the top of a half-width specimen positioned on the specimen rests (see 5.2.3.1). Determine the weight of the pendulum as in 5.2.3.2 and multiply by this distance. The difference in this value and the initial potential energy is the total energy loss in the pendulum and indicator combined. Without resetting the pointer, repeatedly release the pendulum from its initial position until the pointer shows no further movement. The energy loss determined by the final position of the pointer is that due to the pendulum alone. The frictional loss in the indicator alone is then the difference between the combined indicator and pendulum losses and those due to the pendulum alone.

5.2.6.2 To ensure that friction and windage losses are within tolerances allowed (see 4.1.4), a simple weekly procedure may be adopted for direct-reading machines. The following steps are recommended: (*a*) release the pendulum from its upright position without a specimen in the machine, and the energy reading should be 0 J (0 ft·lbf); (*b*) without resetting the pointer, again release the pendulum and permit it to swing 11 half cycles; and after the pendulum starts its 11th cycle, move the pointer to between 5 and 10 % of scale range capacity and record the value obtained. This value, divided by 11, shall not exceed 0.4 % of scale range capacity. If this value does exceed 0.4 %, the bearings should be cleaned or replaced.

5.2.7 *Indicating Mechanism*—To ensure that the scale is recording accurately over the entire range, check it at graduation marks corresponding to approximately 0, 10, 20, 30, 50, and 70 % of each range. With the striking edge of the pendulum scribed to indicate the center of strike, lift the pendulum and set it in a position where the indicator reads, for example, 13 J (10 ft·lbf). Determine the height of the pendulum to within 0.1 %. The height of the pendulum multiplied by its weight, as determined in 5.2.3.3, is the residual energy. Increase this value by friction and windage losses in accordance with 5.2.6 and subtract from the potential energy determined in 5.2.3. Make similar calculations at other points of the scale. The scale pointer shall not overshoot or drop back with the pendulum. Make test swings from various heights to check visually the operation of the pointer over several portions of the scale.

5.2.8 The impact value shall be taken as the energy absorbed in breaking the specimen and is equal to the difference between the energy in the striking member at the instant of impact with the specimen and the energy remaining after breaking the specimen.

6. Precaution in Operation of Machine

6.1 *Safety Precautions*—Precautions should be taken to protect personnel from the swinging pendulum, flying broken specimens, and hazards associated with specimen warming and cooling media.

7. Sampling

7.1 Specimens shall be taken from the material as specified by the applicable specification.

8. Test Specimens

8.1 *Material Dependence*—The choice of specimen depends to some extent upon the characteristics of the material to be tested. A given specimen may not be equally satisfactory for soft nonferrous metals and hardened steels; therefore, a number of types of specimens are recognized. In general, sharper and deeper notches are required to distinguish differences in the more ductile materials or with lower testing velocities.

8.1.1 The specimens shown in Figs. 6 and 7 are those most widely used and most generally satisfactory. They are particularly suitable for ferrous metals, excepting cast iron.[2]

8.1.2 The specimen commonly found suitable for die cast alloys is shown in Fig. 8.

8.1.3 The specimens commonly found suitable for powdered metals (P/M) are shown in Figs. 9 and 10. The specimen surface may be in the as-produced condition or smoothly machined, but polishing has proven generally unnecessary. Unnotched specimens are used with P/M materials. In P/M materials, the impact test results will be affected by specimen orientation. Therefore, unless otherwise specified, the position of the specimen in the machine shall be such that the pendulum will strike a surface that is parallel to the compacting direction.

8.2 *Sub-Size Specimen*—When the amount of material available does not permit making the standard impact test specimens shown in Figs. 6 and 7, smaller specimens may be used, but the results obtained on different sizes of specimens cannot be compared directly (X1.3). When Charpy specimens other than the standard are necessary or specified, it is recommended that they be selected from Fig. 4.

8.3 *Supplementary Specimens*—For economy in preparation of test specimens, special specimens of round or rectangular cross section are sometimes used for cantilever beam test. These are shown as Specimens X, Y, and Z in Figs. 11 and 12. Specimen Z is sometimes called the Philpot specimen after the name of the original designer. In the case of hard materials, the machining of the flat surface struck by the pendulum is sometimes omitted. Types Y and Z require a different vise from that shown in Fig. 3, each half of the vise having a semicylindrical recess that closely fits the clamped portion of the specimen. As previously stated, the results cannot be reliably compared to those obtained using specimens of other sizes or shapes.

8.4 *Specimen Machining:*

8.4.1 When heat-treated materials are being evaluated, the specimen shall be finish machined, including notching, after the final heat treatment, unless it can be demonstrated that

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[2] For testing cast iron, see 1933 Report of Subcommittee XV on Impact Testing of Committee A-3 on Cast Iron, *Proceedings*, Am. Soc. Testing Mats., Vol 33, Part 1, 1933.

there is no difference when machined prior to heat treatment.

8.4.2 Notches shall be smoothly machined but polishing has proven generally unnecessary. However, since variations in notch dimensions will seriously affect the results of the tests, it is necessary to adhere to the tolerances given in Fig. 6 (X1.2 illustrates the effects from varying notch dimensions on Type A specimens). In keyhole specimens, the round hole shall be carefully drilled with a slow feed. The slot may be cut by any feasible method. Care must be exercised in cutting the slot to see that the surface of the drilled hole opposite the slot is not marked.

8.4.3 Identification marks shall not be placed on any surface of the specimen that contacts the striking edge or specimen supports. All stamping shall be done in a way that avoids cold deforming of the specimen at the notch root or at any other portion of the specimen that is visibly deformed during fracture.

9. Preparation of Apparatus

9.1 *Daily Checking Procedure*—After the testing machine has been ascertained to comply with Sections 4 and 5, the routine daily checking procedures shall be as follows:

9.1.1 Prior to testing a group of specimens and before a specimen is placed in position to be tested, check the machine by a free swing of the pendulum. With the indicator at the maximum energy position, a free swing of the pendulum shall indicate zero energy on machines reading directly in energy, which are compensated for frictional losses. On machines recording in degrees, the indicated values when converted to energy shall be compensated for frictional losses that are assumed to be proportional to the arc of swing.

10. Verification of Charpy Machines

10.1 Verification consists of inspecting those parts subjected to wear to ensure that the requirements of Sections 4 and 5 are met and the testing of standardized specimens (Notes 4 to 6). It is not intended that parts not subjected to wear (such as pendulum and scale linearity) need to be remeasured during verification unless a problem is evident. The average value at each energy level determined for the standardized specimens shall correspond to the nominal values of the standardized specimens within 1.4 J (1.0 ft·lbf) or 5.0 %, whichever is greater.

NOTE 4—Standardized specimens are available for Charpy machines only.

NOTE 5—Information pertaining to the availability of standardized specimens may be obtained by addressing: Director, Army Materials and Mechanics Research Center, ATTN: DRXMR-MQ, Watertown, Mass. 02172.

NOTE 6—The Army Materials and Mechanics Research Center has for many years conducted a Charpy machine qualification program whereby standardized specimens are used to certify the machines of laboratories using the test as an inspection requirement on government contracts.[3] If the user desires, the results of tests with the standardized specimens will be evaluated. Participants desirous of the evaluation should complete the questionnaire provided with the standardized specimens. The questionnaire provides for information such as testing temperature, the dimensions of certain critical parts, the cooling and testing techniques, and the results of the test. The broken standardized specimens are to be returned along with the completed questionnaire for evaluation (see Note 5 for address). Upon completion of the evaluation, the Army Materials and Mechanics Research Center will return a report. If a machine is producing values outside the standardized specimen tolerances, the report may suggest changes in machine design, repair or replacement of certain machine parts, a change in testing techniques, etc.

10.2 *Frequency of Verification*—Charpy machines shall be verified within one year prior to the time of testing. Charpy machines shall, however, be verified immediately after replacing parts, making repairs or adjustments, after they have been moved, or whenever there is reason to doubt the accuracy of the results, without regard to the time interval.

11. Procedure

11.1 The Daily Checking Procedure (Section 9) shall be performed at the beginning of each day or each shift.

11.2 *Charpy Test Procedure*—The Charpy test procedure may be summarized as follows: the test specimen is removed from its cooling (or heating) medium, if used, and positioned on the specimen supports; the pendulum is released without vibration, and the specimen is broken within 5 s after removal from the medium. Information is obtained from the machine and from the broken specimen. The details are described as follows:

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[3] Driscoll, D. E., "Reproducibility of Charpy Impact Test," *Symposium on Impact Testing, ASTM STP 176*, Am. Soc. Testing Mats., 1955, p. 170.

11.2.1 *Temperature of Testing*—In most materials, impact values vary with temperature. Unless otherwise specified, tests shall be made at 15 to 32°C (60 to 90°F). Accuracy of results when testing at other temperatures requires the following procedure: For liquid cooling or heating fill a suitable container, which has a grid raised at least 25 mm (1 in.) from the bottom, with liquid so that the specimen when immersed will be covered with at least 25 mm (1 in.) of the liquid. Bring the liquid to the desired temperature by any convenient method. The device used to measure the temperature of the bath should be placed in the center of a group of the specimens. Verify all temperature-measuring equipment at least twice annually. When using a liquid medium, hold the specimens in an agitated bath at the desired temperature within ±1°C (±2°F) for at least 5 min. When using a gas medium, position the specimens so that the gas circulates around them and hold the gas at the desired temperature within ±1°C (±2°F) for at least 30 min. Leave the mechanism used to remove the specimen from the medium in the medium except when handling the specimens.

NOTE 7—Temperatures up to +260°C (+500°F) may be obtained with certain oils, but "flash-point" temperatures must be carefully observed.

11.2.2 *Placement of Test Specimen in Machine*—It is recommended that self-centering tongs similar to those shown in Fig. 13 be used in placing the specimen in the machine (see 4.3.1). The tongs illustrated in Fig. 13 are for centering V-notch specimens. If keyhole specimens are used, modification of the tong design may be necessary. If an end-centering device is used, caution must be taken to ensure that low-energy high-strength specimens will not rebound off this device into the pendulum and cause erroneously high recorded values. Many such devices are permanent fixtures of machines, and if the clearance between the end of a specimen in test position and the centering device is not approximately 13 mm (0.5 in.), the broken specimens may rebound into the pendulum.

11.2.3 *Operation of the Machine:*

11.2.3.1 Set the energy indicator at the maximum scale reading; take the test specimen from its cooling (or heating) medium, if used; place it in proper position on the specimen anvils; and release the pendulum smoothly. This entire sequence shall take less than 5 s if a cooling or heating medium is used.

11.2.3.2 If any specimen fails to break, do not repeat the blow but record the fact, indicating whether the failure to break occurred through extreme ductility or lack of sufficient energy in the blow. Such results of such tests shall not be included in the average.

11.2.3.3 If any specimen jams in the machine, disregard the results and check the machine thoroughly for damage or maladjustment, which would affect its calibration.

11.2.3.4 To prevent recording an erroneous value caused by jarring the indicator when locking the pendulum in its upright position, read the value from the indicator prior to locking the pendulum for the next test.

11.2.4 *Information Obtainable from the Test*:

11.2.4.1 *Impact Energy*—The amount of energy required to fracture the specimen is determined from the machine reading.

11.2.4.2 *Lateral Expansion*—The method for measuring lateral expansion must take into account the fact that the fracture path seldom bisects the point of maximum expansion on both sides of a specimen. One half of a broken specimen may include the maximum expansion for both sides, one side only, on neither. The technique used must therefore provide an expansion value equal to the sum of the higher of the two values obtained for each side by measuring the two halves separately. The amount of expansion on each side of each half must be measured relative to the plane defined by the undeformed portion of the side of the specimen, Fig. 16. Expansion may be measured by using a gage similar to that shown in Figs. 17 and 18. Measure the two broken halves individually. First, though, check the sides perpendicular to the notch to ensure that no burrs were formed on these sides during impact testing; if such burrs exist, they must be removed, for example, by rubbing on emery cloth, making sure that the protrusions to be measured are not rubbed during the removal of the burr. Next, place the halves together so that the compression sides are facing one another. Take one half and press it firmly against the reference supports, with the protrusion against the gage anvil. Note the reading, then repeat this step with the other broken half, ensuring that the same side of the specimen is measured. The larger of the two values is the expansion of that side of the specimen. Next, repeat this procedure to measure the protrusions on the opposite side, then add the larger values obtained for each side. Measure each specimen.

NOTE 8—Examine each fracture surface to ascertain that the protrusions have not been damaged by contacting the anvil, machine mounting surface, etc. Such specimens should be discarded since this may cause erroneous readings.

11.2.4.3 *Fracture Appearance*—The percentage of shear fracture may be determined by any of the following methods: (*1*) measure the length and width of the cleavage portion of the fracture surface, as shown in Fig. 14, and determine the percent shear from either Table 1 or Table 2 depending on the units of measurement; (*2*) compare the appearance of the fracture of the specimen with a fracture appearance chart such as that shown in Fig. 15; (*3*) magnify the fracture surface and compare it to a precalibrated overlay chart or measure the percent shear fracture by means of a planimeter; or (*4*) photograph the fracture surface at a suitable magnification and measure the percent shear fracture by means of a planimeter.

NOTE 9—Because of the subjective nature of the evaluation of fracture appearance, it is not recommended that it be used in specifications.

11.3 *Izod Test Procedure*—The Izod test procedure may be summarized as follows: the test specimen is positioned in the specimen-holding fixture and the pendulum is released without vibration. Information is obtained from the machine and from the broken specimen. The details are described as follows:

11.3.1 *Temperature of Testing*—The specimen-holding fixture for Izod specimens is in most cases part of the base of the machine and cannot be readily cooled (or heated). For this reason, Izod testing is not recommended at other than room temperature.

11.3.2 Clamp the specimen firmly in the support vise so that the centerline of the notch is in the plane of the top of the vise within 0.125 mm (0.005 in.). Set the energy indicator at the maximum scale reading, and release the pendulum smoothly. Sections 11.2.3.2 to 11.2.3.4 inclusively, also apply when testing Izod specimens.

11.3.3 *Information Obtainable from the Test*—The impact energy, lateral expansion, and fracture appearance, may be determined as described in 11.2.4.

12. Report

12.1 For commercial acceptance testing, the following is considered sufficient:

12.1.1 Type of specimen used (and size if not the standard size).

12.1.2 Temperature of the specimen.

12.1.3 When required any or all of the following shall be reported:

12.1.3.1 Energy absorbed,

12.1.3.2 Lateral expansion, and

12.1.3.3 Fracture appearance (see Note 9).

13. Precision and Accuracy

13.1 The precision and accuracy of these methods are being established.

TABLE 1 Percent Shear for Measurements Made in Millimetres

NOTE—100 % shear is to be reported when either *A* or *B* is zero.

Dimension *B*, mm	Dimension *A*, mm																		
	1.0	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0	5.5	6.0	6.5	7.0	7.5	8.0	8.5	9.0	9.5	10
1.0	99	98	98	97	96	96	95	94	94	93	92	92	91	91	90	89	89	88	88
1.5	98	97	96	95	94	93	92	92	91	90	89	88	87	86	85	84	83	82	81
2.0	98	96	95	94	92	91	90	89	88	86	85	84	82	81	80	79	77	76	75
2.5	97	95	94	92	91	89	88	86	84	83	81	80	78	77	75	73	72	70	69
3.0	96	94	92	91	89	87	85	83	81	79	77	76	74	72	70	68	66	64	62
3.5	96	93	91	89	87	85	82	80	78	76	74	72	69	67	65	63	61	58	56
4.0	95	92	90	88	85	82	80	77	75	72	70	67	65	62	60	57	55	52	50
4.5	94	92	89	86	83	80	77	75	72	69	66	63	61	58	55	52	49	46	44
5.0	94	91	88	85	81	78	75	72	69	66	62	59	56	53	50	47	44	41	37
5.5	93	90	96	83	79	76	72	69	66	62	59	55	52	48	45	42	38	35	31
6.0	92	89	85	81	77	74	70	66	62	59	55	51	47	44	40	36	33	29	25
6.5	92	88	84	80	76	72	67	63	59	55	51	47	43	39	35	31	27	23	19
7.0	91	87	82	78	74	69	65	61	56	52	47	43	39	34	30	26	21	17	12
7.5	91	86	81	77	72	67	62	58	53	48	44	39	34	30	25	20	16	11	6
8.0	90	85	80	75	70	65	60	55	50	45	40	35	30	25	20	15	10	5	0

TABLE 2 Percent Shear for Measurements Made in Inches

NOTE—100 % shear is to be reported when either *A* or *B* is zero.

Dimension *B*, in.	Dimension *A*, in.																
	0.05	0.10	0.12	0.14	0.16	0.18	0.20	0.22	0.24	0.26	0.28	0.30	0.32	0.34	0.36	0.38	0.40
0.05	98	96	95	94	94	93	92	91	90	90	89	88	87	86	85	85	84
0.10	96	92	90	89	87	85	84	82	81	79	77	76	74	73	71	69	68
0.12	95	90	88	86	85	83	81	79	77	75	73	71	69	67	65	63	61
0.14	94	89	86	84	82	80	77	75	73	71	68	66	64	62	59	57	55
0.16	94	87	85	82	79	77	74	72	69	67	64	61	59	56	53	51	48
0.18	93	85	83	80	77	74	72	68	65	62	59	56	54	51	48	45	42
0.20	92	84	81	77	74	72	68	65	61	58	55	52	48	45	42	39	36
0.22	91	82	79	75	72	68	65	61	57	54	50	47	43	40	36	33	29
0.24	90	81	77	73	69	65	61	57	54	50	46	42	38	34	30	27	23
0.26	90	79	75	71	67	62	58	54	50	46	41	37	33	29	25	20	16
0.28	89	77	73	68	64	59	55	50	46	41	37	32	28	23	18	14	10
0.30	88	76	71	66	61	56	52	47	42	37	32	27	23	18	13	9	3
0.31	88	75	70	65	60	55	50	45	40	35	30	25	20	18	10	5	0

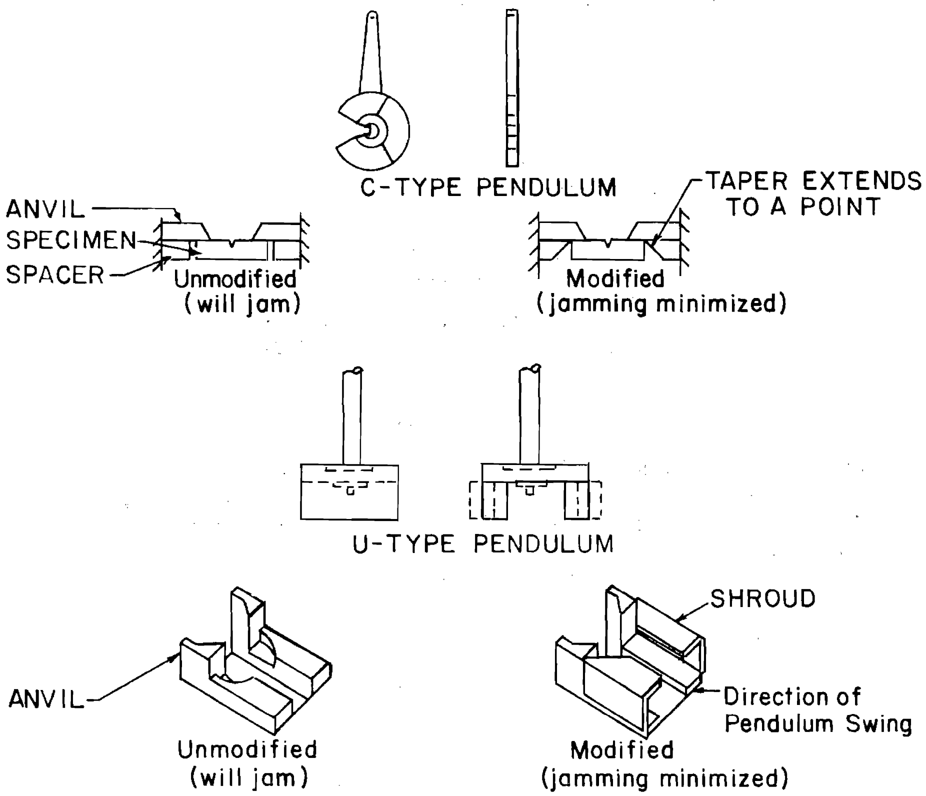
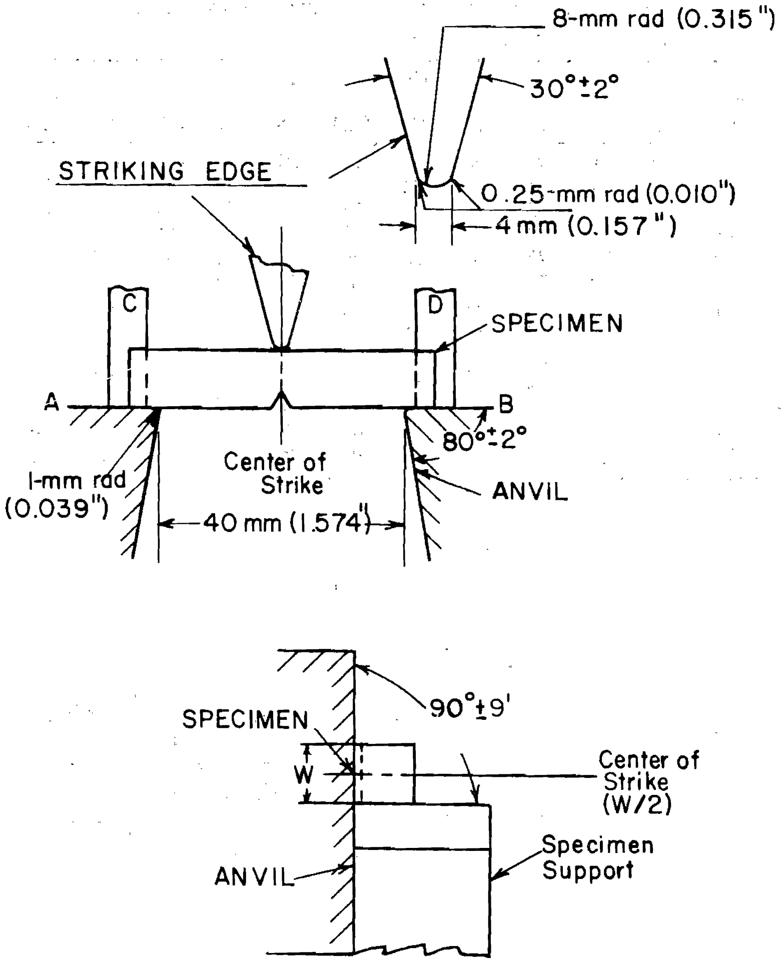


FIG. 1 Typical Pendulums and Anvils for Charpy Machines, Shown with Modifications to Minimize Jamming



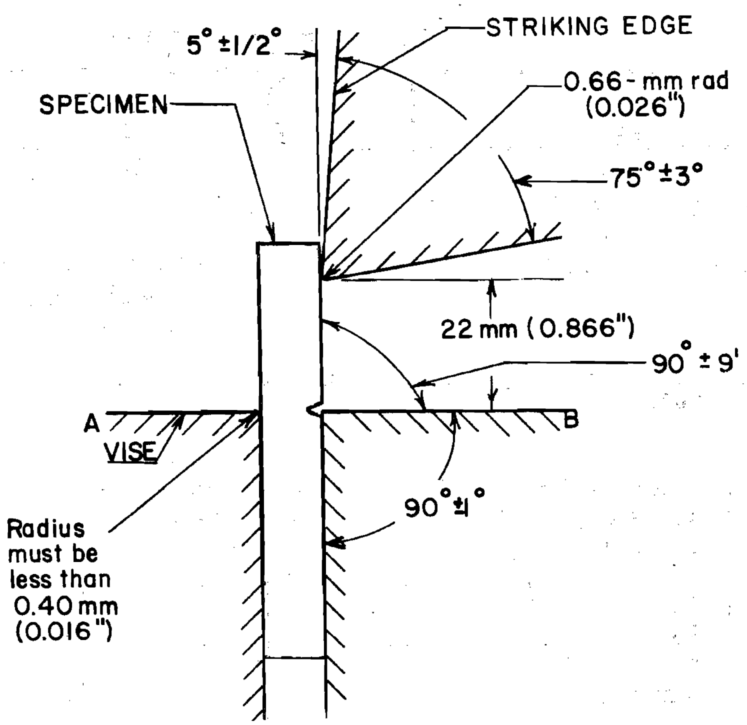
All dimensional tolerances shall be ±0.05 mm (0.002 in.) unless otherwise specified.

NOTE 1—A shall be parallel to B within 2:1000 and coplanar with B within 0.05 mm (0.002 in.).

NOTE 2—C shall be parallel to D within 2.0:1000 and coplanar with D within 0.125 mm (0.005 in.).

NOTE 3—Finish on unmarked parts shall be 4 μm (125 μin.).

FIG. 2 Charpy (Simple-Beam) Impact Test



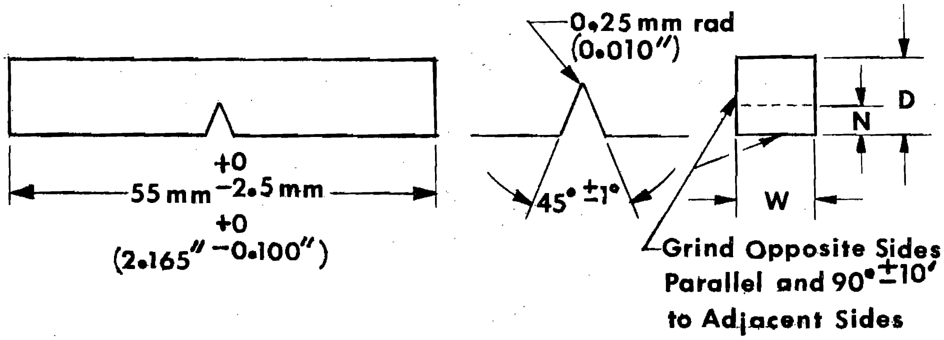
All dimensional tolerances shall be ±0.05 mm (0.002 in.) unless otherwise specified.

NOTE 1—The clamping surfaces of A and B shall be flat and parallel within 0.025 mm (0.001 in.).

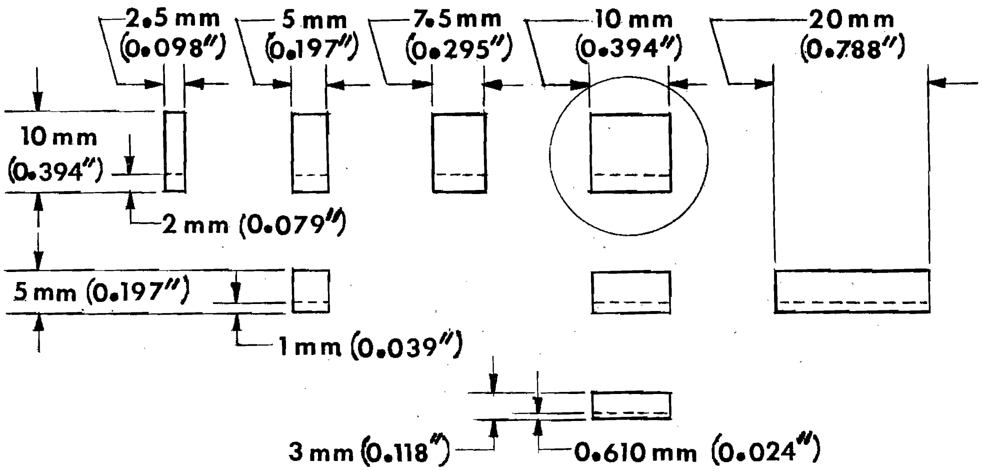
NOTE 2—Finish on unmarked parts shall be 2 μm (63 μin.).

NOTE 3—Striker width must be greater than that of the specimen being tested.

FIG. 3 Izod (Cantilever-Beam) Impact Test



On subsize specimens the length, notch angle, and notch radius are constant (see Fig. 6); depth (*D*), notch depth (*N*), and width (*W*) vary as indicated below.



NOTE 1—Circled specimen is the standard specimen (see Fig. 6).

NOTE 2—Permissible variations shall be as follows:

Cross-section dimensions	±1% or ±0.075 mm (0.003 in.), whichever is smaller
Radius of notch	±0.025 mm (0.001 in.)
Depth of notch	±0.025 mm (0.001 in.)
Finish requirements	2 µm (63 µin.) on notched surface and opposite face; 4 µm (125 µin.) on other two surfaces

FIG. 4 Charpy (Simple-Beam) Subsize (Type A) Impact Test Specimens

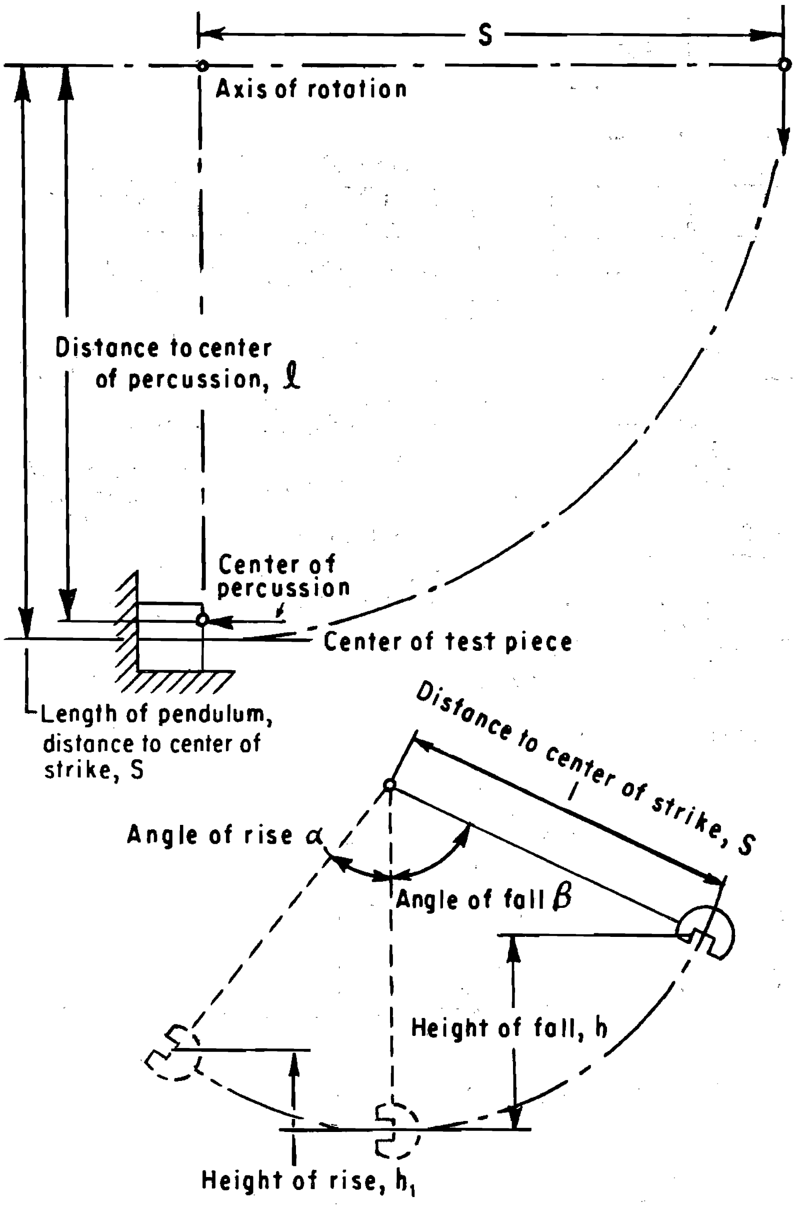
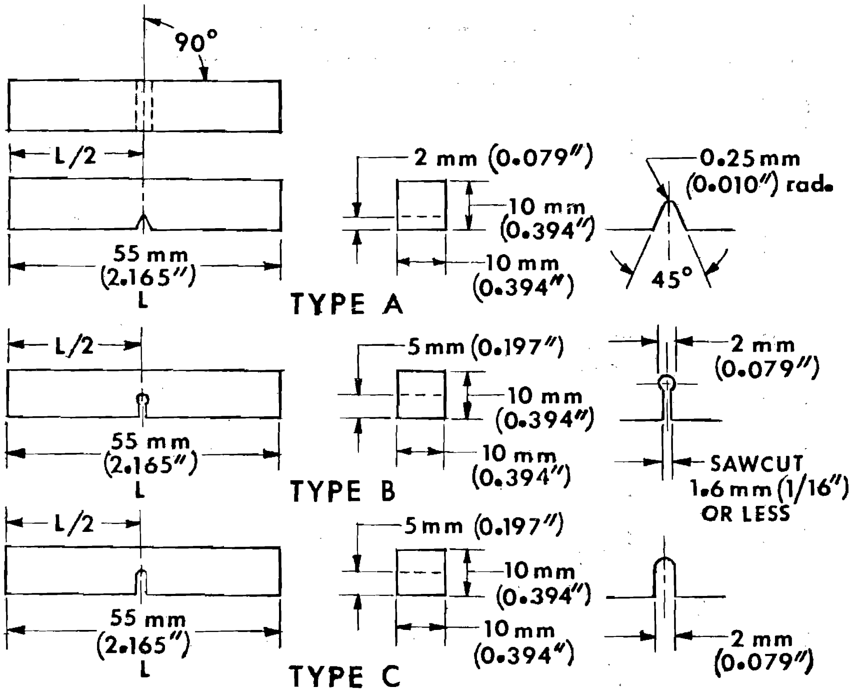


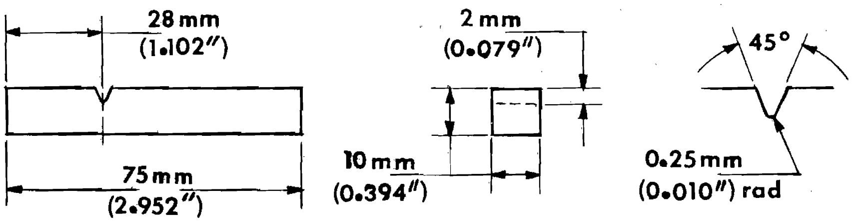
FIG. 5 Dimensions for Calculations



NOTE—Permissible variations shall be as follows:

Notch length to edge	±2°
Adjacent sides shall be at	90° ±10 min
Cross-section dimensions	±0.075 mm (±0.003 in.)
Length of specimen (*L*)	+0, −2.5 mm (+0, −0.100 in.)
Centering of notch (*L*/2)	±1 mm (±0.039 in.)
Angle of notch	±1°
Radius of notch	±0.025 mm (±0.001 in.)
Notch depth:	
Type A specimen	±0.025 mm (±0.001 in.)
Types B and C specimen	±0.075 mm (±0.003 in.)
Finish requirements	2 μm (63 μin.) on notched surface and opposite face; 4 μm (125 μin.) on other two surfaces

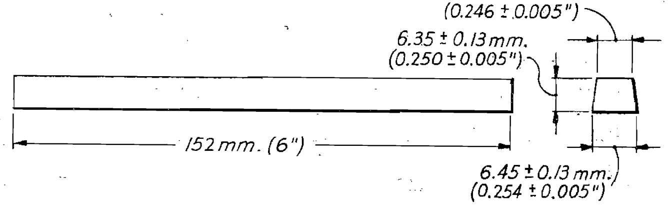
FIG. 6 Charpy (Simple-Beam) Impact Test Specimens, Types A, B, and C



NOTE—Permissible variations shall be as follows:

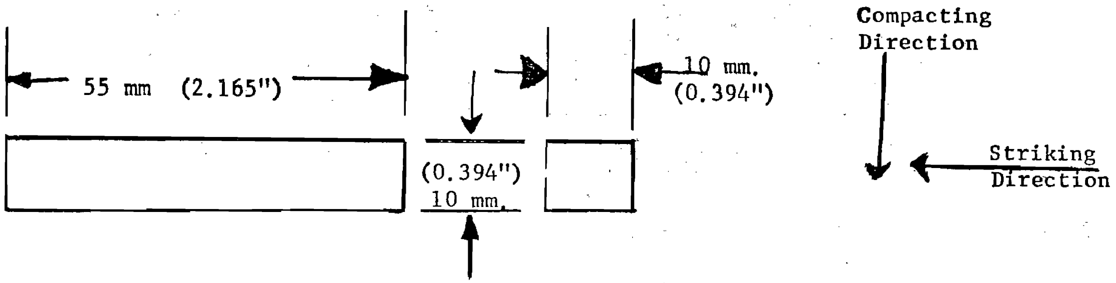
Notch length to edge	90 ±2°
Cross-section dimensions	±0.025 mm (±0.001 in.)
Length of specimen	+0, −2.5 mm (±0, −0.100 in.)
Angle of notch	±1°
Radius of notch	±0.025 mm (±0.001 in.)
Notch depth	±0.025 mm (±0.001 in.)
Adjacent sides shall be at	90° ± 10 min
Finish requirements	2 μm (63 μin.) on notched surface and opposite face; 4 μm (125 μin.) on other two surfaces

FIG. 7 Izod (Cantilever-Beam) Impact Test Specimen, Type D



NOTE 1—Two test specimens may be cut from this bar.
NOTE 2—Blow shall be struck on narrowest face.

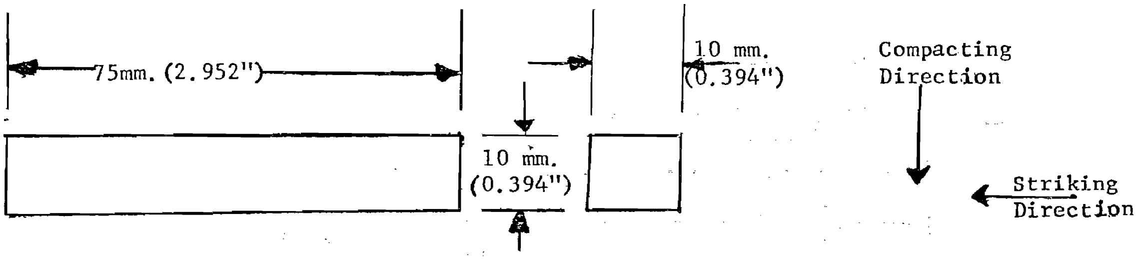
FIG. 8 Simple Beam Impact Test Bar for Die Castings Alloys



NOTE—Permissible variations shall be as follows:

Adjacent sides shall be at	90° ± 10 min
Cross section dimensions	±0.125 mm (0.005 in.)
Length of specimen	±0, −2.5 mm (0.100 in.)

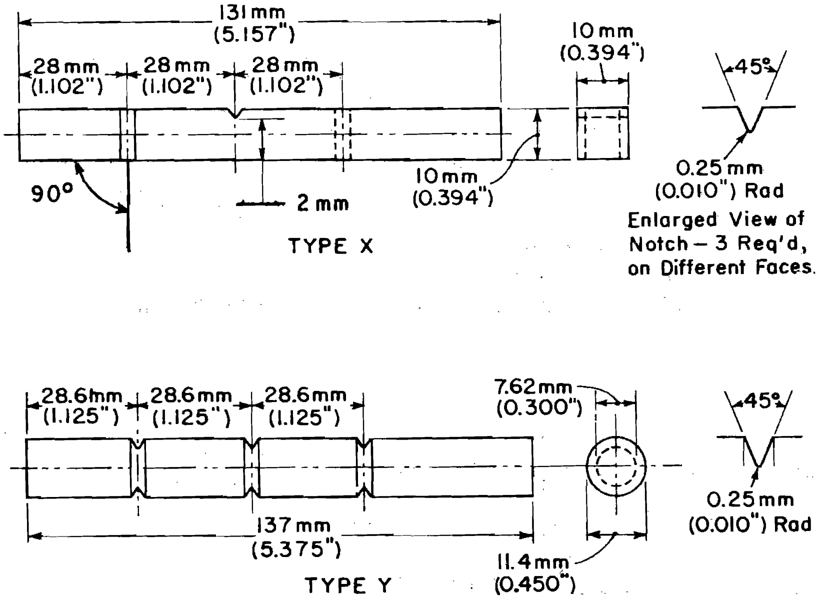
FIG. 9 Charpy (Simple Beam) Impact Test Specimens for Metal Powder Structural Parts



NOTE—Permissible variations shall be as follows:

Adjacent sides shall be at	90° ± 10 min.
Cross section dimensions	±0.125 mm (0.005 in.)
Length of specimens	+0, −2.5 mm (0.100 in.)

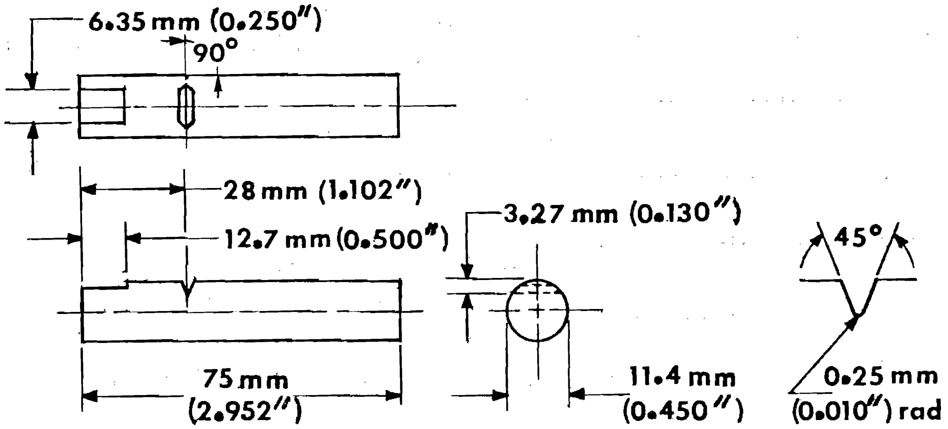
FIG. 10 Izod (Cantilever-Beam) Impact Test Specimen for Metal Powder Structural Parts



NOTE—Permissible variations shall be as follows:

Notch length to edge	±2 mm
Adjacent sides shall be at	90° ±10 min
Cross-section dimensions	±0.025 mm (±0.001 in.)
Lengthwise dimensions	+0, −2.5 mm (±0.100 in.)
Angle of notch	±1°
Radius of notch	±0.025 mm (±0.001 in.)
Notch depth of Type X specimen	±0.025 mm (±0.001 in.)
Notch diameter of Type Y specimen	±0.025 mm (±0.001 in.)

FIG. 11 Izod (Cantilever-Beam) Impact Test Specimens, Types X and Y

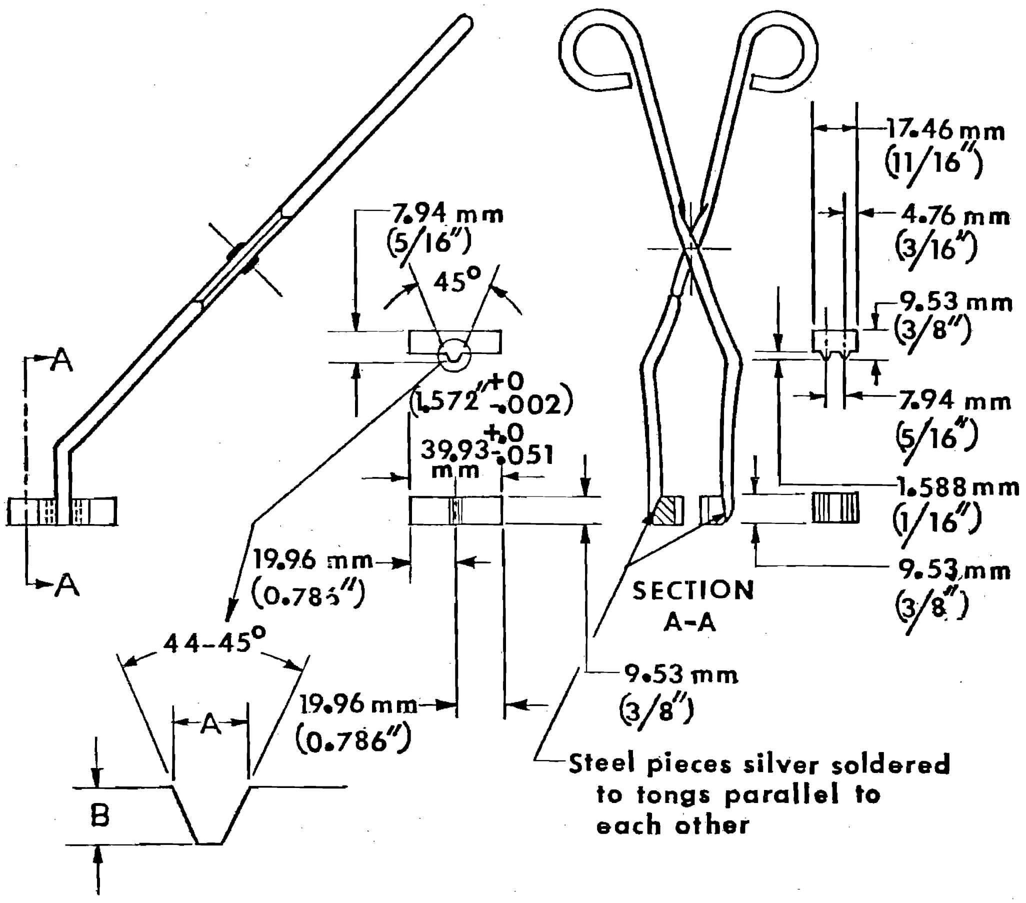


The flat shall be parallel to the longitudinal centerline of the specimen and shall be parallel to the bottom of the notch within 2:1000.

NOTE—Permissible variations shall be as follows:

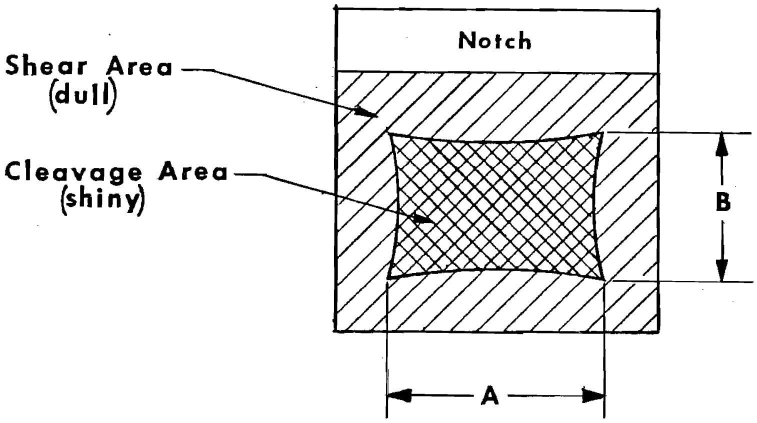
Notch length to longitudinal centerline	±2°
Cross-section dimensions	±0.025 mm (−0.001 in.)
Length of specimen	+0, −2.5 mm (+0 −0.100 in.)
Angle of notch	±1°
Radius of notch	±0.025 mm (±0.001 in.)
Notch depth	±0.025 mm (.130 ±0.001 in.)

FIG. 12 Izod (Cantilever-Beam) Impact Test Specimen (Philpot), Type Z



Specimen Depth, mm (in.)	Base Width (A), mm (in.)	Height (B), mm (in.)
10 (0.394)	1.60 to 1.70 (0.063 to 0.067)	1.52 to 1.65 (0.060 to 0.065)
5 (0.197)	0.74 to 0.80 (0.029 to 0.033)	0.69 to 0.81 (0.027 to 0.032)
3 (0.118)	0.45 to 0.51 (0.016 to 0.020)	0.36 to 0.48 (0.014 to 0.019)

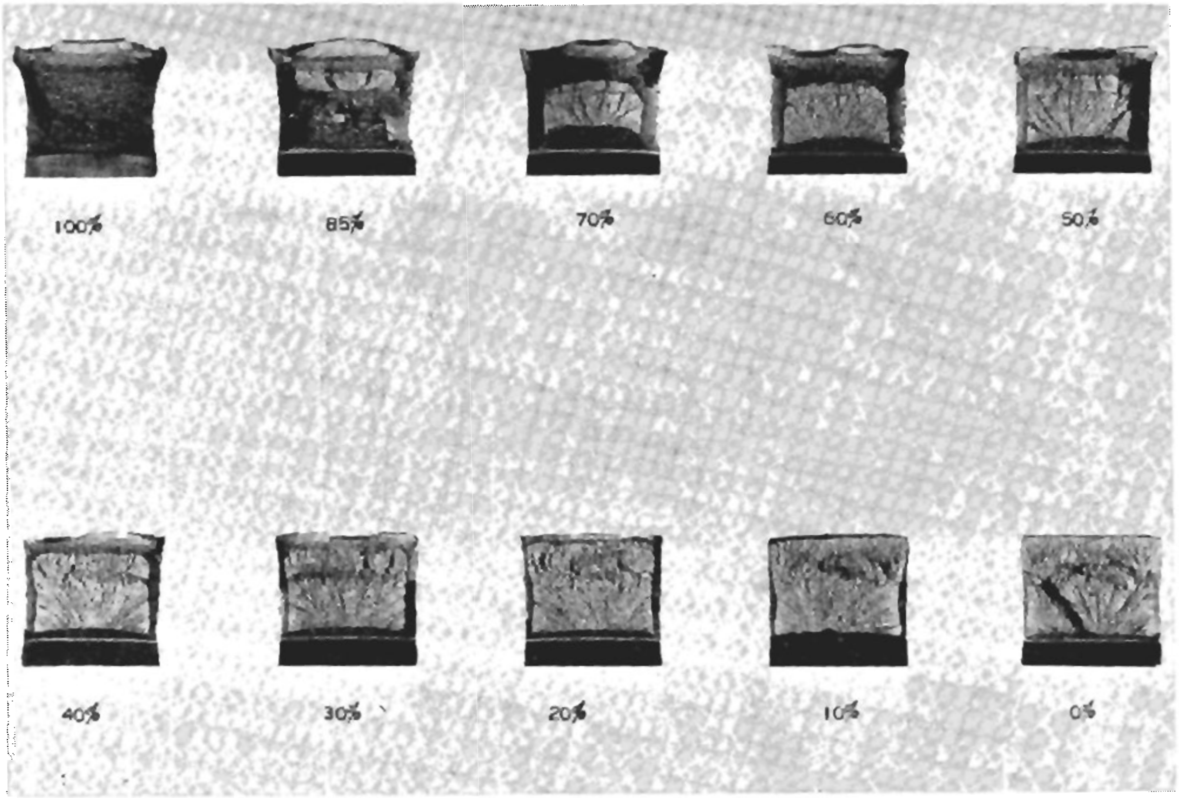
FIG. 13 Centering Tongs for V-Notch Charpy Specimens



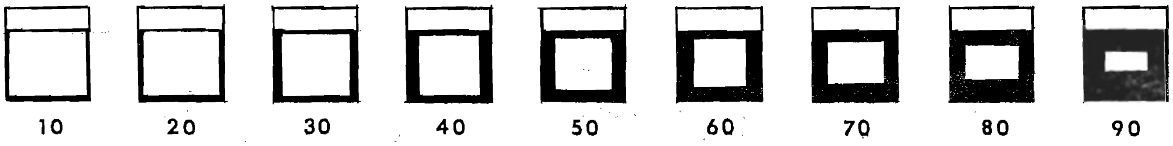
NOTE 1—Measure average dimensions *A* and *B* to the nearest 0.5 mm or 0.02 in.

NOTE 2—Determine the percent shear fracture using Table 1 or Table 2.

FIG. 14 Determination of Percent Shear Fracture



(*a*) Fracture Appearance Charts and Percent Shear Fracture Comparator



(*b*) Guide for Estimating Fracture Appearance Using SuLAG Method

FIG. 15 Fracture Appearance

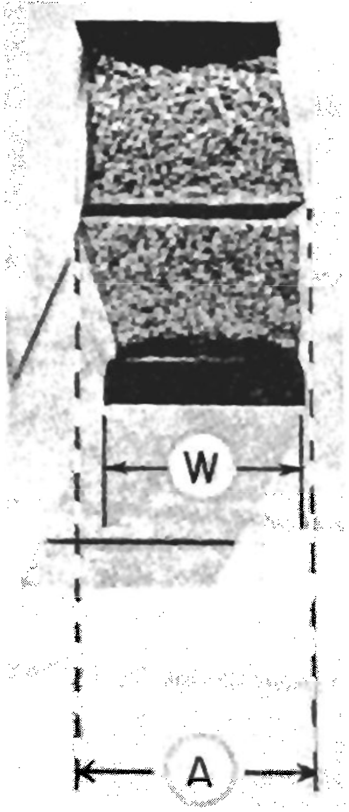


FIG. 16 Halves of Broken Charpy V-Notch Impact Specimen Positioned to Illustrate the Measurement of Lateral Expansion, Dimension *A* and Original Width, Dimension *W*

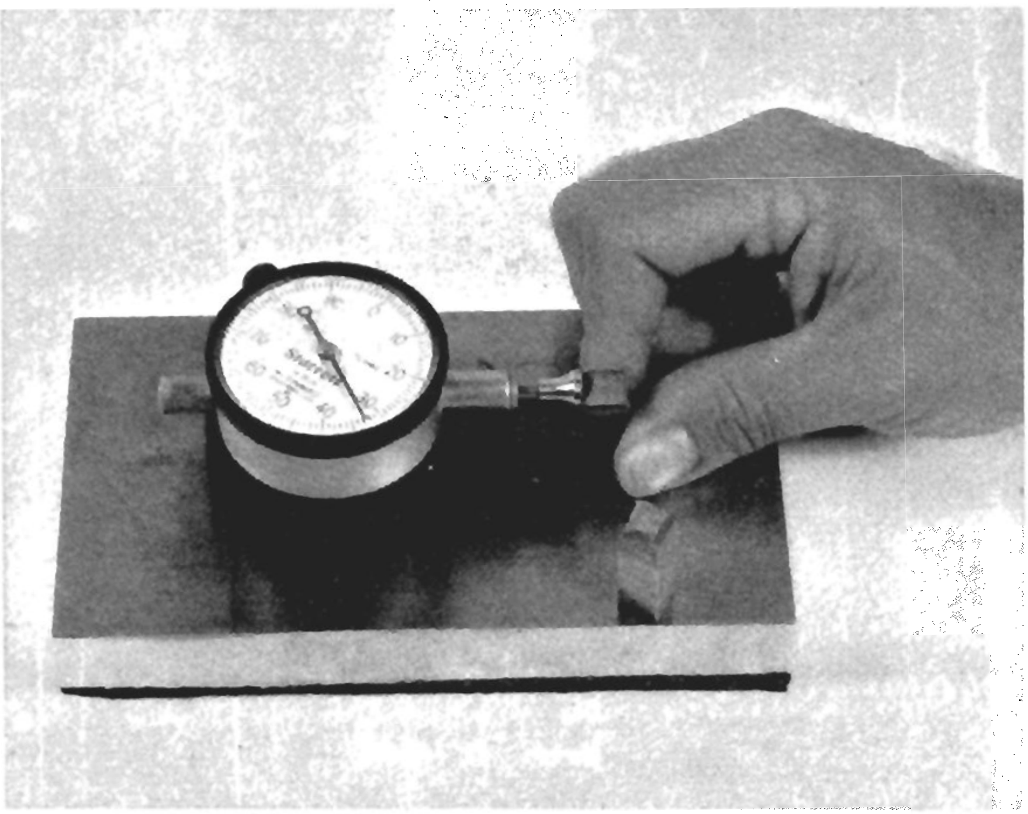
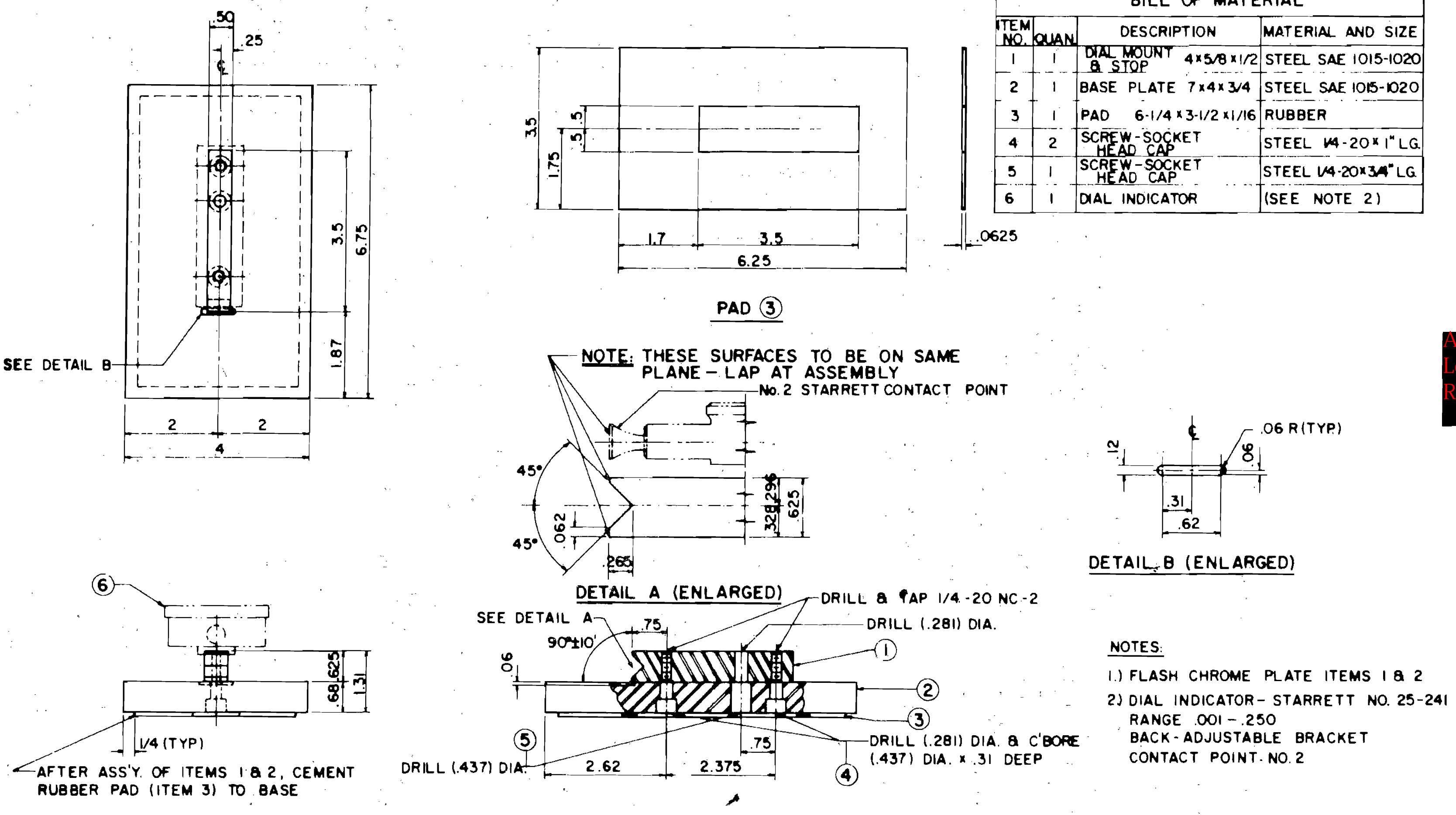


FIG. 17 Lateral Expansion Gage for Charpy Impact Specimens



NOTE: THESE SURFACES TO BE ON SAME PLANE – LAP AT ASSEMBLY

BILL OF MATERIAL

ITEM NO.	QUAN.	DESCRIPTION	MATERIAL AND SIZE
1	1	DIAL MOUNT & STOP 4×5/8×1/2	STEEL SAE 1015-1020
2	1	BASE PLATE 7×4×3/4	STEEL SAE 1015-1020
3	1	PAD 6-1/4×3-1/2×1/16	RUBBER
4	2	SCREW-SOCKET HEAD CAP	STEEL 1/4-20×1" LG.
5	1	SCREW-SOCKET HEAD CAP	STEEL 1/4-20×3/4" LG.
6	1	DIAL INDICATOR	(SEE NOTE 2)

NOTES:

1.) FLASH CHROME PLATE ITEMS 1 & 2

2.) DIAL INDICATOR – STARRETT NO. 25-241
RANGE .001 – .250
BACK-ADJUSTABLE BRACKET
CONTACT POINT NO. 2

FIG. 18 Assembly and Details for Lateral Expansion Gage

APPENDIX

X1. NOTES ON SIGNIFICANCE OF NOTCHED-BAR IMPACT TESTING

X1.1 Notch Behavior

X1.1.1 The Charpy and Izod type tests bring out notch behavior (brittleness versus ductility) by applying a single overload of stress. The energy values determined are quantitative comparisons on a selected specimen but cannot be converted into energy values that would serve for engineering desgin calculations. The notch behavior indicated in an individual test applies only to the specimen size, notch geometry, and testing conditions involved and cannot be generalized to other sizes of specimens and conditions.

X1.1.2 The notch behavior of the face-centered cubic metals and alloys, a large group of nonferrous materials and the austenitic steels can be judged from their common tensile properties. If they are brittle in tension they will be brittle when notched, while if they are ductile in tension they will be ductile when notched, except for unusually sharp or deep notches (much more severe than the standard Charpy or Izod specimens). Even low temperatures do not alter this characteristic of these materials. In contrast, the behavior of the ferritic steels under notch conditions cannot be predicted from their properties as revealed by the tension test. For the study of these materials the Charpy and Izod type tests are accordingly very useful. Some metals that display normal ductility in the tension test may nevertheless break in brittle fashion when tested or when used in the notched condition. Notched conditions include restraints to deformation in directions perpendicular to the major stress, or multiaxial stresses, and stress concentrations. It is in this field that the Charpy and Izod tests prove useful for determining the susceptibility of a steel to notch-brittle behavior though they cannot be directly used to appraise the serviceability of a structure.

X1.2 Notch Effect

X1.2.1 The notch results in a combination of multiaxial stresses associated with restraints to deformation in directions perpendicular to the major stress, and a stress concentration at the base of the notch. A severely notched condition is generally not desirable, and it becomes of real concern in those cases in which it initiates a sudden and complete failure of the brittle type. Some metals can be deformed in a ductile manner even down to the low temperatures of liquid air, while others may crack. This difference in behavior can be best understood by considering the cohesive strength of a material (or the property that holds it together) and its relation to the yield point. In cases of brittle fracture, the cohesive strength is exceeded before significant plastic deformation occurs and the fracture appears crystalline. In cases of the ductile or shear type of failure, considerable deformation precedes the final fracture and the broken surface appears fibrous instead of crytalline. In intermediate cases the fracture comes after a moderate amount of deformation and is part crystalline and part fibrous in appearance.

X1.2.2 When a notched bar is loaded, there is a normal stress across the base of the notch which tends to initiate fracture. The property that keeps it from cleaving, or holds it together, is the "cohesive strength." The bar fractures when the normal stress exceeds the cohesive strength. When this occurs without the bar deforming it is the condition for brittle fracture.

X1.2.3 In testing, though not in service because of side effects, it happens more commonly that plastic deformation precedes fracture. In addition to the normal stress, the applied load also sets up shear stresses which are about 45° to the normal stress. The elastic behavior terminates as soon as the shear stress exceeds the shear strength of the material and deformation or plastic yielding sets in. This is the condition for ductile failure.

X.1.2.4 This behavior, whether brittle or ductile, depends on whether the normal stress exceeds the cohesive strength before the shear stress exceeds the shear strength. Several important facts of notch behavior follow from this. If the notch is made sharper or more drastic, the normal stress at the root of the notch will be increased in relation to the shear stress and the bar will be more prone to brittle fracture (see Table X1.1). Also, as the speed of deformation increases, the shear strength increases and the likelihood of brittle fracture increases. On the other hand, by raising the temperature, leaving the notch and the speed of deformation the same, the shear strength is lowered and ductile behavior is promoted, leading to shear failure.

X1.2.5 Variations in notch dimensions will seriously affect the results of the tests. Tests on E 4340 steel specimens[4] have shown the effect of dimensional variations on Charpy results (see Table X1.1).

X1.3 Size Effect

X1.3.1 Increasing either the width or the depth of the specimen tends to increase the volume of metal subject to distortion, and by this factor tends to

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[4] N. H. Fahey, "Effects of Variables in Charpy Impact Testing," *Materials Research & Standards*, Vol 1, No. 11, November 1961, p. 872.

increase the energy absorption when breaking the specimen. However, any increase in size, particularly in width, also tends to increase the degree of restraint and by tending to induce brittle fracture, may decrease the amount of energy absorbed. Where a standard-size specimen is on the verge of brittle fracture, this is particularly true, and a doublewidth specimen may actually require less energy for rupture than one of standard width.

X1.3.2 In studies of such effects where the size of the material precludes the use of the standard specimen, as for example when the material is 6.35 mm (0.25-in.) plate, subsize specimens are necessarily used. Such specimens (Fig. 4) are based on the Type A specimen of Fig. 6.

X1.3.3 General correlation between the energy values obtained with specimens of different size or shape is not feasible, but limited correlations may be established for specification purposes on the basis of special studies of particular materials and particular specimens. On the other hand, in a study of the relative effect of process variations, evaluation by use of some arbitrarily selected specimen with some chosen notch will in most instances place the methods in their proper order.

X1.4 Temperature Effect

X1.4.1 The testing conditions also affect the notch behavior. So pronounced is the effect of temperature on the behavior of steel when notched that comparisons are frequently made by examining specimen fractures and by plotting energy value and fracture appearance versus temperature from tests of notched bars at a series of temperatures. When the test temperature has been carried low enough to start cleavage fracture, there may be an extremely sharp drop in impact value or there may be a relatively gradual falling off toward the lower temperatures. This drop in energy value starts when a specimen begins to exhibit some crystalline appearance in the fracture. The transition temperature at which this embrittling effect takes place varies considerably with the size of the part or test specimen and with the notch geometry.

X1.5 Testing Machine

X1.5.1 The testing machine itself must be sufficiently rigid or tests on high-strength low-energy materials will result in excessive elastic energy losses either upward through the pendulum shaft or downward through the base of the machine. If the anvil supports, the pendulum striking edge, or the machine foundation bolts are not securely fastened, tests on ductile materials in the range from 108 J (80 ft·lbf) may actually indicate values in excess of 122 to 136 J (90 to 100 ft·lbf)

X1.5.2 A problem peculiar to Charpy-type tests occurs when high-strength, low-energy specimens are tested at low temperatures. These specimens may not leave the machine in the direction of the pendulum swing but rather in a sidewise direction. To ensure that the broken halves of the specimens do not rebound off some component of the machine and contact the pendulum before it completes its swing, modifications may be necessary in older model machines. These modifications differ with machine design. Nevertheless the basic problem is the same in that provisions must be made to prevent rebounding of the fractured specimens into any part of the swinging pendulum. Where design permits, the broken specimens may be deflected out of the sides of the machine and yet in other designs it may be necessary to contain the broken specimens within a certain area until the pendulum passes through the anvils. Some low-energy high-strength steel specimens leave impact machines at speeds in excess of 15.2 m/s (50 ft/s) although they were struck by a pendulum traveling at speeds approximately 5.2 m/s (17 ft/s). If the force exerted on the pendulum by the broken specimens is sufficient, the pendulum will slow down and erroneously high energy values will be recorded. This problem accounts for many of the inconsistencies in Charpy results reported by various investigators within the 14 to 34-J (10 to 25-ft·lb) range. Figure 1 illustrates a modification found to be satisfactory in minimizing jamming.

X1.6 Velocity of Straining

X1.6.1 Velocity of straining is likewise a variable that affects the notch behavior of steel. The impact test shows somewhat higher energy absorption values than the static tests above the transition temperature and yet, in some instances, the reverse is true below the transition temperature.

X1.7 Correlation with Service

X1.7.1 While Charpy or Izod tests may not directly predict the ductile or brittle behavior of steel as commonly used in large masses or as components of large structures, these tests can be used as acceptance tests or tests of identity for different lots of the same steel or in choosing between different steels, when correlation with reliable service behavior has been established. It may be necessary to make the tests at properly chosen temperatures other than room temperature. In this, the service temperature or the transition temperature of full-scale specimens does not give the desired transition temperatures for Charpy or Izod tests since the size and notch geometry may be so different. Chemical analysis, tension, and hardness tests may not indicate the influence of some of the important processing factors that affect susceptibility to brittle fracture nor do they comprehend the effect of low temperatures in inducing brittle behavior.

TABLE X1.1 Effect of Varying Notch Dimensions on Standard Specimens

	High-Energy Specimens, J (ft·lbf)	High-Energy Specimens, J (ft·lbf)	Low-Energy Specimens, J (ft·lbf)
Specimen with standard dimensions	103.0 ± 5.2 (76.0 ± 3.8)	60.3 ± 3.0 (44.5 ± 2.2)	16.9 ± 1.4 (12.5 ± 1.0)
Depth of notch, 2.13 mm (0.084 in.)[A]	97.9 (72.2)	56.0 (41.3)	15.5 (11.4)
Depth of notch, 2.04 mm (0.0805 in.)[A]	101.8 (75.1)	57.2 (42.2)	16.8 (12.4)
Depth of notch, 1.97 mm (0.0775 in.)[A]	104.1 (76.8)	61.4 (45.3)	17.2 (12.7)
Depth of notch, 1.88 mm (0.074 in.)[A]	107.9 (79.6)	62.4 (46.0)	17.4 (12.8)
Radius at base of notch 0.13 mm (0.005 in.)[B]	98.0 (72.3)	56.5 (41.7)	14.6 (10.8)
Radius at base of notch 0.38 mm (0.015 in.)[B]	108.5 (80.0)	64.3 (47.4)	21.4 (15.8)

[A] Standard 2.0 ± 0.025 mm (0.079 ± 0.001 in.).

[B] Standard 0.25 ± 0.025 mm (0.010 ± 0.001 in.).

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Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[1]

This standard is issued under the fixed designation E 29; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This practice is intended to assist the various technical committees in the use of uniform methods of indicating the number of digits which are to be considered significant in specification limits, for example, specified maximum values and specified minimum values. Its aim is to outline methods which should aid in clarifying the intended meaning of specification limits with which observed values or calculated test results are compared in determining conformance with specifications. Reference to this practice is valid only when a choice of method has been indicated, that is, either *absolute method or rounding-off method.*

1.2 This practice is intended to be used in determining conformance with specifications when the applicable ASTM specifications or standards make direct reference to this practice.

1.3 This practice describes two commonly accepted methods of rounding data, identified as the Absolute Method and the Rounding-Off Method. In the application of this practice to a specific material or materials it is essential to specify which method is intended to apply. In the absence of such specification, reference to this practice, which expresses no preference as to which method should apply, would be meaningless. The choice of method is arbitrary, depending upon the current practice of the particular branch of industry or technology concerned, and should therefore be specified in the prime publication.

1.4 Section 7 of this practice gives guidelines for use in recording, calculating, and reporting the final result for test data.

2. Referenced Documents

2.1 *ASTM Standards:*

E 456 Terminology Related to Quality and Statistics[2]

E 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)[2]

2.1 *ANSI Standard:*

ANSI Z25.1 Rules for Rounding Off Numerical Values[3]

3. Terminology

3.1 *significant digit, n*—any of the figures 0 through 9, excepting leading zeros and some trailing zeros, which is used with its place value to denote a numerical quantity to some desired approximation.

3.1.1 The digit zero may either indicate a specific value or indicate place only. Zeros leading the first nonzero digit of a number indicate order of magnitude only and are not significant digits. For example, the number 0.0034 has two significant digits. Zeros trailing the last nonzero digit for numbers represented with a decimal point are significant digits. For example, the numbers 1270. and 32.00 each have four significant digits. The significance of trailing zeros for numbers represented without use of a decimal point can only be identified from knowledge of the source of the value. For example, a tensile strength, stated as 140 000 psi, may have as few as two or as many as six significant figures. To avoid ambiguity, the exponential notation may be used; thus, 1.40×10^5 psi indicates that the tensile strength is reported to the nearest 0.01×10^5 or 1000 psi.

4. Expression of Numerical Requirements

4.1 The unqualified statement of a numerical limit, such as "2.50 in. max," cannot, in view of different established practices and customs, be regarded as carrying a definite operational meaning concerning the number of digits to be retained in an observed or a calculated value for purposes of determining conformance with specifications.

4.2 *Absolute Method*—In some fields, specification limits of 2.5 in. max, 2.50 in. max, and 2.500 in. max are all taken to imply the same absolute limit of exactly two and a half inches and for purposes of determining conformance with specifications, an observed value or a calculated value is to be compared directly with the specified limit. Thus, any deviation, however small, outside the specification limit signifies nonconformance with the specifications. This will be referred to as the *absolute method.*

4.3 *Rounding-Off Method*—In other fields, specification limits of 2.5 in. max, 2.50 in. max, 2.500 in. max are taken to imply that, for the purposes of determining conformance with specifications, an observed value or a calculated value should be rounded off to the nearest 0.1 in., 0.01 in., 0.001 in., respectively, and then compared with the specification limit. This will be referred to as the *rounding-off method.*

5. Absolute Method

5.1 *Where Applicable*—The absolute method applies

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[1] This practice is under the jurisdiction of ASTM Committee E-11 on Quality and Statistics and is the direct responsibility of Subcommittee E11.03 on Statistical Analysis and Control Techniques.

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[2] *Annual Book of ASTM Standards*, Vol 14.02.

[3] Available from American National Standards Institute, 1430 Broadway, New York, NY 10018.

where it is the intent that all digits in an observed value or a calculated value are to be considered significant for purposes of determining conformance with specifications. Under these conditions, the specified limits are referred to as absolute limits.

5.2 *How Applied*—With the absolute method, an observed value or a calculated value is not to be rounded off, but is to be compared directly with the specified limiting value. Conformance or nonconformance with the specification is based on this comparison.

5.3 *How Expressed*—This intent may be expressed in the standard in one of the following forms:

5.3.1 If the absolute method is to apply to all specified limits in the standard, this may be indicated by including the following sentence in the standard:

> For purposes of determining conformance with these specifications, all specified limits in this standard are absolute limits, as defined in ASTM Practice E 29, for Using Significant Digits in Test Data to Determine Conformance with Specifications.

5.3.2 If the absolute method is to apply to all specified limits of some general type in the standard (such as dimensional tolerance limits), this may be indicated by including the following sentence in the standard:

> For purposes of determining conformance with these specifications, all specified (dimensional tolerance) limits are absolute limits, as defined in ASTM Practice E 29, Using Significant Digits in Test Data to Determine Conformance with Specifications.

5.3.3 If the absolute method is to apply to all specified limits given in a table, this may be indicated by including a footnote with the table as follows:

	Width Tolerances[A] Plus and Minus, in.	
Width, in.	0.032 in. and under in Thickness	Over 0.032 in. in Thickness
2 and under	0.005	0.010
Over 2 to 8 incl	0.008	0.013
Over 8 to 14 incl	0.010	0.015
Over 14 to 20 incl	0.013	0.018

[A] Tolerance limits specified are absolute limits as defined in ASTM Practice E 29, for Using Significant Digits in Test Data to Determine Conformance with Specifications.

6. Rounding-Off Method

6.1 *Where Applicable*—The rounding-off method applies where it is the intent that a limited number of digits in an observed value or a calculated value are to be considered significant for purposes of determining conformance with specifications.

6.2 *How Applied*—With the rounding-off method, an observed value or a calculated value should be rounded off by the procedure prescribed in 4.3 to the nearest unit in the designated place of figures stated in the standard, as, for example, "to the nearest 100 psi," "to the nearest 10 ohms," "to the nearest 0.1 percent," etc. The rounded-off value should then be compared with the specified limit, and conformance or nonconformance with the specification based on this comparison.

6.3 *How Expressed*—This intent may be expressed in the standard in one of the following forms:

6.3.1 If the rounding-off method is to apply to all specified limits in the standard, and if all digits expressed in the specification limit are to be considered significant, this may be indicated by including the following statement in the standard:

> The following applies to all specified limits in this standard: For purposes of determining conformance with these specifications, an observed value or a calculated value shall be rounded off "to the nearest unit" in the last right-hand digit used in expressing the specification limit, in accordance with the rounding-off method of ASTM Practice E 29, for Using Significant Digits in Test Data to Determine Conformance with Specifications.

6.3.2 If the rounding-off method is to apply only to the specified limits for certain selected requirements, this may be indicated by including the following statement in the standard:

> The following applies to specified limits for requirements on (tensile strength), (elongation), and (. . .) given in . . ., (applicable section number and title) and (. . .) of this standard: For purposes of determining conformance with these specifications, an observed value or a calculated value shall be rounded off to the nearest (1000 psi) for (tensile strength), to the nearest (1 percent) for (elongation), and to the nearest (. . .) for (. . .) in accordance with the rounding-off method of ASTM Practice E 29 Using Significant Digits in Test Data to Determine Conformance with Specifications.

6.3.3 If the rounding-off method is to apply to all specified limits in a table, this may be indicated by a note in the manner shown in the following examples:

6.3.3.1 *Example 1*—Same significant digits for all items:

	Chemical Composition, %
Copper	4.5 ± 0.5
Iron	1.0 max
Silicon	2.5 ± 0.5
Other constituents (magnesium + zinc + manganese)	0.5 max
Aluminum	remainder

NOTE 1—For purposes of determining conformance with these specifications, an observed value or a calculated value shall be rounded off to the nearest 0.1 percent, in accordance with the rounding-off method of ASTM Practice E 29, for Using Significant Digits in Test Data to Determine Conformance with Specifications.

6.3.3.2 *Example 2*—Significant digits not the same for all items; similar requirements:

	Chemical Composition, percent	
	min	max
Nickel	57	. . .
Chromium	14	18
Manganese		3
Silicon	. . .	0.40
Carbon	. . .	0.25
Sulfur	. . .	0.03
Iron	remainder	

NOTE 2—For purposes of determining conformance with these specifications, an observed value or a calculated value shall be rounded off "to the nearest unit" in the last right-hand significant digit used in expressing the limiting value, in accordance with the round-off method of ASTM Practice E 29, Using Significant Digits in Test Data to Determine Conformance with Specifications.

6.3.3.3 *Example 3*—Significant digits not the same for all items; dissimilar requirements:

	Tensile Requirements
Tensile strength, psi	60 000 to 72 000
Yield point, min, psi	33 000
Elongation in 2 in., min %	22

NOTE 3—For purposes of determination of conformance with these specifications, an observed value or a calculated value shall be rounded off to the nearest 1000 psi for tensile strength and yield point and to the nearest 1 percent for elongation, in accordance with the rounding-off

method of ASTM Practice E 29 for Using Significant Digits in Test Data to Determine Conformance with Specifications.

6.4 *Rounding-Off Procedure*—The actual rounding-off procedure[4] shall be as follows:

6.4.1 When the digit next beyond the last place to be retained is less than 5, retain unchanged the digit in the last place retained.

6.4.2 When the digit next beyond the last place to be retained is greater than 5, increase by 1 the digit in the last place retained.

6.4.3 When the digit next beyond the last place to be retained is 5, and there are no digits beyond this 5, or only zeros, increase by 1 the digit in the last place retained if it is odd, leave the digit unchanged if it is even. Increase by 1 the digit in the last place retained, if there are digits beyond this 5.

6.4.4 This rounding-off procedure may be restated simply as follows: When rounding off a number to one having a specified number of significant digits, choose that which is nearest. If two choices are possible, as when the digits dropped are exactly a 5 or a 5 followed only by zeros, choose that ending in an even digit. Table 1 gives examples of applying this rounding-off procedure.

6.5 The rounded-off value should be obtained in one step by direct rounding off of the most precise value available and not in two or more successive roundings. For example: 89 490 psi rounded off to the nearest 1 000 psi is at once 89 000; it would be incorrect to round off first to the nearest 100, giving 89 500 and then to the nearest 1 000, giving 90 000.

6.6 *Special Case, Rounding Off to the Nearest 50, 5, 0.5, 0.05, etc.*—If in special cases it is desired to specify rounding off to the nearest 50, 5, 0.5, 0.05, etc., this may be done by so indicating in the standard. In order to round off to the nearest 50, 5, 0.5, 0.05, etc., double the observed or calculated value, round off to the nearest 100, 10, 1.0, 0.10, etc., in accordance with the procedure in 6.4, and divide by 2. For example, in rounding off 6 025 to the nearest 50, 6 025 is doubled giving 12 050 which becomes 12 000 when rounded off to the nearest 100 (6.4.3). When 12 000 is divided by 2, the resulting number, 6 000, is the rounded-off value of 6 025. In rounding off 6 075 to the nearest 50, 6 075 is doubled giving 12 150 which becomes 12 200 when rounded off to the nearest 100 (6.4.3). When 12 200 is divided by 2, the resulting number, 6 100, is the rounded-off value of 6 075.

7. Guidelines for Retaining Significant Figures in Calculation and Reporting of Test Results

7.1 *General Discussion*—Rounding test results avoids a misleading impression of precision while preventing loss of information due to coarse resolution. Any approach to retention of significant digits of necessity involves some loss of information; therefore, the level of rounding should be carefully selected considering both planned and potential uses for the data. The number of significant digits must, first, be adequate for comparison against specification limits (see 6.2). The following guidelines are intended to preserve the data for statistical summaries. For certain purposes, such as where calculations involve differences of measurements close in magnitude, and for some statistical calculations, such as paired t-tests, autocorrelations, and nonparametric tests, reporting data to a greater number of significant digits may be advisable.

TABLE 1 Examples[A] of Rounding Off

Specified Limit	Observed Value or Calculated Value	To Be Rounded Off to Nearest	Rounded-Off Value to be Used for Purposes of Determining Conformance	Conforms with Specified Limit
Yield point, 36 000 psi, min	35 940	100 psi	35 900	no
	35 950	100 psi	36 000	yes
	35 960	100 psi	36 000	yes
Nickel, 57 %, min	56.4	1 %	56	no
	56.5	1 %	56	no
	56.6	1 %	57	yes
Water extract conductivity, 40 micromhos/cm, max	40.4	1 micromho/cm	40	yes
	40.5	1 micromho/cm	40	yes
	40.6	1 micromho/cm	41	no
Sodium bicarbonate 0.5 %, max	0.54	0.1 %	0.5	yes
	0.55	0.1 %	0.6	no
	0.56	0.1 %	0.6	no

[A] These examples are meant to illustrate rounding rules and do not necessarily reflect the usual number of digits associated with these test methods.

7.2 *Recording Test Data*—When recording direct measurements, as in reading marks on a buret, ruler, or dial, all digits known exactly, plus one digit which may be uncertain due to estimation, should be recorded. For example, if a buret is graduated in units of 0.1 mL, then an observation would be recorded as 9.76 mL where it is observed between 9.7 and 9.8 marks on the buret, and estimated about six tenths of the way between those marks. When the measuring device has a vernier scale, the last digit recorded is the one from the vernier.

7.2.1 The number of significant digits given by a digital display or printout from an instrument should be greater than or equal to those given by the rule for reporting test results in 7.4 below.

7.3 *Calculation of Test Result from Test Data*—When calculating a test result from test data, avoid rounding of intermediate quantities. As far as is practicable with the calculating device or form used, carry out calculations with the test data exactly and round only the final result.

7.4 *Reporting Test Results*—A suggested rule relates the significant digits of the test result to the precision of the measurement expressed as the standard deviation σ. The applicable standard deviation is the repeatability standard deviation (see Terminology E 456). Test results should be round to not greater than 0.5 σ nor less than 0.05 σ, provided that this value is not greater than the unit specified in the specification (see 6.2). When only an estimate, s, is available for σ, s may be used in place of σ in the preceding sentence.

Example: A test result is calculated as 1.45729. The standard deviation of the test method is estimated to be, 0.0052. Round to 1.457 or the nearest 0.001 since this rounding unit, 0.001, is between 0.05 σ = 0.00026 and 0.5 σ = 0.0026.

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[4] The rounding-off procedure given in this practice is the same as the one given in the American National Standard Rules for Rounding Off Numerical Values (ANSI Z25.1) and in the *ASTM Manual on Presentation of Data and Control Chart Analysis, STP 15-D*.

NOTE 4—A rationale for this rule is derived from representing the standard deviation of a rounded test result by $\sqrt{\sigma^2 + w^2/12}$ where σ is the standard deviation of the unrounded test result. The quantity $w/\sqrt{12}$ is the standard deviation of an error uniformly distributed over the range w. Rounding so that w is below 0.5 σ ensures that the standard deviation is increased by at most 1 %, while adding more digits would give a misleading impression of precision.

7.4.1 When no estimate of the standard deviation σ is known, then rules for retention of significant digits of computed quantities may be used to derive a number of significant digits to be reported, based on significant digits of test data.

7.4.1.1 The rule when adding or subtracting test data is that the result shall contain no significant digits beyond the place of the last significant digit of any datum.

Examples:

(*1*) 11.24 + 9.3 + 6.32 = 26.9, since the last significant digit of 9.3 is the first following the decimal place,

(*2*) 26.9 is obtained by rounding the exact sum, 26.86, to this place of digits.

(*3*) 926 − 923.4 = 3

140,000 + 91,460 = 231,000 when the first value was recorded to the nearest thousand.

7.4.1.2 The rule when multiplying or dividing is that the result shall contain no more significant digits than the value with the smaller number of significant digits.

Examples:

(*1*) 11.38 × 4.3 = 49, since the factor 4.3 has two siginificant digits

(*2*) (926 − 923.4)/4.3 = 0.6 Only one figure is significant since the numerator difference has only one significant digit.

7.4.1.3 The rules for logarithms and exponentials are: Digits of $ln(x)$ or $\log_{10}(x)$ are significant through the n-th place after the decimal when x has *n* significant digits. The number of significant digits of e^x or 10^x is equal to the place of the last significant digit in x after the decimal.

Examples: ln(3.46) = 1.241 to three places after the decimal, since 3.46 has three significant digits. $10^{3.46}$ = 2900 has two significant digits, since 3.46 is given to two places after the decimal.

7.4.1.4 The rule for numbers representing exact counts or mathematical constants is that they are to be treated as having an infinite number of significant digits.

Examples:

(*1*) 1 − 0.23/2 = 0.88 where the numbers 1 and 2 are exact and 0.23 is an approximate quantity.

(*2*) A count of 50 pieces times a measured thickness 0.124 mm is 50 × 0.124 = 6.20 mm, having three significant figures.

(*3*) A measurement of 1.634 in. to the nearest thousandth, is converted to mm. The result, 1.634 × 25.4 = 41.50 mm, has four significant digits. The conversion constant, 25.4, is exact.

NOTE 5—More extensive discussion of dimensional conversion can be found in Practice E 380.

7.5 *Specification Limits*—When the rounding off method is to apply to given specified limits, it is desirable that the significant digits of the specified limits should conform to the precision of the test following the rule of 7.3. That is, the rounding unit for the specification limits should be between 0.05 and 0.5 times the standard deviation of the test.

7.6 *Averages and Standard Deviations*—When reporting the average and standard deviation of replicated measurements or repeated samplings of a material, a suggested rule for most cases is to round the standard deviation to two significant digits and round the average to the same last place of significant digits. When the number of observations is large (more than 15 when the lead digit of the standard deviation is 1, more than 50 with lead digit 2, more than 100 in other cases), an additional digit may be advisable.

7.6.1 Alternative approaches for averages include reporting $\bar{x}$ to within 0.05 to 0.5 times the standard deviation of the average $\sigma/\sqrt{n}$, or applying rules for retaining significant digits to the calculation of $\bar{x}$. The ASTM STP 15-D provides methods for reporting $\bar{x}$ and s for these applications.[4]

NOTE 6—A rationale for the suggested rule comes from the uncertainty of a calculated standard deviation s. The standard deviation of s based on sampling from a normal distribution with n observations is approximately $\sigma/\sqrt{2n}$. Reporting s to within 0.05 to 0.5 of this value, following the rule of 7.4, leads to two significant digits for most values of σ when the number of observations n is 100 or fewer.

Example: Analyses on six specimens give values of 3.56, 3.88, 3.95, 4.07, 4.21, and 4.47 for a constituent. The average and standard deviation, unrounded, are $\bar{x}$ = 4.0233. . . and s = 0.3089. . . . The suggested rule would report $\bar{x}$ and s as 4.02 and 0.31.

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Standard Methods of
CONDUCTING STRENGTH TESTS OF PANELS FOR BUILDING CONSTRUCTION[1]

This standard is issued under the fixed designation E 72; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

Sound engineering design of structures, using existing or new materials requires accurate technical data on the strength and rigidity of the basic elements employed in various construction systems. It is the purpose of these test methods to provide a systematic basis for obtaining engineering data on various construction elements and structural details of value to designers, builders, building officials, and others interested in this field. The results should closely approximate the performance in actual service.

1. Scope

1.1 These methods cover the following procedures for determining the structural properties of segments of wall, floor, and roof constructions:

	Section
Test Specimens	3
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Deformation Measurements	5
Reports	6
Precision and Accuracy	7
TESTING WALLS	
Significance	8
Compressive Load	9
Tensile Load	10
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Concentrated Load	13
Impact Load—See Methods E 695 and E 661	
Racking Load—Evaluation of Sheathing Materials on a Standard Wood Frame	14
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TESTING FLOORS	
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Impact Load—See Methods E 695 and E 661	
TESTING ROOFS	
Significance	19
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APPENDIX	
Technical Interpretation	X1

1.2 Metric units are to be considered as the primary standard units.

2. Applicable Documents

2.1 *ASTM Standards:*

E 4 Practices for Load Verification of Testing Machines[2]

E 73 Methods of Testing Truss Assemblies[3]

E 564 Method of Static Load Test for Shear Resistance of Framed Walls for Buildings[3]

E 575 Practice for Reporting Data from Structural Tests of Building Constructions, Elements, Connections, and Assemblies[3]

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[1] These methods are under the jurisdiction of ASTM Committee E-6 on Performance of Building Constructions and are the direct responsibility of Subcommittee E 06.12 on Structural Performance of Vertical Structures.

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[2] *Annual Book of ASTM Standards*, Vol 03.01.

[3] *Annual Book of ASTM Standards*, Vol 04.07.

E 661 Test Method for Performance of Wood and Wood-Based Floor and Roof Sheathing Under Concentrated Static and Impact Loads[3]

E 695 Method for Measuring Relative Resistance of Wall, Floor, and Roof Constructions to Impact Loading[3]

3. Test Specimens

3.1 *Size*—The specimens shall be representative as to material and workmanship and shall be as large as practicable to minimize the effect of variations in the material and workmanship, in order to obtain results representative of the construction. Obviously, the size of the specimens shall be limited to the size that can be tested in the larger testing machines available in a well equipped laboratory, and which can be subjected to loads in accordance with good testing procedure, and for which the deformation can be measured with sufficient accuracy.

3.2 *Length or Height*—The length or height of specimen for each element shall be chosen to conform to the length or height of that element in actual use.

3.3 *Width*—The width of specimen shall be chosen, insofar as possible, to include several of the principal load-carrying members to ensure that the behavior under load will simulate that under service conditions. With the exception of specimens for the racking load test, the nominal width of wall specimens shall be 1.2 m (4 ft). The actual width of specimens shall be a whole number multiplied by the spacing of the principal load-carrying members except for prefabricated panels, for which the actual width shall be the width of panel used. If the structural properties of a particular construction are to be compared with another construction, there should not be a great difference in the actual widths of the specimens.

3.4 *Age*—Constructions, such as concrete and masonry (brick, structural clay tile, concrete block) for which the structural properties depend upon the age of the specimen, shall be tested not less than 25 days nor more than 31 days after fabrication. This age requirement applies also to plastered and stuccoed constructions.

4. Loading

4.1 *Apparatus*—The testing machine or load-measuring apparatus shall comply with the requirements prescribed in Methods E 4.

4.2 *Application of Load*—Apply the load to all of the specimens in any test in increments so chosen that a sufficient number of readings will be obtained to determine definitely the load-deformation curve (see Section 6). Record the initial reading of the load and the reading of the deformation, either with no load on the specimen or under a small initial load. Increase the load to the first increment and record the deformation. Unless otherwise specified, decrease the load to the initial load and record the set (sometimes designated "permanent set"). Increase the load to two increments and record the set, when it is released to the initial load. Follow this sequence of readings for three increments, four increments, etc., of load. When for each specimen the behavior of the specimen under load indicates that the specimen might fail suddenly and damage the deformation-measuring apparatus, remove this apparatus from the specimen and increase the load continuously until the maximum load that can be applied to the specimen is determined.

4.3 *Duration of Load Application*—Except for racking tests, after each increment of load is applied, maintain the load level as constant as possible for a period of 5 min (see Note 1). Take deformation readings as soon as practical after load application, at the end of the 5-min period under constant load, and immediately and at the end of the 5-min period after any partial or complete load release. Plot initial and 5-min readings in the form of load-deformation curves. Maintain complete load-deformation-time records throughout the test. If application of a given load is required for a certain period, such as 24 h, take deformation readings at the beginning, at intervals during this period, and at the end of this period, to allow the satisfactory plotting of a time-deformation curve for the complete period.

NOTE 1—Reasons for the 5-min application of constant-level increment loads are as follows:

(1) To permit the assembly to come to a substantial rest prior to taking the second set of readings (Depending on the method employed for applying the test load, it may be necessary to continue, at a reduced rate, the motion of the loading device in order to maintain the constant load level during the 5-min period.)

(2) To provide sufficient time for making all observations. (Longer time intervals may be required under certain conditions.)

(3) To observe any time-dependent deformation

or load redistribution, or both, and to record accurately the load level when time-dependent deformation starts, that is, at the divergence of the immediate and delayed load-deformation curves. This load level may, under certain conditions, have an important bearing on the design load.

(4) To be able to stop the test, if this should be desirable, prior to total failure, after initial failure has been anticipated as a result of the observations.

(5) To assure uniformity in test performance and consistency in test results.

5. Deformation Measurements

5.1 Measure the deformations with sufficient precision to define the load-deformation relationship, and report at least to the nearest 0.25 mm (0.01 in.). The deformation-measuring apparatus specified for any loading may be replaced by other apparatus, provided that it permits readings of deformation that are equivalent in accuracy to those from the specified apparatus.

6. Reports

6.1 Show the results of each of the tests graphically, as illustrated in Fig. 1. Plot loads as ordinates and the deformations as abscissas for all tests. There shall be at least three specimens for each test, and the results for each test shall be shown on the same graph. Show the points for deformation under load by open circles and those for set by solid circles. Average the three values for either the deformation or the set and plot this average value in pencil on the graph. Draw a smooth curve among the average points to show the average behavior of the construction. The load-deformation curves shall be continuous lines and the load-set curves shall be dashed lines. Although the particular specimen for each point on the graph is not designated, record it on the laboratory data sheets. If readings are obtained under greater loads for some specimens than for others, plot all the values, but draw the curves only to the average values for which there are three values.

6.2 Prepare the test report in accordance with Recommended Practice E 575.

7. Precision and Accuracy

7.1 No statement is made either on the precision or on the accuracy of these methods due to the variety of materials and combinations of materials involved.

TESTING WALLS

8. Significance

8.1 The procedures described are those that will test the behavior of segments of wall construction under conditions representative of those encountered in service. Performance criteria based on data from those procedures can ensure structural adequacy and service life.

9. Compressive Load

9.1 *Test Specimens*—Tests shall be made on three like specimens, each having a height equal to the length of the element and a nominal width of 1.2 m (4 ft) (see Section 3).

9.2 *Apparatus*—The apparatus shall be assembled as shown in Fig. 2 and shall conform to the detailed requirements for component parts prescribed in 9.2.1 and 9.2.2, or the equivalent.

9.2.1 *Compressometer*—A bracket shall be attached to the specimen near the upper end, supporting a metal rod. A bracket shall also be attached to the specimen near its lower end, supporting a dial micrometer with the spindle up and the gage length shall be recorded. The conical end of the rod shall seat in a hole in the end of the spindle and the rod and spindle shall be held in contact by stretched rubber bands. The dial shall be graduated to 0.025 mm (0.001 in.).

9.2.2 *Deflectometer*—A fine wire shall be attached to a clamp near the upper end of the specimen. The free end connected to stretched rubber bands shall be attached to a clamp near the lower end of the specimen. A mirror having a paper scale one-half the width of the mirror shall be attached horizontally to the edge of the specimen at midheight. The scale shall be graduated to 2.5 mm (0.1 in.).

9.3 *Procedure:*

9.3.1 *Loading*—Test the specimen as a column having a flat end at the bottom (Fig. 2). Apply compressive loads to a steel plate covering the upper end of the specimen. Apply the load uniformly along a line parallel to the inside face, and one-third the thickness of the specimen from the inside face. For wood construction, a rate of loading corresponding to a movement of the testing machine crosshead of nominally 0.8 mm/min (0.03 in./min) has been found satisfactory.

9.3.2 *Load-Deformation Data*—Attach four compressometers to the faces of the specimen, one near each corner of the specimen as shown in Fig. 2, to measure the shortening of the specimen. Record the readings to the nearest 0.025 mm (0.001 in.).

9.3.3 *Lateral Deflection*—Attach two deflectometers, one to each edge of the specimen, as shown in Fig. 2. Record the readings, when the image of the wire coincides with the wire, to the nearest 0.25 mm (0.01 in.).

9.4 *Calculations and Report:*

9.4.1 *Deformation*—For each compressometer, calculate the shortening under each load as the difference between the reading of the compressometer when the load is applied and the initial reading. Calculate the shortening of the specimen as the average of the shortenings for each of the four compressometers multiplied by the ratio: specimen length divided by the compressometer gage length. Obtain the sets in a similar manner.

9.4.2 *Lateral Deflection*—Calculate the lateral deflection and the lateral set under each load for each deflectometer as the difference between the reading of the deflectometer when the load is applied and the initial reading. Calculate the lateral deflection and lateral set for the specimen as the average of the lateral deflection and lateral set of the two deflectometers.

9.4.3 *Data Presentation*—Record the maximum load for each specimen and report the results of load-deformation and load-deflection measurements in the form of a graph in accordance with Section 6. Report gage lengths of all deflection or deformation gages.

10. Tensile Load

10.1 *Test Specimens*—Tests shall be made on three like specimens, each having a height equal to the length of the element and a nominal width of 1.2 m (4 ft) (see Section 3).

10.2 *Apparatus*—The apparatus preferably shall be assembled in a vertical testing machine and shall conform to the detailed requirements for component parts prescribed in 9.2.1 and 9.2.2, or the equivalent, with the exception that the compressometers prescribed in 9.2.1 shall be replaced by extensometers which shall be like the compressometers but so adjusted before load is applied that the stretch of the specimen can be measured.

10.3 *Procedure:*

10.3.1 *Loading*—Test the specimen as a tension specimen by uniform application of tensile forces along the line of the fastenings at the top and the bottom of the wall in a building. The top and bottom pulling fixtures may be attached to the specimen by fastenings similar to those used in a building, provided that, under the maximum load, failure of the specimen occurs between the top and the bottom of the specimen, not in either the pulling fixtures or the fastenings. If, under the tensile load, failure occurs either in a pulling fixture or in a fastening, the results of the test determine only the properties of the fixtures or the fastenings, not of the wall construction. When the failure occurs in fastenings, the tensile load indicates the maximum tensile strength of the construction that can be realized in actual service unless improved fastenings are provided.

10.3.1.1 *Masonry Constructions*—The construction may be continued upward beyond the top of the specimen and downward below the bottom of the specimen to enclose attachments for the pulling fixtures.

10.3.1.2 *Framed Wall Constructions*—If the construction has studs (either of wood or metal) the studs may be extended upward and downward beyond the top and bottom of the specimen and attached to the pulling fixtures. If the framed wall has plates at the top and the bottom, attach the pulling fixtures to the plates in the specimen.

10.3.2 *Load-Deformation Data*—Attach four extensometers to the faces of the specimen, one near each corner, as shown in Fig. 2, to measure the stretch of the specimen. Record the readings to the nearest 0.025 mm (0.001 in.).

10.3.3 *Lateral Deflection*—Attach two deflectometers, one to each edge of the specimen, as shown in Fig. 2. Record the readings, when the image of the wire coincides with the wire, to the nearest 0.25 mm (0.01 in.). Lateral deflection (if any) may be caused by nonaxial loading of the specimen.

10.4 *Calculations and Report*—For tensile loads, the calculations and report shall be similar to those required for compressive loads (see 9.4).

11. Transverse Load—Specimen Horizontal

11.1 *Test Specimens*—Tests shall be made on three like specimens on symmetrical assemblies and six like specimens on unsymmetrical assemblies, each having a length equal to the length of the element and a nominal width of 1.2 m (4 ft) (see Section 3).

11.2 *Apparatus*—The apparatus shall be assembled as shown in Fig. 3 and shall conform to the detailed requirements for component parts prescribed in 11.2.1 through 11.2.3, or the equivalent.

11.2.1 *Supports*—Two steel rollers with a steel plate between each supporting roller and the specimen.

11.2.2 *Loading Assembly*—Two steel rollers with a steel plate between each loading roller and the specimen.

11.2.3 *Deflection Gage*—A frame shall be placed on the upper face of the specimen. To prevent stresses deforming the frame as the specimen deforms under load, this frame shall rest on three hardened steel balls each supported by a steel block on the face of the specimen. Two of the balls shall be placed in a line vertically above one support and the third ball vertically above the other support. Two dial micrometers, one near each longitudinal edge of the specimen, shall be attached to the frame at midspan. The spindles shall rest on the upper face of the specimen. The micrometers shall be graduated to 0.025 mm (0.001 in.).

11.3 *Procedure:*

11.3.1 *Loading*—Use "two-point" loading for transverse load tests. Test the specimen as a simple beam (Fig. 3) on a span 150 mm (approximately 6 in.) less than the specimen length. Apply two equal loads, each at a distance of one quarter of the span from the supports, toward the middle of the span. For wall specimens tested horizontally (Fig. 3), the load on the specimen shall include the weight of specimen between the supports. Apply the transverse loads to the outside face for three of the specimens and to the inside face for three of the specimens. For symmetrical assemblies, test only three specimens.

11.3.1.1 Uniformly distributed loading may be used instead of quarter-point loading, if a satisfactory method is available. The transverse strength for any span may be greater for some constructions under uniformly distributed load than under loads applied at the quarter-points of the span. Transverse load, uniformly distributed, may be applied by air pressure, either in a bag or in a chamber having the specimen as one face. Support specimens tested under uniform loading by rollers as for quarter-point loading.

11.3.1.2 The bag method of loading is shown schematically in Fig. 4. Connect a reaction platform parallel to the face to be loaded and wider than the specimen to the supports by tie rods. Place an airtight bag of rubberized cloth as wide as the specimen and as long as the span between the specimen and the reaction platform. Apply transverse load to the specimen by increasing the air pressure in the bag. Measure the pressure by means of a manometer. Water is usually the liquid in the manometer, but the specific gravity of the liquid shall be such that the error in pressure readings does not exceed 1 %.

11.3.1.3 When the chamber method of loading is used with the specimen horizontal, place the specimen near the floor, which should be practically airtight. An airtight frame or curb shall surround the specimen closely and be about flush with the upper surface of the specimen. A rubber blanket covers the specimen, overlaps the frame, and is sealed so that it is reasonably airtight. Use a small vacuum pump or positive action exhaust blower to reduce air pressure between the specimen and floor. Measure the difference in pressure above and below the specimen by means of a manometer.

11.3.2 *Strength on Short Span*—The transverse strength of any construction increases as the span is shortened. If the strength of the construction for a shorter span is desired, do not compute it, but test the construction on the short span.

11.4 *Calculations and Report:*

11.4.1 *Load-Deflection Data*—For each micrometer, calculate the deflection under a given load as the difference between the reading to the nearest division of the micrometer when the load is applied and the initial reading. Calculate the deflection of the specimen for the span as the average of the deflections obtained from each of the two micrometers. Calculate the sets under the initial load by using a similar method. Record the maximum load for each specimen.

11.4.2 *Data Presentation*—Report the results in the form of a graph in accordance with Section 6.

12. Transverse Load—Specimen Vertical

12.1 *Test Specimens*—Tests shall be made on three like specimens on symmetrical assemblies and six like specimens on unsymmetrical assemblies each having a length equal to the length of the element and a nominal width of 1.2 m (4 ft) (see Section 3).

12.2 *Apparatus*—The apparatus shall be assembled as shown in Fig. 3 and shall conform to the requirements for component parts prescribed in 12.2.1 through 12.2.5, or the equivalent.

12.2.1 *Steel Channel.*

12.2.2 *Rollers*—Cylindrical rollers, two supporting rollers, two loading rollers.

12.2.3 *Screw Jack.*

12.2.4 *Ring Dynamometer.*

12.2.5 *Deflectometers*—Two taut-wire mirror-scale deflectometers similar to those described in 9.2.2.

12.3 *Procedure*—Transverse loads cannot be applied satisfactorily to some wall constructions, such as masonry, with the specimen in a horizontal position. For such constructions, apply the loads with the specimen in a vertical position, as shown in Fig. 3, thus simulating service conditions. The specimen, on a steel channel, shall rest on cylindrical rollers to prevent restrained end conditions. The axes of the rollers shall be parallel to the faces of the specimen. The two supporting rollers shall be in contact with the vertical surface of the frame and each roller shall rest horizontally on sponge rubber about 10 mm (0.4 in.) thick to prevent longitudinal restraint. Each of the two loading rollers shall also rest on sponge rubber. Apply the loads horizontally by a screw jack and measure by a ring dynamometer between the jack and the specimen. The error in the load indicated by the dynamometer shall not exceed 1 %. Attach two taut-wire mirror-scale deflectometers to the specimen, one to each vertical edge.

12.3.1 Apply the transverse load to the outside face for three of the specimens, and to the inside face for three of the specimens. For symmetrical assemblies, test only three specimens.

12.3.2 When the Chamber Method of loading is used with the specimen vertical, the specimen forms one face of an airtight chamber from which the air is exhausted. If all four edges of the specimen bear on the chamber, this loading determines the strength of the specimen as a plate supported at the four edges, not the transverse strength as defined in these methods.

12.3.3 If a specimen tested by the chamber method, either horizontally or vertically, has an airtight cavity, vent each cavity to the low-pressure face by a hole in the face of the specimen not less than 5 mm (0.2 in.) in diameter, located where it will least affect the transverse strength of the specimen.

12.4 *Calculations and Report*—Calculate the results of test and report as described in 11.4, and report deflectometer readings to the nearest 0.25 mm (0.01 in.).

13. Concentrated Load

13.1 *Test Specimens*—Concentrated load tests shall be made on each transverse specimen after the transverse load tests, the concentrated load being applied to the same face to which the transverse load was applied.

13.2 *Apparatus*—The apparatus shall be assembled as shown in Fig. 5 and shall conform to the requirements for component parts prescribed in 13.2.1 through 13.2.3, or the equivalent.

13.2.1 *Steel Bar*—Steel bar having a diameter of 25.4 mm (1 in.) and the edge of the face contacting the specimen rounded to a radius of 1.3 mm (0.05 in.).

13.2.2 *Depth Gage*—The depth gage shall consist of a dial micrometer graduated to 0.025 mm (0.001 in.) mounted on a three-legged support. The support shall be notched to permit placing the micrometer directly adjacent to the bar and shall be long enough to permit placing the supporting legs on undisturbed areas of the face of the specimen.

13.2.3 *Loading Device*—Any convenient means for applying a compressive load up to 5 kN (1100 lbf) and means for measuring the load within 1 %.

13.3 *Procedure:*

13.3.1 *Loading*—Place the entire specimen or portion of the specimen on a horizontal support and properly level. Place the steel bar on the face of the specimen at what is judged to be the weakest place and, also, at what is judged to be the strongest place. Apply a load vertically downward to the upper surface of the bar. Continue loading until maximum load or 4.45 kN (1000 lbf) is attained.

13.3.2 *Depth of Indentation*—Measure the depth of indentation, by means of the depth gage, and record the reading of the micrometer to the nearest 0.025 mm (0.001 in.).

13.4 *Calculations and Report:*

13.4.1 *Depth of Indentation*—Calculate the depth of indentation (set) after a given load has been applied and the bar removed to the nearest 0.025 mm (0.001 in.) as the difference between the depth for that load and the initial reading of the micrometer before a load has been applied to the specimen.

13.4.2 *Data Presentation*—Report the results in the form of a graph in accordance with Section 6.

14. Racking Load—Evaluation of Sheathing Materials on a Standard Wood Frame

NOTE 2—If the test objective is to measure the performance of the complete wall, Method E 564 is recommended.

14.1 *Scope*—This test method measures the resistance of panels, having a standard wood frame, and sheathed with sheet materials such as structural insulating board, plywood, gypsum board, transite, etc., to a racking load such as would be imposed by winds blowing on a wall oriented at 90° to the panel. It is intended to provide a reliable, uniform procedure for determining the resistance to racking load provided by these sheet materials as commonly employed in building construction. Since a standard frame is employed, the relative performance of the sheathing is the test objective.

14.1.1 This test is conducted with standardized framing, loading procedures, and method of measuring deflection, as detailed in the method to ensure reproducibility. Provision is made for following the sheathing manufacturers' recommendations for attaching the sheathing to the frame, and for reporting the behavior of the specimen over its entire range of use.

14.1.2 In applying the results, due allowance shall be made for any variation in construction details or test conditions from those in actual service.

14.2 *Test Specimens:*

14.2.1 *Size and Number*—The test specimen shall be 2.4 by 2.4 m (8 by 8 ft) and the framing shall be constructed as shown in Fig. 6 and a minimum of three panels of each construction shall be tested. It is the intent of this test procedure to evaluate the stiffening effect of the sheathing material; therefore, the frame shall be constructed as nearly like the frames shown in Fig. 6 as possible. Frames shall be newly constructed for each test. All individual framing members shall be continuous. The moisture content of framing material shall be between 12 and 15 % when the panel is fabricated, and shall not vary by more than 3 % from the initial moisture content when the panel is tested.

14.2.2 *Application of Sheathing*—The method of applying the sheathing shall be exactly as specified by the manufacturer. The spacing of fasteners shall be as recommended. Fasteners shall be driven through the sheathing into only the outside stud of each corner post shown in Fig. 6. The importance of the attachment of sheathing to the framing cannot be overemphasized. Slight differences in edge clearances, angle of fastener, and amounts of penetration of heads of fasteners into the sheathing have appreciable effects on the results of test. Unless otherwise specified, fasteners shall be driven perpendicular to the surface of the sheathing with the center of each fastener the specified distance from the edge of the sheathing. Fasteners shall be driven so that the head of the fastener contacts the surface of the sheathing but not so deep as to crush the surface, unless specified differently by the manufacturers.

14.3 *Apparatus*—The apparatus shall be assembled as shown in Fig. 7. Load shall be measured by means of a testing machine, or a dynamometer attached to cables that load the specimen, or in linkage with a hydraulic jack used to apply load. The essential parts of the testing apparatus, exclusive of the loading frame, are as described in 14.3.1 through 14.3.5.

14.3.1 *Base and Loading Frame*—The test panel shall be attached to a timber or steel plate that is in turn attached rigidly to the base of the loading frame in such a manner that when the panel is racked, the sheathing will not bear on the loading frame. This member may be of any convenient cross section, but it shall be at least as long as the panel and not greater in width than the thickness of the frame, 89 mm (3½ in.). Means shall be provided to bolt or otherwise attach the sole plate of the panel firmly to this member. For illustrative purposes, two bolts are shown in Fig. 7. More may be used if required.

14.3.2 *Hold-Down*—A hold-down shall be provided as shown in Fig. 7 to overcome the tendency of one end of the panel to rise as the racking load is applied. Plates and rollers shall be provided between the test specimen and the hold-down so that the top of the specimen can deflect horizontally with respect to the bottom without unnecessary interference from the hold-down. Because the amount of tension in the rods of the hold-down may have an effect on the results of the test, nuts on the hold-down rods shall be tightened prior to load application so that the total force in each rod does not exceed 90 N (20 lbf) at the beginning of test as determined by previous calibration.

14.3.3 *Loading Apparatus*—Load shall be applied to the specimen through an 89 by 89-mm (3.5 by 3.5-in.) timber firmly bolted to the upper plates of the panel. Loading shall be a compressive force against the end of the timber attached to the upper plate. When a testing machine is used, pulleys and cables may be used to transmit the vertical movement of the tension head of the machine to the horizontal movement in the specimen.

14.3.4 *Lateral Guides*—Lateral guides shall be provided so that the specimen will deflect in a plane. The rollers should be bearing-supported to reduce friction to a minimum. The lateral guides shall be firmly attached to the loading frame. Plates for the rollers may be up to 300 mm (12 in.) in length as required.

14.3.5 *Indicating Dials*—Indicating dials, or scales and wires, shall be provided to measure the displacement of the different parts of the panel during test. The readings shall be recorded to the nearest 0.25 mm (0.01 in.). The locations of the dials shall be as shown in the lower left, lower right, and upper right corners of the side view of the test assembly in Fig. 7. The dial at the lower left, which is attached to the stud, measures any rotation of the panel, the dial at the lower right measures any slippage of the panel, and the dial at the upper right measures the total of the other two plus the deformation of the panel. Therefore, the horizontal deflection of the panel at any load is the reading of the dial at the upper right less the sum of the readings of the other two.

14.4 *Procedure:*

14.4.1 *Loading*—Apply the load continuously throughout test at a uniform rate of motion of the loading device used. The recommended speed of testing shall be such that the loading to 3.5 kN (790 lbf) total load shall be completed in not less than 2 min from the start of the test. The loading to 7.0 to 10.5 kN (1570 to 2360 lbf) total load and to failure shall employ the same rate of travel of the loading device as for the loading to 3.5 kN. Give the speed of testing used in the report of test.

14.4.2 *Loading Procedure*—Load the specimen in three stages to 3.5, 7.0, and 10.5 kN (790, 1570, and 2360 lbf) total load at a uniform rate.

14.4.2.1 To provide data to meet performance requirements, other values of total load may be included in the test procedure. Use the same rate of loading as for the loadings specified and indicate additional loadings evaluated and the results obtained in the report.

14.4.2.2 After the load of 3.5 kN (790 lbf) is placed on the specimen, remove all of the load and any residual deflection (set) in the panel noted. Then load the specimen to 7.0 kN (1570 lbf) and again remove the load and note any additional set; after this increase the loading to 10.5 kN (2360 lbf), remove the load again, and note the set. Apply load continuously for each of the increment loads specified above and obtain load-deflection data. Obtain these data for at least each 900 N (200 lbf) of loading. Obtain deflections during the loading cycle and, if desired, during the unloading cycle as well.

14.4.2.3 After the specimen is loaded as specified to 3.5, 7.0, and 10.5 kN (790, 1570, and 2360 lbf) load it again to failure or until the total deflection of the panel becomes 100 mm (4 in.). Obtain readings of deflection for the same intervals of load as were used for the other loadings.

14.5 *Calculations and Report:*

14.5.1 *Deformation*—For each dial, or other measuring device, calculate the movement under each racking load as the difference between the readings when load is applied and the initial readings at the start of the test. Calculate set readings as the difference between the readings when the load is removed and the initial readings.

14.5.2 *Data Presentation*—Report the deflections at 3.5, 7.0, and 10.5 kN (790, 1570, and 2360 lbf) and the set after loading to these amounts. Present load-deflection curves obtained during loading to failure and to 3.5, 7.0, and 10.5 kN in the form of a graph as prescribed in Section 6. Include maximum load and any observations on the behvaior of the panel during test and at failure. Express residual deflections (sets) as percentages of the deflections that produced the sets as well as in millimetres or inches. If the specimen fails, describe the visible failure. If the specimen has been subjected to any special conditioning prior to test, describe this treatment in detail. Describe in the report the sheathing used, the method of applying the sheathing, the type and spacing of fasteners, and the method and rate of loading employed.

15. Racking Load—Evaluation of Sheathing Materials (Wet) on a Standard Wood Frame

15.1 *Scope*—This test has been developed to simulate the degree of wetting possible during construction of a structure when, because of rain, the framing and sheathing may be wetted on one or both sides. Both sides of the wall panel are wetted because this represents the maximum exposure possible during the stage of construction before the structure is roofed.

15.2 *Test Specimens*—The test specimens shall conform in size and fabrication details to the requirements of 14.2.

15.3 *Specimen Conditioning*—Mount the fabricated test specimens or suspend them in a vertical position in such a manner as to prevent continuous immersion of the bottom edge of the specimen. Expose both sides of the test specimen to a water spray applied at or near the top along the entire length to ensure that the top of the specimen is being wetted. The spray shall have no jet action that cuts into the sheathing material, and the spray areas shall overlay sufficiently so that a continuous sheet of water flows down both surfaces of the specimen. Maintain the temperature of the water in the line to the spray nozzle at 24 ± 3°C (75 ± 5°F). Wet the specimens for a period of 6 h and then allow to dry for a period of 18 h. Dry in laboratory air, preferably at a temperature of 24 ± 3°C (75 ± 5°F). Make no attempt to increase the air movement over the specimens by fans or blowers. Subject the test specimens to two complete wetting and drying cycles and then a third wetting cycle.

15.3.1 No more than 2 h shall elapse between the completion of the third wetting cycle and the start of the racking test.

15.4 *Procedure*—Test the specimens in accordance with the procedure described in 14.4.

15.5 *Moisture Content Determination*—After the racking test is completed, cut moisture samples from the sheathing material, and determine moisture content on a weight basis with the moisture content expressed as a percentage of the oven dry weight in accordance with 15.5.1. Preferably, take five moisture content samples at least 100 by 150 mm (4 by 6 in.) in size from each 1.2 by 2.4-m (4 by 8-ft) sheathing panel of the test specimen: one from the center of each sheathing panel at the top and bottom edges, one from midlength on each side, and one from the panel center. Weigh the moisture content samples immediately upon being cut from the test specimen to an accuracy of not less than ±0.2 %. Carefully remove all loose particles from the sample before weighing. Then dry the samples to constant weight in an oven at 103 ± 2°C (217 ± 4°F). If large amounts of volatile matter or substances other than free water are removed from the sheathing material by drying at 103 ± 2°C, the sheathing material may be dried to constant weight at a lower temperature and the drying time and temperature given in the report.

15.5.1 *Calculation*—Calculate the moisture content as follows:

$$M = 100\,[(W - F)/F]$$

where:

M = moisture content, %,
W = initial weight, and
F = final weight when oven dry.

15.6 *Calculations and Report*—The report shall include the racking test data as specified in 14.5. It shall also include the line temperature of the water sprayed on the test specimens; the air temperature and relative humidity during the drying portion of the cycle; and the location of the moisture content samples and the moisture content of each.

TESTING FLOORS

16. Significance

16.1 The procedures outlined will serve to evaluate the performance of floor segments under conditions representative of those sustained in service. Performance criteria based on data from these procedures can ensure structural adequacy and effective service.

17. Transverse Load

17.1 *Test Specimens*—Tests shall be made on three like specimens, each having a length equal to the length of the floor panel and a nominal width of 1.2 m (4 ft) (see Section 3).

17.2 *Apparatus*—The apparatus shall conform to the requirements of 11.2.

17.3 *Procedure*—Conduct the test in accordance with 11.3 on transverse load tests of walls, except apply the loads only to the upper (finish floor) face of the specimen. If practicable, test floor specimens in the horizontal position. If tested in the vertical position, conduct the test in accordance with 12.1 through 12.4 on transverse load tests on walls in the vertical position. If tested in the vertical position, deduct transverse load equal to the weight of the specimen from each recorded load to obtain the applied load on the specimen.

17.3.1 *Strength on Short Span*—The transverse strength of any floor construction increases as the span is shortened. If the strength of the construction for a shorter span is desired, do not compute it, but test the construction on the shorter span.

17.4 *Calculations and Report*—Report the results as indicated in 11.4.

18. Concentrated Load

18.1 *Test Specimens*—Tests shall be made on each of the transverse specimens after the transverse tests are completed.

18.2 *Apparatus*—The apparatus shall conform to the requirements of 13.2.

18.3 *Procedure*—Conduct the test in accordance with 13.3 on concentrated load tests on walls, except apply the loads only to the upper (finish floor) face of the specimen.

18.4 *Calculations and Report*—Report the results as indicated in 13.4.

TESTING ROOFS

19. Significance

19.1 These procedures will serve to evaluate performance of roof segments under simulated service conditions. Roof trusses shall be evaluated under Methods E 73.

20. Transverse Load

20.1 *Test Specimens*—Tests shall be made on three like specimens, each having a length equal to the length of the roof panel and a nominal width of 1.2 m (4 ft) (see Section 3).

20.2 *Apparatus*—The apparatus shall conform to the requirements of 11.2.

20.3 *Procedure*—Conduct the test in accordance with 11.3 on transverse load tests of walls, except normally apply the loads only to the upper (weatherproofed) face of the specimen. The transverse strength of a roof construction under loads acting outward may appear to be less than the strength under loads acting inward. For such constructions, apply loads acting outward to specimens.

20.3.1 *Strength on Short Span*—The transverse strength of any roof construction increases as the span decreases. If the strength of the construction for a shorter span is desired, do not compute it, but test the construction on the shorter span.

20.4 *Calculations and Report*—Report the results as indicate in 11.4.

21. Concentrated Load

21.1 *Test Specimens*—Tests shall be made on each of the transverse specimens after the transverse tests are completed.

21.2 *Apparatus*—The apparatus shall conform to the requirements of 13.2.

21.3 *Procedure*—Conduct the test in accordance with 13.3 on concentrated load tests of walls, except apply the loads only to the upper (weatherproofed) face of the specimen.

21.4 *Calculations and Report*—Report the results as indicated in 13.4.

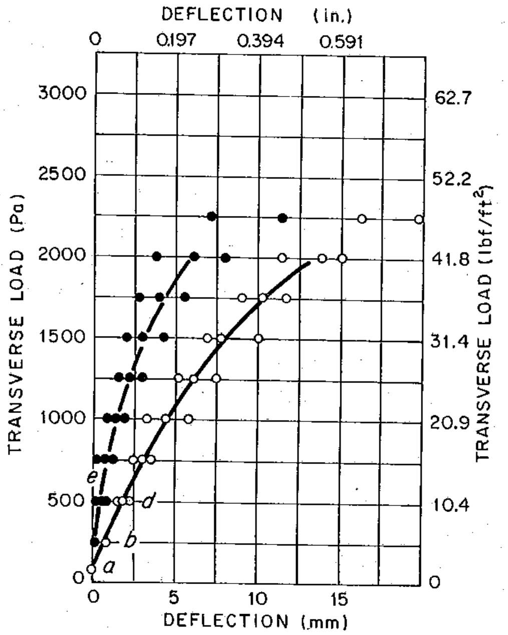


FIG. 1 Typical Graph Showing Results

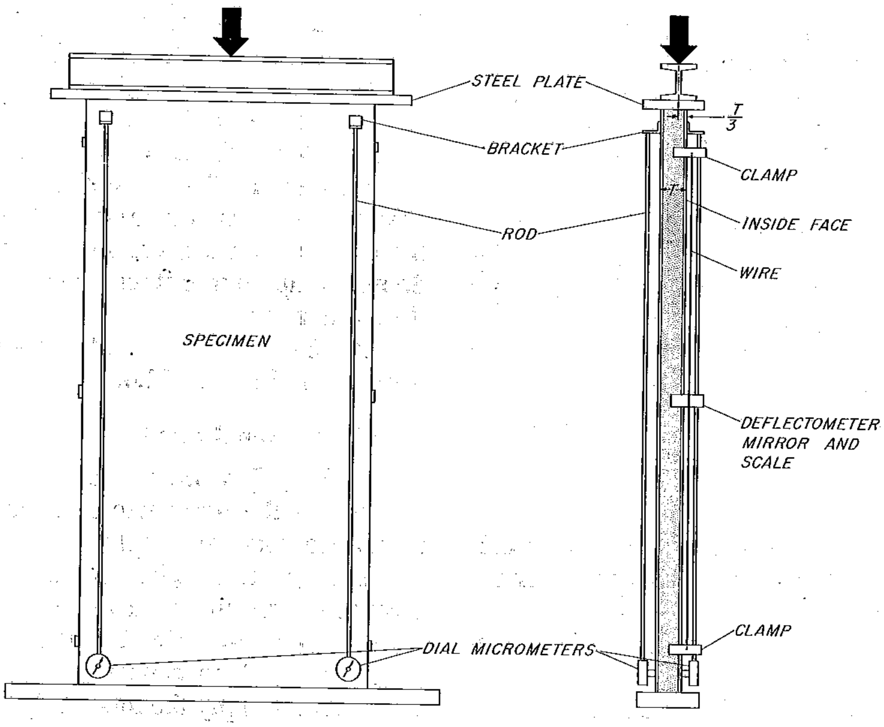


FIG. 2 Compressive Load Test on Wall Specimen

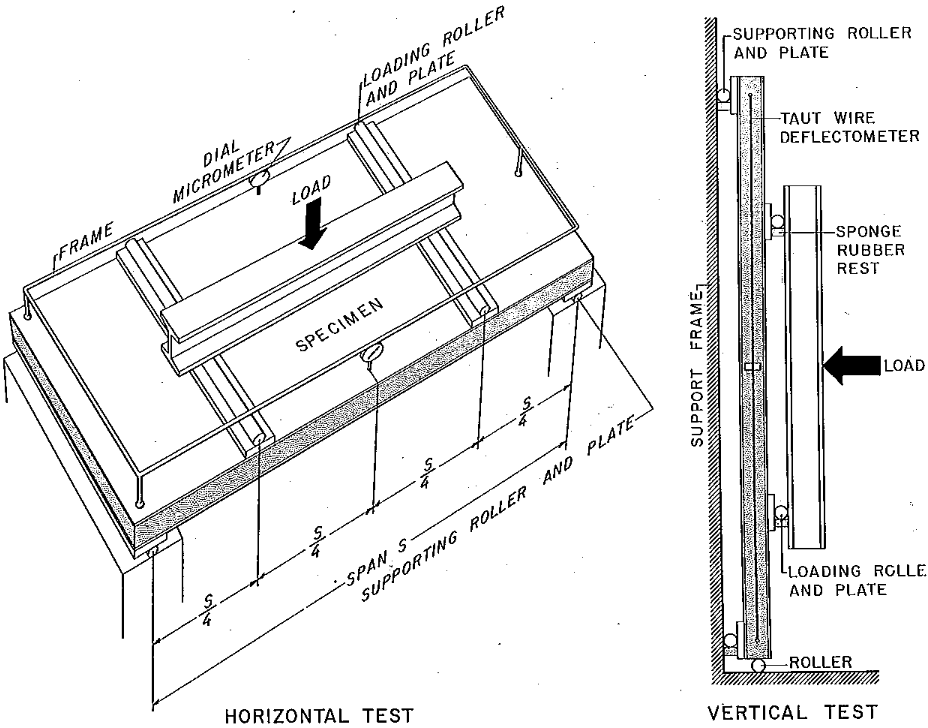


FIG. 3 Transverse Load Test on Wall Specimen

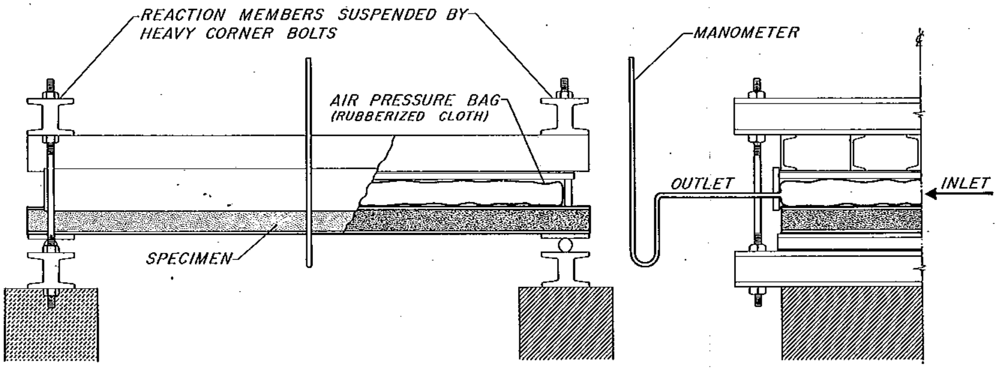


FIG. 4 Apparatus for Uniformly Distributed Transverse Load (Bag Method)

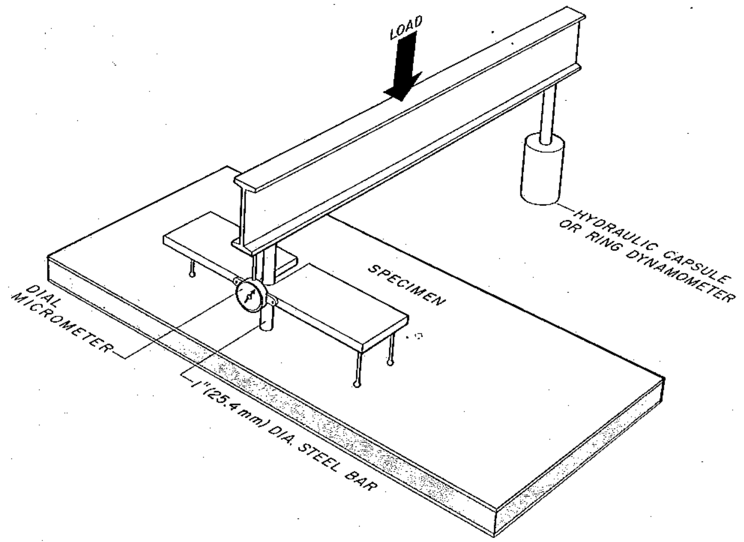
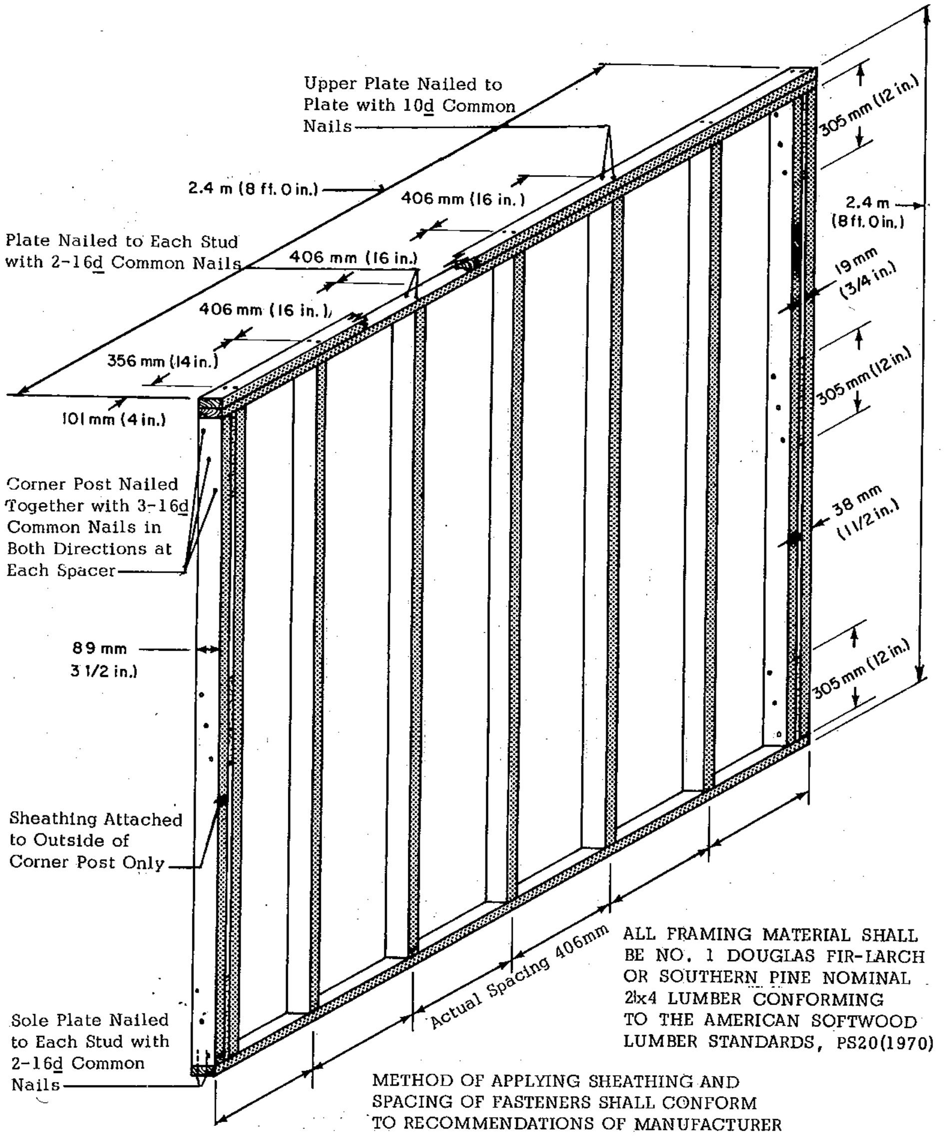


FIG. 5 Concentrated Load Test

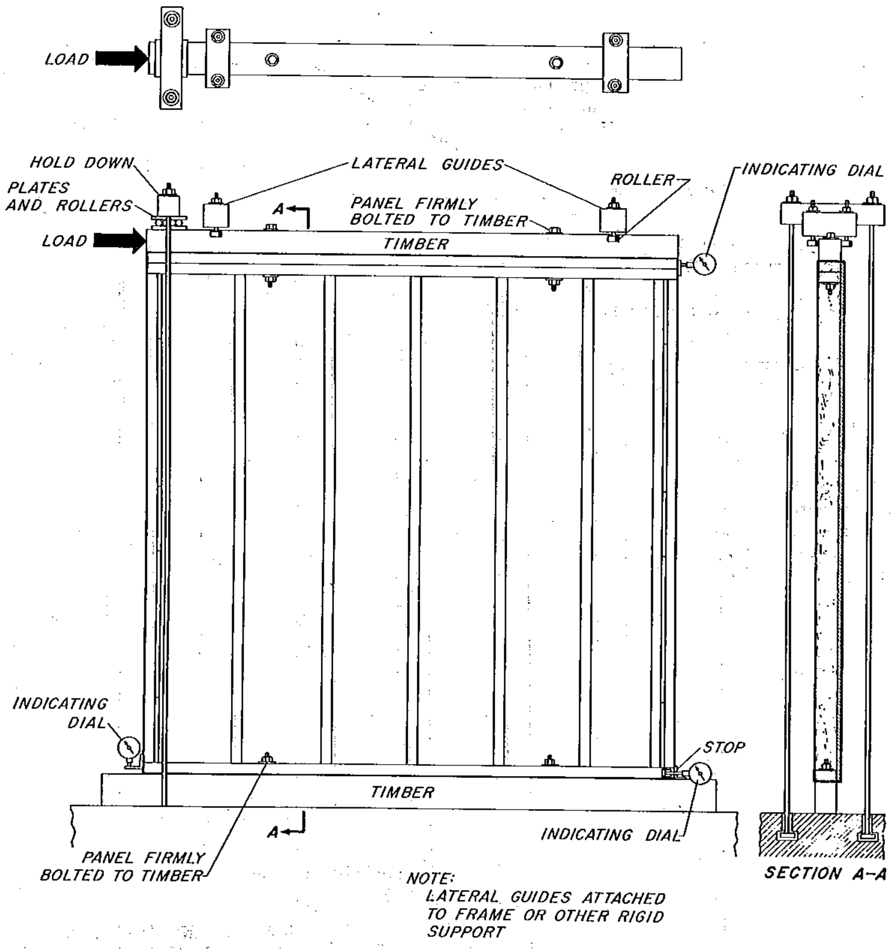


ALL FRAMING MATERIAL SHALL BE NO. 1 DOUGLAS FIR-LARCH OR SOUTHERN PINE NOMINAL 2x4 LUMBER CONFORMING TO THE AMERICAN SOFTWOOD LUMBER STANDARDS, PS20(1970)

METHOD OF APPLYING SHEATHING AND SPACING OF FASTENERS SHALL CONFORM TO RECOMMENDATIONS OF MANUFACTURER

NOTE—To eliminate test data that may be misleading, use lumber of average density for the species involved.

FIG. 6 Standard Wood Frame



NOTE:
LATERAL GUIDES ATTACHED
TO FRAME OR OTHER RIGID
SUPPORT

FIG. 7 Racking Load Assembly

APPENDIX

(Nonmandatory Information)

X1. TECHNICAL INTERPRETATION

X1.1 It is the purpose of these test methods to provide a systematic basis for obtaining comparable engineering data on various construction elements and structural details of value to designers, builders, building officials, and others interested in this field.

X1.2 Subjecting complete structures to known loads is very expensive and requires much time; therefore, that method of carrying out investigations to establish structural properties is not likely to be used to any great extent. Such tests have the further disadvantage that only the strength of the weakest elements of a particular structure could be measured.

X1.3 For these reasons, it seems more practicable to apply loads to specimens that accurately reproduce a structural portion of a finished building. These portions of a building have been designated as "elements"; for example, floor, wall, roof, etc. For the procedure described in these methods, the elements have been restricted to those most important structurally. For each element, methods of loading are described that simulate the loads to which the element would be subjected under service conditions. It is believed that the results of these measurements on the structural elements will be more useful to architects and engineers than the results of tests on specimens of the materials from which the structure was fabricated, or the results of tests of the individual structural members. Although it may be impracticable to determine all of the structural properties of each element of a building, it is believed that the more important properties may be determined by tests described in these methods.

X1.4 The test method, involving the application of the loads in increments and the concurrent measurement of deformation and set, simulates, to some extent, the conditions of repeated loading under service conditions. Therefore, results by such a method of loading may be more useful than those obtained by increasing the load continuously throughout the test. The results from increment loading tests may show whether different portions of a construction act as a unit under load, whether the fastenings or bonds have adequate strength, or whether they rupture under repeated loads. For any engineering structure, including small houses, it is necessary not only that the strength be adequate, but also that the deformation under load shall not appreciably decrease the usefulness of the structure. If the working load and the allowable deformation for an element for a structure are known, constructions complying with these requirements may be selected by inspection of the graphs from tests of such constructions.

X1.5 A structure is elastic if, after a load has been applied and then removed, the set is inappreciable. If the set is small for an element of a building, it may be assumed that the construction has neither been damaged nor appreciably deformed by the load. The set, therefore, is another property that may be used when comparing different constructions and may be useful when selecting a construction for a particular purpose.

X1.6 The variations in the properties of a construction as used commercially for buildings, in all probability, will be greater than the variations for the three specimens tested as directed in these methods because these specimens will be all fabricated at the same time by the same workmen and from the same lot of material. This fact should be clearly indicated in any general report based on these test procedures.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

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Standard Test Methods for Water Vapor Transmission of Materials[1]

This standard is issued under the fixed designation E 96; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

NOTE—Table 1 was corrected editorially and the year date was changed on March 6, 1995.

1. Scope

1.1 These test methods cover the determination of water vapor transmission (WVT) of materials through which the passage of water vapor may be of importance, such as paper, plastic films, other sheet materials, fiberboards, gypsum and plaster products, wood products, and plastics. The test methods are limited to specimens not over 1¼ in. (32 mm) in thickness except as provided in Section 9. Two basic methods, the Desiccant Method and the Water Method, are provided for the measurement of permeance, and two variations include service conditions with one side wetted and service conditions with low humidity on one side and high humidity on the other. Agreement should not be expected between results obtained by different methods. That method should be selected which more nearly approaches the conditions of use.

1.2 The values stated in inch-pound units are to be regarded as the standard. Metric inch-pound conversion factors for WVT, permeance, and permeability are stated in Table 1. All conversions of mm Hg to Pa are made at a temperature of 0°C.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 168 Terminology Relating to Thermal Insulating Materials[2]

D 449 Specification for Asphalt Used in Dampproofing and Waterproofing[3]

D 2301 Specification for Vinyl Chloride Plastic Pressure-Sensitive Electrical Insulating Tape[4]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[5]

TABLE 1 Metric Units and Conversion Factors[A,B]

Multiply	by	To Obtain (for the same test condition)
	WVT	
g/h·m^2	1.43	grains/h·ft^2
grains/h·ft^2	0.697	g/h·m^2
	Permeance	
g/Pa·s·m^2	1.75×10^{7}	1 Perm (inch-pound)
1 Perm (inch-pound)	5.72×10^{-8}	g/Pa·s·m^2
	Permeability	
g/Pa·s·m	6.88×10^{8}	1 Perm inch
1 Perm inch	1.45×10^{-9}	g/Pa·s·m

[A] These units are used in the construction trade. Other units may be used in other standards.

[B] All conversions of mm Hg to Pa are made at a temperature of 0°C.

3. Terminology

3.1 Definitions of terms used in this standard will be found in Terminology C 168, from which the following is quoted:

"*water vapor permeability*—the time rate of water vapor transmission through unit area of flat material of unit thickness induced by unit vapor pressure difference between two specific surfaces, under specified temperature and humidity conditions.

DISCUSSION—Permeability is a property of a material, but the permeability of a body that performs like a material may be used. Permeability is the arithmetic product of permeance and thickness.

water vapor permeance—the time rate of water vapor transmission through unit area of flat material or construction induced by unit vapor pressure difference between two specific surfaces, under specified temperature and humidity conditions.

DISCUSSION—Permeance is a performance evaluation and not a property of a material.

water vapor transmission rate—the steady water vapor flow in unit time through unit area of a body, normal to specific parallel surfaces, under specific conditions of temperature and humidity at each surface."

4. Summary of Test Methods

4.1 In the Desiccant Method the test specimen is sealed to the open mouth of a test dish containing a desiccant, and the assembly placed in a controlled atmosphere. Periodic weighings determine the rate of water vapor movement

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[1] These test methods are under the jurisdiction of ASTM Committee C-16 on Thermal Insulation and are the direct responsibility of Subcommittee C16.33 on Thermal Insulation Finishes and Vapor Transmission.

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[2] *Annual Book of ASTM Standards*, Vol 04.06.

[3] *Annual Book of ASTM Standards*, Vol 04.04.

[4] *Annual Book of ASTM Standards*, Vol 10.01.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

through the specimen into the desiccant.

4.2 In the Water Method, the dish contains distilled water, and the weighings determine the rate of vapor movement through the specimen from the water to the controlled atmosphere. The vapor pressure difference is nominally the same in both methods except in the variation, with extremes of humidity on opposite sides.

5. Significance and Use

5.1 The purpose of these tests is to obtain, by means of simple apparatus, reliable values of water vapor transfer through permeable and semipermeable materials, expressed in suitable units. These values are for use in design, manufacture, and marketing. A permeance value obtained under one set of test conditions may not indicate the value under a different set of conditions. For this reason, the test conditions should be selected that most closely approach the conditions of use. While any set of conditions may be used and those conditions reported, standard conditions that have been useful are shown in Appendix X1.

6. Apparatus

6.1 *Test Dish*—The test dish shall be of any noncorroding material, impermeable to water or water vapor. It may be of any shape. Light weight is desirable. A large, shallow dish is preferred, but its size and weight are limited when an analytical balance is chosen to detect small weight changes. The mouth of the dish shall be as large as practical and at least 4.65 in.^{2} (3000 mm^2). The desiccant or water area shall be not less than the mouth area except if a grid is used, as provided in 12.1, its effective area shall not exceed 10 % of the mouth area. An external flange or ledge around the mouth, to which the specimen may be attached, is useful when shrinking or warping occurs. When the specimen area is larger than the mouth area, this overlay upon the ledge is a source of error, particularly for thick specimens. This overlay material should be masked as described in 10.1 so that the mouth area defines the test area. The overlay material results in a positive error, indicating excessive water vapor transmission. The magnitude of the error is a complex function of the thickness, ledge width, mouth area, and possibly the permeability. This error is discussed by Joy and Wilson.[6] This type of error should be limited to about 10 to 12 %. For a thick specimen the ledge should not exceed ¾ in. (19 mm) for a 10-in. (254-mm) or larger mouth (square or circular) or ⅛ in. (3 mm) for a 5-in. (127-mm) mouth (square or circular). For a 3-in. (76-mm) mouth (square or circular) the ledge should not exceed 0.11 in. (2.8 mm) wide. An allowable ledge may be interpolated for intermediate sizes or calculated according to Joy and Wilson.[6] A rim around the ledge (Fig. X2.1) may be useful. If a rim is provided, it shall be not more than ¼ in. (6 mm) higher than the specimen as attached. Different depths may be used for the Desiccant Method and Water Method, but a ¾-in. (19-mm) depth (below the mouth) is satisfactory for either method.

6.2 *Test Chamber*—The room or cabinet where the assembled test dishes are to be placed shall have a controlled temperature and relative humidity. The temperature chosen shall be between 70 and 90°F (21 and 32°C), and shall be maintained constant within 1°F (0.6°C). A temperature of 90°F (32°C) is recommended (Note 1). The relative humidity shall be maintained at 50 ± 2 %, except where extremes of humidities are desired, when the conditions shall be 100 ± 1°F (38 ± 0.6°C) and 90 ± 2 % relative humidity. Both temperature and relative humidity shall be measured frequently, or preferably recorded continuously. Air shall be continuously circulated throughout the chamber, with a velocity sufficient to maintain uniform conditions at all test locations. The air velocity over the specimen in feet per minute shall be, numerically, not less than ten times the permeance of the specimen expressed in perms, not exceeding a maximum of 600 ft/min (3.05 m/s). However, for designed materials with a permeance of greater than 55 perms the velocity shall be 550 ± 50 ft/min (2.80 ± 25 m/s). Suitable racks shall be provided on which to place the test dishes within the test chamber.

NOTE 1—Simple temperature control by heating alone is usually made possible at 90°F (32°C). However, it is very desirable to enter the controlled space, and a comfortable temperature is more satisfactory for that arrangement. Temperatures of 73.4°F (23°C) and 80°F (26.7°C) are in use and are satisfactory for this purpose. With cyclic control, the average test temperature may be obtained from a sensitive thermometer in a mass of dry sand. The temperature of the chamber walls facing a specimen over water should not be cooler than the water to avoid condensation on the test specimen.

6.3 *Balance and Weights*—The balance shall be sensitive to a change smaller than 1 % of the weight change during the period when a steady state is considered to exist. The weights used shall be accurate to 1 % of the weight change during the steady-state period. For example: A 1-perm (5.7×10^{-11} $kg \cdot Pa^{-1} \cdot s^{-1} \cdot m^{-2}$) specimen 10 in. (254 mm) square at 80°F (26.7°C) passes 8.6 grains or 0.56 g/day. In 18 days of steady state, the transfer is 10 g. For this usage, the balance must have a sensitivity of 1 % of 10 g or 0.1 g and the weights must be accurate to 0.1 g. If, however, the balance has a sensitivity of 0.2 g or the weights are no better than 0.2 g, the requirements of this paragraph can be met by continuing the steady state for 36 days. An analytical balance that is much more sensitive will permit more rapid results on specimens below 1 perm (5.7×10^{-11} $kg \cdot Pa^{-1} \cdot s^{-1} \cdot m^{-2}$) when the assembled dish is not excessively heavy. A light wire sling may be substituted for the usual pan to accommodate a larger and heavier load.

7. Materials

7.1 *Desiccant and Water:*

7.1.1 For the Desiccant Method, anhydrous calcium chloride in the form of small lumps that will pass a No. 8 (2.36-mm) sieve, and free of fines that will pass a No. 30 (600-µm) sieve, shall be used (Note 2). It shall be dried at 400°F (200°C) before use.

NOTE 2—If $CaCl_2$ will react chemically on the specimen, an adsorbing desiccant such as silica gel, activated at 400°F (200°C), may be used; but the moisture gain by this desiccant during the test must be limited to 4 %.

7.1.2 For the Water Method, distilled water shall be used in the test dish.

7.2 *Sealant*—The sealant used for attaching the specimen

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[6] Joy, F. A., and Wilson, H. G., "Standardization of the Dish Method for Measuring Water Vapor Transmissions," National Research Council of Canada, Research Paper 279, January 1966, p. 263.

to the dish, in order to be suitable for this purpose, must be highly resistant to the passage of water vapor (and water). It must not lose weight to, or gain weight from, the atmosphere in an amount, over the required period of time, that would affect the test result by more than 2 %. It must not affect the vapor pressure in a water-filled dish. Molten asphalt or wax is required for permeance tests below 4 perms (2.3×10^{-10} $kg \cdot Pa^{-1} \cdot s^{-1} \cdot m^{-2}$). Sealing methods are discussed in Appendix X2.

8. Sampling

8.1 The material shall be sampled in accordance with standard methods of sampling applicable to the material under test. The sample shall be of uniform thickness. If the material is of nonsymmetrical construction, the two faces shall be designated by distinguishing marks (for example, on a one-side-coated sample, "I" for the coated side and "II" for the uncoated side).

9. Test Specimens

9.1 Test specimens shall be representative of the material tested. When a product is designed for use in only one position, three specimens shall be tested by the same method with the vapor flow in the designated direction. When the sides of a product are indistinguishable, three specimens shall be tested by the same method. When the sides of a product are different and either side may face the vapor source, four specimens shall be tested by the same method, two being tested with the vapor flow in each direction and so reported.

9.2 A slab, produced and used as a laminate (such as a foamed plastic with natural "skins") may be tested in the thickness of use. Alternatively, it may be sliced into two or more sheets, each being separately tested and so reported as provided in 9.4, provided also, that the "overlay upon the cup ledge" (6.1) of any laminate shall not exceed ⅛ in. (3 mm).

9.3 When the material as used has a pitted or textured surface, the tested thickness shall be that of use. When it is homogeneous, however, a thinner slice of the slab may be tested as provided in 9.4.

9.4 In either case (9.2 or 9.3), the tested overall thickness, if less than that of use, shall be at least five times the sum of the maximum pit depths in both its faces, and its tested permeance shall be not greater than 5 perms (3.3 metric perms).

9.5 The overall thickness of each specimen shall be measured at the center of each quadrant and the results averaged. Measurement of specimens of 0.125 in. or less in thickness shall be made to the nearest 0.0001 in. Measurement of specimens greater than 0.125 in. in thickness shall be made to the nearest 0.001 in.

9.6 When testing any material with a permeance less than 0.05 perms or when testing a low permeance material that may be expected to lose or gain weight throughout the test (because of evaporation or oxidation), it is strongly recommended that an additional specimen, or "dummy," be tested exactly like the others, except that no desiccant or water is put in the dish. Failure to use this dummy specimen to establish modified dish weights may significantly increase the time required to complete the test. Because time to reach equilibrium of water permeance increases as the square of thickness, thick, particularly hygroscopic, materials may take as long as 60 days to reach equilibrium conditions.

10. Attachment of Specimen to Test Dish

10.1 Attach the specimen to the dish by sealing (and clamping if desired) in such a manner that the dish mouth defines the area of the specimen exposed to the vapor pressure in the dish. If necessary, mask the specimen top surface, exposed to conditioned air so that its exposure duplicates the mouth shape and size and is directly above it. A template is recommended for locating the mask. Thoroughly seal the edges of the specimen to prevent the passage of vapor into, or out of, or around the specimen edges or any portion thereof. The same assurance must apply to any part of the specimen faces outside their defined areas. Suggested methods of attachment are described in Appendix X2.

NOTE 3—In order to minimize the risk of condensation on the interior surface of the sample when it is placed in the chamber, the temperature of the water prior to preparation of the test specimen should be within ±2°F (±1.1°C) of the test condition.

11. Procedure for Desiccant Method

11.1 Fill the test dish with desiccant within ¼ in. (6 mm) of the specimen. Leave enough space so that shaking of the dish, which must be done at each weighing, will mix the desiccant.

11.2 Attach the specimen to the dish (see 10.1) and place it in the controlled chamber, specimen up, weighing it at once. (This weight may be helpful to an understanding of the initial moisture in the specimen.)

11.3 Weigh the dish assembly periodically, often enough to provide eight or ten data points during the test. A data point is the weight at a particular time. The time that the weight is made should be recorded to a precision of approximately 1 % of the time span between successive weighing. Thus, if weighings are made every hour, record the time to the nearest 30 s; if recordings are made every day, a time to the nearest 15 min would be allowed. At first the weight may change rapidly; later a steady state will be reached where the rate of change is substantially constant. Weighings should be accomplished without removal of the test dishes from the controlled atmosphere, but if removal is prescribed necessary, the time the specimens are kept at different conditions, temperature or relative humidity, or both, should be kept to a minimum. When results of water vapor transmission are expected to be less than .05 perm, a dummy specimen is strongly recommended. Such a dummy specimen should be attached to an empty cup in the normal manner. The environmental effects of temperature variation and buoyancy variability due to barometric pressure fluctuation can be arithmetically tared out of the weighing values. This precaution permits earlier and more reliable achievement of equilibrium conditions. Analyze the results as prescribed in 13.1.

11.4 Terminate the test or change the desiccant before the water added to the desiccant exceeds 10 % of its starting weight (Notes 1 and 3). This limit cannot be exactly determined and judgement is required. The desiccant gain may be more or less than the dish weight-gain when the moisture content of the specimen has changed.

NOTE 4—The WVT of some materials (especially wood) may depend on the ambient relative humidity immediately before the test. An apparent hysteresis results in higher WVT if the prior relative humidity was above the test condition and vice versa. It is therefore recommended that specimens of wood and paper products be conditioned to constant weight in a 50 % relative humidity atmosphere before they are tested. Some specimens may be advantageously preconditioned to minimize the moisture that the specimen will give up to the desiccant. This applies when the specimen is likely to have high moisture content or when it is coated on the top (vapor source) side.

12. Procedure for Water Method

12.1 Fill the test dish with distilled water to a level ¾ ± ¼ in. (19 ± 6 mm) from the specimen. The air space thus allowed has a small vapor resistance, but it is necessary in order to reduce the risk of water touching the specimen when the dish is handled. Such contact invalidates a test on some materials such as paper, wood, or other hygroscopic materials. The water depth shall be not less than ⅛ in. (3 mm) to ensure coverage of the dish bottom throughout the test. However, if the dish is of glass, its bottom must be visibly covered at all times but no specific depth is required. Water surges may be reduced in placing a grid of light noncorroding material in the dish to break the water surface. This grid shall be at least ¼ in. (6 mm) below the specimen, and it shall not reduce the water surface by more than 10 % (Note 4).

NOTE 5—For the Water Method, baking the empty dish and promptly coating its mouth with sealant before assembly is recommended. The water may be added most conveniently after the specimen is attached, through a small sealable hole in the dish above the water line.

12.2 Attach the specimen to the dish (see 10.1). Some specimens are likely to warp and break the seal during the test. The risk is reduced by preconditioning the specimen, and by clamping it to the dish ledge (if one is provided).

12.3 Weigh the dish assembly and place it in the controlled chamber on a true horizontal surface. Follow the procedure given in 11.3. If the test specimen cannot tolerate condensation on the surface, the dish assembly shall not be exposed to a temperature that differs by more than 5°F (2.8°C) from the control atmosphere to minimize the risk of condensation on the specimen. When results of water vapor transmission are expected to be less than .05 perm, a dummy specimen is strongly recommended. Such a dummy specimen should be attached to an empty cup in the normal manner. The environment effects of temperature variation and buoyancy variability due to barometric pressure fluctuation can be arithmetically tared out of the weighing values. This precaution permits earlier and more reliable achievement of equilibrium conditions. Analyze the results as prescribed in 13.1.

12.4 Where water is expected to be in contact with the barrier in service, proceed as in 11.3 except place the dish in an inverted position. The dish must be sufficiently level so that water covers the inner surface of the specimen despite any distortion of the specimen due to the weight of the water. With highly permeable specimens it is especially important to locate the test dish so that air circulates over the exposed surface at the specified velocity. The test dishes may be placed on the balance in the upright position for weighing, but the period during which the wetted surface of the specimen is not covered with water must be kept to a minimum.

13. Calculation and Analysis of Results

13.1 The results of the rate of water vapor transmission may be determined either graphically or numerically.

13.1.1 *Dummy Specimen*—If a dummy specimen has been used to compensate for variability in test conditions, due to temperature or barometric pressure, or both, the daily recorded weights can be adjusted by calculating the weight change from initial to time of weighing. This adjustment is made by reversing the direction of the dummy's weight change, relative to its initial weight, and modifying all the appropriate specimen weight(s) recorded at this time. This permits earlier achievement of equilibrium conditions. An alternate procedure, particulary for tests of long duration and more than six weighings, is to subtract the arithmetic mean slope of the rate of weight change of the dummy specimen from the arithmetic mean slope of each similar specimen to get an effective rate of weight change. These procedures are also desirable if the specimen is changing weight due to a curing process while under test.

13.1.2 *Graphic Analysis*—Plot the weight, modified by the dummy specimen when used, against elapsed time, and inscribe a curve which tends to become straight. Judgment here is required and numerous points are helpful. When a straight line adequately fits the plot of at least six properly spaced points (periodic weight changes matching, or exceeding 20 % of the multiple of 100 times the scale sensitivity), a nominally steady state is assumed, and the slope of the straight line is the rate of water vapor transmission.

13.1.3 *Numerical Analysis*—A mathematical least squares regression analysis of the weight, modified by the dummy specimen when used, as a function of time will give the rate of water vapor transmission. An uncertainty, or standard deviation of this rate, can also be calculated to define the confidence band. For very low permeability materials, this method can be used to determine the results after 30 to 60 days when using an analytical balance, with a sensitivity of ±1 mg, even if the weight change does not meet the 100 times the sensitivity requirement of 6.3. Specimens analyzed in this manner must be clearly identified in the report.

13.2 Calculate the water vapor transmission, WVT, and permeance as follows:

13.2.1 *Water Vapor Transmission:*

$$\mathrm{WVT} = G/tA = (G/t)/A$$

where:

In inch-pound units:

G = weight change, grains (from the straight line),

t = time during which G occurred, h,

G/t = slope of the straight line, grains/h,

A = test area (cup mouth area), ft^2, and

WVT = rate of water vapor transmission, grains/h·ft^2.

In metric units:

G = weight change (from the straight line), g,

t = time, h,

G/t = slope of the straight line, g/h,

A = test area (cup mouth area), m^2, and

WVT = rate of water vapor transmission, g/h·m^2.

13.2.2 *Permeance:*

$$\text{Permeance} = \text{WVT}/\Delta p = \text{WVT}/S(R_1 - R_2)$$

where:

In inch-pound units:

Δp = vapor pressure difference, in. Hg,
S = saturation vapor pressure at test temperature, in. Hg,
R_1 = relative humidity at the source expressed as a fraction (the test chamber for desiccant method; in the dish for water method), and
R_2 = relative humidity at the vapor sink expressed as a fraction

In metric units:

Δp = vapor pressure difference, mm Hg (1.333×10^2 Pa),
S = saturation vapor pressure at test temperature, mm Hg (1.333×10^2 Pa),
R_1 = relative humidity at the source expressed as a fraction (the test chamber for desiccant method; in the dish for water method), and
R_2 = relative humidity at the vapor sink expressed as a fraction.

13.2.3 In the controlled chamber the relative humidity and temperature are the average values actually measured during the test and (unless continuously recorded) these measurements shall be made as frequently as the weight measurements. In the dish the relative humidity is nominally 0 % for the desiccant and 100 % for the water. These values are usually within 3 % relative humidity of the actual relative humidity for specimens below 4 perms (2.3×10^{-7} $\text{g}\cdot\text{Pa}^{-1}\cdot\text{s}^{-1}\cdot\text{m}^{-2}$) when the required conditions are maintained (no more than 10 % moisture in $CaCl_2$ and no more than 1 in. (25 mm) air space above water).

13.3 Only when the test specimen is homogeneous (not laminated) and not less than ½ in. (12.5 mm) thick, calculate its average permeability (perm in.) (metric perm-cm) as follows:

$$\text{Average permeability} = \text{permeance} \times \text{thickness}$$

NOTE 6: *Example*—In a desiccant test that ran 288 h (12 days) on an exposed area of 100 in.^{2} (0.0645 m^2), it was found that the rate of gain was substantially constant after 48 h and during the subsequent 240 h, the weight gain was 12 g. The controlled chamber conditions were measured at 89.0°F (31.7°C) and 49 % relative humidity.

Required: WVT and permeance

Calculation (inch-pound units):

$G/t = 12\text{ g} \times \dfrac{15.43\text{ grains}}{\text{g}} \div 240\text{ h}$
$= 0.771$ grains/h,
$A = 100\text{ in.}^2 \times \dfrac{1\text{ ft}^2}{144\text{ in.}^2} = 0.695\text{ ft}^2$,
S = 1.378 in. Hg (from standard references tables),
R_1 = 49 % (in chamber),
R_2 = 0 % (vapor sink), and
WVT = 0.771 grains/h ÷ 0.694 ft^2 = 1.11 grains/ft$^2\cdot$h.
Permeance = $\text{WVT}/\Delta P = \text{WVT}/S\,(R_1 - R_2)$
= 1.11 grains/ft$^2\cdot$h ÷ 1.378 in. Hg (0.49 − 0)
= 1.64 grains/ft$^2\cdot$h$\cdot$in. Hg = 1.64 perms

Calculation (metric units):

G/t = 12 g/240 h = 0.05 g/h,
A = 0.0645 m^2,
S = 35 mm Hg (from reference tables),
= 35 mm Hg × 1.333×10^2 Pa/mm Hg = 46.66×10^2 Pa,
R_1 = 49 % (in chamber),
R_2 = 0 % (vapor sink), and
WVT = 0.05 g/h ÷ 0.0645 m^2 = 0.775 g/h$\cdot$m^2.
Permeance = $\text{WVT}/\Delta P - \text{WVT}/S\,(R_1 - R_2)$
= 0.775 g/h$\cdot$m^2 × 1 h/3600s ÷ 46.66×10^2 Pa × (0.49 − 0)
= 9.42×10^{-8} g/Pa$\cdot$s$\cdot$m^2

13.4 Metric units and conversion factor are given in Table 1.

14. Report

14.1 The report shall include the following:

14.1.1 Identification of the material tested, including its thickness.

14.1.2 Test method used (desiccant or water).

14.1.3 Test temperature.

14.1.4 Relative humidity in the test chamber.

14.1.5 Permeance of each specimen in perms (to two significant figures).

14.1.6 The side of each specimen on which the higher vapor pressure was applied. (The sides shall be distinguished as "side A" and "side B" when there is no obvious difference between them. When there is an obvious difference, this difference shall also be stated, such as "side A waxed" and "side B unwaxed.").

14.1.7 The average permeance of all specimens tested in each position.

14.1.8 The permeability of each specimen (as limited by 13.3), and the average permeability of all specimens tested.

14.1.9 Include a portion of the plot indicating the section of the curve used to calculate permeability.

14.1.10 State design of cup and type or composition of sealant.

15. Precision and Bias[7]

15.1 *Precision*—Table 2 is based on an interlaboratory tests conducted in 1988 and 1991.[7] In 1988 four materials (A, B, C, D) were tested using the dessicant method and the water method in triplicate. Fifteen laboratories contributed data, with full results secured from four laboratories. In 1991 ten laboratories contributed data for material E, using triplicate specimens, again using both the dessicant method and the water method.

15.1.1 Test results were analyzed using Practice E 691.

15.2 *Bias*—This test method has no bias because water vapor transmission of materials is defined in terms of this test method.

16. Keywords

16.1 permeability; plastics (general), plastic sheet and film; sheet material; thermal-insulating materials; thermal insulation permeability films; water vapor transmission (WVT).

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[7] Supporting data have been filed at ASTM Headquarters. Request RR: C-16-1014.

TABLE 2 Precision Results from Interlaboratory Testing

For Desiccant Method at 73.4°F:

Material	Thickness, in.	WVT (mean), perm	Repeatability^A			Reproducibility^A		
			S	*CV* %	*LSD*	*S*	*CV* %	*LSD*
A	0.001	0.606	0.0166	2.70	0.047	0.098	15.0	0.278
B	0.0055	0.0129	0.0028	22.1	0.008	0.0055	42.6	0.016
C	0.5	0.0613	0.0044	7.22	0.012	0.0185	30.6	0.052
D	1.0	0.783	0.0259	3.30	0.073	0.0613	7.8	0.174
E	0.014	0.0461	0.0023	4.99	0.007	0.0054	11.7	0.015

For Water Method at 73.4°F:

Material	Thickness, in.	WVT (mean), perm	Repeatability^A			Reproducibility^A		
			S	*CV* %	*LSD*	*S*	*CV* %	*LSD*
A	0.001	0.715	0.0134	1.95	0.039	0.156	21.9	0.44
B	0.0055	0.0157	0.0022	13.8	0.0062	0.0021	19.4	0.006
C	0.5	0.097	0.0055	5.7	0.016	0.0195	20.9	0.055
D	1.0	1.04	0.0192	1.8	0.054	0.217	20.9	0.62
E	0.014	0.0594	0.0034	5.7	0.010	0.0082	13.8	0.023

[A] For this data,

S = standard deviation,
CV = percent coefficient of variation ($S \times 100/\text{mean}$), and
LSD = least significant difference between two individual test results based on a 95 % confidence level = $2\sqrt{2}S$.

[B] Material B was Teflon[8] PTFE fluorocarbon resin brand of tetrafluoroethylene. It was extremely difficult to provide a seal to this sample, which accounts for the poor repeatability.

APPENDIXES

(Nonmandatory Information)

X1. STANDARD TEST CONDITIONS

X1.1 Standard test conditons that have been useful are:

X1.1.1 *Procedure A*—Desiccant Method at 73.4°F (23°C).

X1.1.2 *Procedure B*—Water Method at 73.4°F (23°C).

X1.1.3 *Procedure BW*—Inverted Water Method at 73.4°F (23°C).

X1.1.4 *Procedure C*—Desiccant Method at 90°F (32.2°C).

X1.1.5 *Procedure D*—Water Method at 90°F (32.2°C).

X1.1.6 *Procedure E*—Desiccant Method at 100°F (37.8°C).

X2. CUP DESIGN AND SEALING METHODS

X2.1 An ideal sealing material has the following properties:

X2.1.1 Impermeability to water in either vapor or liquid form.

X2.1.2 No gain or loss of weight from or to the test chamber (evaporation, oxidation, hygroscopicity, and water solubility being undersirable).

X2.1.3 Good adhesion to any specimen and to the dish (even when wet).

X2.1.4 Complete conformity to a rough surface.

X2.1.5 Compatibility with the specimen and no excessive penetration into it.

X2.1.6 Strength or pliability (or both).

X2.1.7 Easy handleability (including desirable viscosity and thermal of molten sealant).

X2.1.8 Satisfactory sealants possess these properties in varying degrees and the choice is a compromise, with more tolerance in items at the beginning of this list for the sake of those at the latter part of the list when the requirements of 7.2 are met (Note A2). Molten asphalt or wax is required for permeance tests below 4 perms (2.6 metric perms). Tests to determine sealant behavior should include:

X2.1.8.1 An impervious specimen (metal) normally sealed to the dish and so tested, and

X2.1.8.2 The seal normally assembled to an empty dish with no specimen and so tested.

X2.2 The following materials are recommended for general use when the test specimen will not be affected by the temperature of the sealant:

X2.2.1 Asphalt, 180 to 200°F (82 to 93°C) softening point, meeting the requirements of Specification D 449, Type C. Apply by pouring.

X2.2.2 Beeswax and rosin (equal weights). A temperature of 275°F (135°C) is desirable for brush application. Pour at lower temperature.

X2.2.3 Microcrystalline wax[9] (60 %), mixed with refined crystalline paraffin wax (40 %).

X2.3 The materials listed in X2.3.1 are recommended for particular uses such as those shown in Figure X2.1. The suggested procedure described in X2.3.2 applies to an 11-3/8-in. (289-mm) square specimen if its permeance exceeds 4 perms (2.6 metric perms) (limited by evaporation of sealants).

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[8] Available from E. I. DuPont de Nemours & Co., Inc., Polymer Products Dept., Wilmington, DE 19898.

[9] Grade Nos. 2305 or 2310 of the Mobil Oil Corp., or their equivalent, have been found satisfactory for this purpose.

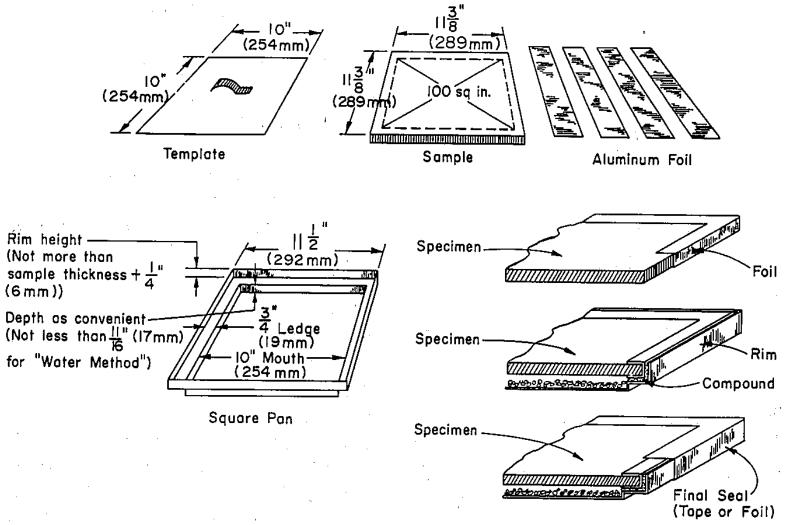

FIG. X2.1 Apparatus for Water Vapor Transmission Tests of Large Thick Specimens

X2.3.1 *Materials:*

X2.3.1.1 Aluminum foil, 0.005 in. (0.125 mm) minimum thickness.

X2.3.1.2 Tape, meeting the requirements of Specification D 2301, vinyl chloride plastic pressure-sensitive, electrical insulating tape.

X2.3.1.3 Cement, contact bond, preferably rubber base.

X2.3.2 *Procedure:*

X2.3.2.1 *Step 1*—Seal aluminum foil around edges of specimen, leaving a 100-in.^{2} (0.0654-m^2) exposed test area on each side. Use contact bond cement as directed by the manufacturer.

X2.3.2.2 *Step 2*—Spread sealant on inside of rim and ledge. Place desiccant (dry), or water and surge control material (wet) in pan. Press specimen in place. Avoid squeezing compound into the test area.

X2.3.2.3 *Step 3*—Coat outside of rim and bottom of ledge with contact bond cement, and place foil strips from edge of template, around rim, and bottom of ledge.

X2.4 A method of using hot asphalt, as applied to a 10-in. (254-mm) square-mouth dish with ledge and rim, is as follows:

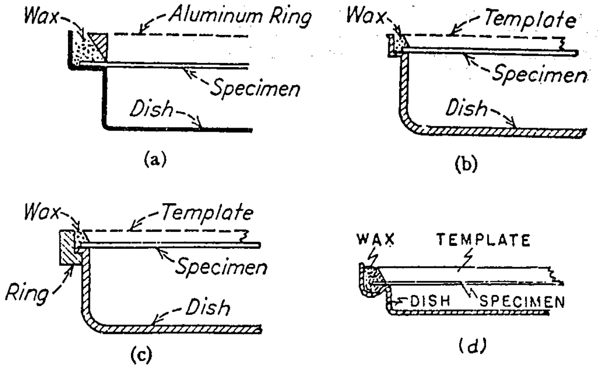

FIG. X2.2 Several Types of Dishes for Water Vapor Transmission Tests of Materials in Sheet Form

X2.4.1 *Apparatus:*

X2.4.1.1 *Template*—A square frame of brass or steel, 3/16 in. (5 mm) thick and ¾ in. (19 mm) deep. The 3/16-in. (5-mm) thickness is tapered to zero at the bottom of the frame where it will touch the test specimen and maintain a 10-in. (254-mm) square test area.

X2.4.1.2 *Sealant*—Asphalt (see X2.2.1 used at the proper pouring consistency of 375 to 450°F (179 to 232°C).

X2.4.1.3 *Melting Pot* for the asphalt, electrically heated, with one dimension greater than 11⅜ in. (289 mm).

X2.4.1.4 *Small Ladle* for pouring.

X2.4.2 *Procedure*—Mark the 11⅜-in. (289-mm) square specimen with a line at an equal distance from each edge, so that the area enclosed by the lines is as nearly as possible a 10-in. (254-mm) square. The template may be used for marking. Dip each edge of the specimen in molten asphalt up to the line, so that the test area is defined and all edges are coated with a heavy layer of asphalt. Place the specimen over the pan containing water or desiccant. Lightly oil the template or coat with petroleum jelly on its outer side, and place on the specimen. Pour molten asphalt into the space between the template and the rim of the pan. After the asphalt has cooled for a few minutes, the template should be easily removable.

X2.5 Hot wax may be applied like asphalt. It may also be applied (freely) with a small brush. Its lower working temperature may be advantageous when a specimen contains moisture.

X2.6 Several designs for dishes with supporting rings and flanges are shown in Fig. X2.2. Various modifications of these designs may be made provided that the principle of prevention of edge leakage by means of a complete seal is retained. The dishes may be constructed of any rigid, impermeable, corrosion-resistant material, provided that they can be accommodated on the available analytical balance. A lightweight metal, such as aluminum or one of its alloys, is generally used for larger-size dishes. In some cases

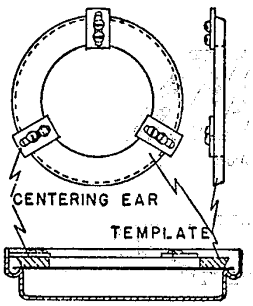

FIG. X2.3 Template Suitable for Use in Making the Wax Seals on Test Dishes

when an aluminum dish is employed and moisture is allowed to condense on its surface, there may be appreciable oxidation of the aluminum with a resulting gain in weight. Any gain in weight will ordinarily depend on the previous history of the dish and the cleanness of the surface. An empty dish carried through the test procedure as a control will help to determine whether any error may be expected from this cause. When aluminum dishes are used for the water methods, a pressure may develop inside the assembly during a test due to corrosion. This can cause seal failure or otherwise affect the result. Where this is a problem, it can be overcome by providing inside the dish a protective coating of baked-on epoxy resin or similar material. Dishes with flanges or rings that project from the inner walls of the dish are to be avoided, as such projections influence the diffusion of the water vapor. The depth of the dish for the water procedures is such that there is a 0.80 ± 0.20 in. (20 ± 5-mm) distance between the water surface and the under surface of the specimen, with a water depth of about 0.20 in. (5 mm).

X2.6.1 For the desiccant-in-dish procedures, the dishes need not be as deep as those required for the water-in-dish procedures. The desiccant is within ¼ in. (6 mm) of the under surface, and a minimum depth of only ½ in. (12 mm) of desiccant is required.

X2.6.2 The dishes shown in Fig. X2.2 require a molten seal.

X2.6.3 A template such as is shown in Fig. X2.3 is usually used for defining the test area and effecting the wax seal. It consists of a circular metal dish ⅛ in. (3.18 mm) or more in thickness with the edge beveled to an angle of about 45°. The diameter of the bottom (smaller) face of the template is approximately equal to, but not greater than, the diameter of the effective opening of the dish in contact with the specimen. Small guides may be attached to the template to center it automatically on the test specimen. A small hole through the template to admit air, and petrolatum applied to the beveled edge of the template facilitate its removal after sealing the test specimen to the dish. In use, the template is placed over the test specimen and when it is carefully centered with the dish opening, molten wax is flowed into the annular space surrounding the beveled edge of the template. As soon as the wax has solidified, the template is removed from the sheet with a twisting motion. The outside flange of the dish should be high enough to extend over the top of the specimen, thus allowing the wax to completely envelop the edge.

X2.6.4 Gasketed types of seals are also in use on appropriately designed dishes. These simplify the mounting of the specimen, but must be used with caution, since the possibility of edge leakage is greater with gasketed seals than with wax seals. Gasketed seals are not permitted for the measurement of permeance less than 44 perms (2.3×10^{-7} $g \cdot Pa^{-1} \cdot s^{-1} \cdot m^2$). As a further precaution when gasketed seals are used instead of preferred sealants, a blank test run is suggested using glass or metal as a dummy specimen.

X2.6.5 A suitable weighing cover consists of a circular disk of aluminum 1⁄32 to 3⁄32 in. (0.8 to 2.4 mm) in thickness provided with a suitable knob in the center for lifting. The cover fits over the test specimen when assembled and makes contact with the inside beveled surface of the wax seal at, or just above, the plane of the specimen. The cover is free of sharp edges which might remove the wax and is numbered or otherwise identified to facilitate its exclusive use with the same dish.

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Standard Specification for Gravity-Convection And Forced-Ventilation Ovens[1]

This standard is issued under the fixed designation E 145; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

$^{\epsilon 1}$ NOTE—Section 7 on Keywords was added editorially March 1995.

1. Scope

1.1 This specification covers the performance requirements for general-purpose air ovens ordinarily used in testing operations, which have a testing chamber up to 0.6 m^3 (25 ft^3) in volume. It is applicable to gravity-convection ovens designed to operate over all or part of the temperature range from 20°C above ambient temperature to 200°C and to forced-ventilation ovens designed to operate over all or part of the temperature range from 20°C above ambient temperature to 500°C.

NOTE 1—Ovens are designed for maximum operating temperatures of about 200°C, 300°C, and 500°C, the thermal insulation and cost of the oven being dependent on the maximum temperature required.

1.2 This specification does not include safety requirements that are essential for ovens used in the presence of combustible vapors or gases.

1.3 The values stated in inch-pound units are to be regarded as the standard. The metric equivalents of inch-pound units may be approximate.

2. Types

2.1 This specification covers the following four types of air ovens:

2.1.1 *Type IA*—An oven ventilated by gravity convection having a uniformity of temperature within ±2 % of the differential between oven and ambient temperatures.

2.1.2 *Type IB*—An oven ventilated by gravity convection having a uniformity of temperature within ±5 % of the differential between oven and ambient temperatures.

2.1.3 *Type IIA*—An oven having forced ventilation and a uniformity of temperature within ±1 % of the differential between oven and ambient temperatures.

2.1.4 *Type IIB*—An oven having forced ventilation and a uniformity of temperature within ±2.5 % of the differential between oven and ambient temperatures.

3. Performance Requirements

3.1 The temperature within the testing chamber shall be controllable by an automatic device, and shall be uniform within the tolerances given in Table 1 for the particular type of oven when tested in accordance with Section 4.

3.2 The "time constant" is an arbitrary measure of the rate at which a standard specimen is heated following the procedure prescribed in Section 5. The value of the time constant shall not exceed the maximum value given in Table 1 for the particular type of oven.

3.3 The rate of ventilation of the testing chamber shall conform to the requirements specified in Table 1 for the particular type of oven when measured in accordance with the procedure given in Section 6.

TEST METHODS

4. Temperature Uniformity

4.1 Place nine calibrated thermocouples (Note 2) made from iron or copper-constantan wire, approximately 0.5 mm in diameter (No. 24 gage) and having a junction size of not more than 2 mm (0.08 in.), in the empty testing chamber with shelves in place and vents open. Locate one thermocouple in each of the eight corners of the oven approximately 5 cm (2 in.) from each wall and place the ninth thermocouple within 2.5 cm (1 in.) of the geometric center of the chamber. A minimum length of 30 cm (12 in.) of lead wire for each thermocouple shall be inside the oven to minimize the conduction of heat from the thermocouple.

NOTE 2—If calibrated thermocouples are not available, nine thermocouples made from the same spool of wire may be used provided they give the same value for temperature when placed adjacent to one another in the testing chamber at the temperature of test.

4.2 Bring the oven to the specified temperature and allow it to reach a steady state (Note 3). Record the temperatures of the nine thermocouples for a period of at least 24 h, and determine from the record the maximum deviation of each point from the desired temperatures. The ambient room temperature shall vary by not more than a total of 10°C, and the line voltage for electrically heated ovens shall vary by not more than a total of 5 % during the test.

NOTE 3—Some ovens may require as much as 24 h to reach a steady state. If a steady state does not exist, there is a drift in the temperature toward the steady-state condition.

5. Time Constant

5.1 Heat the oven to within 10°C of the maximum operating temperature for which it is designed and allow it to stabilize for at least 1 h. Prepare a standard specimen consisting of a smooth brass cylinder 9.5 ± 0.1 mm (0.375 ± 0.005 in.) in diameter and 57 ± 1 mm (2.25 ± 0.05 in.) in length, and solder one junction of a differential thermocouple to it.

5.2 Open the door of the oven for 1 min while the standard specimen and differential thermocouple are being

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[1] This specification is under the jurisdiction of ASTM Committee E-41 on Laboratory Apparatus and is the direct responsibility of Subcommittee E 41.02 on Metalware.

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TABLE 1 Performance Requirements for Ovens

Characteristic	Type IA	Type IB	Type IIA	Type IIB
Deviation from specified temperature of test throughout testing chamber during 24-h period for the following differentials between ambient and test temperatures:				
50°C or less, max, °C	1	2.5	0.5	1.25
More than 50°C, max, percent of differential	2	5	1	2.5
Time constant, max, s	600	720	480	660
Rate of ventilation of testing chamber, air changes per hour:				
min	10	10	50	50
max	...	...	200	200

suspended in the testing chamber. Suspend the specimen vertically within 25 mm (1 in.) of the geometric center of the chamber by means of an asbestos cord of fine wire (0.3 mm maximum diameter, No. 30 gage). Place the free junction of the differential thermocouple in the air space of the chamber at least 75 mm (3 in.) removed from the specimen. Then close the door and either record or measure the temperature differential every 10 s. Determine the time in seconds required for the temperature difference to decrease to one tenth of the original or maximum value (for example, from 120°C to 12°C) and consider this to be the time constant of the oven.

6. Rate of Ventilation (Note 4)

6.1 Seal the ventilation ports, door, and all apertures of electrically heated ovens with adhesive tape or by other means to prevent any air from passing through the oven (Note 5). Connect a watt-hour meter, with the smallest division reading in 0.01 Wh in the electrical supply line to the oven.

NOTE 4—This method is only applicable to electrically heated ovens. Methods are being developed by the committee for determining the rate of ventilation of ovens that are not electrically heated and for determining the uniformity of air-flow within the testing chamber.

NOTE 5—In forced-ventilation ovens, the space around the motor shaft where it enters the oven must be closed, but the fan speed must not be affected by the closure.

6.2 Heat the oven to a temperature of 80 ± 2°C above the ambient room temperature, and while at this temperature measure the consumption of electrical energy for a period of at least ½ h. Start and stop the test at corresponding points of the "on-off" heating cycle, that is, at the moment when the heaters are switched on by the thermostat.

6.3 Then remove the seals, open the ventilation ports, and measure the consumption of electrical energy in the same manner. The ambient room temperature measured at a point approximately 2 m (6 ft) from the oven, approximately level with its base and at least 0.6 m (2 ft) from any solid object, shall be the same within 0.2°C during the two tests.

6.4 Calculate the number of changes per hour of the air in the test chamber from the following equation:

$$N = 3590\,(X - Y)/VD\Delta T$$

where:

N = number of air changes per hour,

X = average power consumption during ventilation, W, obtained by dividing the energy consumption determined from the watt-hour meter readings by the duration of the test in hours,

Y = average power consumption with no ventilation, computed in the same manner, W,

V = volume of the testing chamber, cm^3,

D = density of the ambient room air during the test, g/cm^3, and

ΔT = difference in temperature between the testing chamber and the ambient room air, °C.

7. Keywords

7.1 forced-ventilation; gravity-convection; ovens

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WASHINGTON, D.C.

Designation: E 169 – 87

Standard Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis[1]

1. Scope

1.1 These practices are intended to provide general information on the techniques most often used in ultraviolet and visible quantitative analysis. The purpose is to render unnecessary the repetition of these descriptions of techniques in individual methods for quantitative analysis.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 131 Definitions of Terms and Symbols Relating to Molecular Spectroscopy[2]

E 168 Practices for General Techniques of Infrared Quantitative Analysis[2]

E 275 Practice for Describing and Measuring the Performance of Ultraviolet, Visible, and Near-Infrared Spectrophotometers[2]

E 387 Test Method for Estimating Stray Radiant Power Ratio of Spectrophotometers by the Opaque Filter Method[2]

E 925 Practice for Periodic Calibration of Narrow Band-Pass Spectrophotometers[2]

E 958 Practice for Measuring Practical Spectral Bandwidth of Ultraviolet-Visible Spectrophotometers[2]

3. Summary of Practice

3.1 Quantitative ultraviolet and visible analyses are based upon the absorption law, known as Beer's law. The units of this law are defined in Definitions E 131. Beer's law (Note 1) holds at a single wavelength and when applied to a single component sample it may be expressed in the following form (see Section 10):

$$A = abc$$

When applied to a mixture of n noninteracting components, it may be expressed as follows:

$$A = a_1bc_1 + a_2bc_2 + \dots + a_nbc_n$$

NOTE 1—Detailed discussion of the origin and validity of Beer's law may be found in the books and articles listed in the bibliography at the end of these practices.

3.2 This practice describes the application of Beer's law in typical spectrophotometric analytical applications. It also describes operating parameters that must be considered when using these techniques.

4. Significance and Use

4.1 These practices are a source of general information on the techniques of ultraviolet and visible quantitative analyses. They provide the user with background information that should help ensure the reliability of spectrophotometric measurements.

4.2 These practices are not intended as a substitute for a thorough understanding of any particular analytical method. It is the responsibility of the user to familiarize him or herself with the critical details of a method and the proper operation of the available instrumentation.

5. Sample Preparation

5.1 Accurately weigh the specified amount of the sample (solid or liquid). Dissolve in the appropriate solvent and dilute to the specified volume in volumetric glassware of the required accuracy. (Solvent and flask should be approximately the same temperature as the spectrophotometer). If needed, a dilution should be made with a calibrated pipet and volumetric flask, using adequate volumes for accuracy. Fill the absorption cell with the solution, and fill the comparison or blank cell with the pure solvent.

6. Cell and Base-Line Checks

6.1 Clean and match the cells. Suggested cleaning procedures are presented in Practice E 275.

6.2 Establish the base-line of a recording double-beam spectrophotometer by scanning over the appropriate wavelength region with pure solvent in both cells. Determine apparent absorbance of the sample cell at each wavelength of interest. These absorbances are cell corrections that are subtracted from the absorbance of the sample solution at the corresponding wavelengths.

6.3 For single beam instruments, either use the same cell for pure solvent and sample measurements, use matched cells, or apply appropriate cell corrections.

6.4 For some newer instruments, the cell corrections or the blank cell absorbance is stored in memory and automatically incorporated into the sample absorbance measurement.

6.5 An accurate determination of cell path length in the 1-cm range is not practical in most laboratories, and

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[1] These practices are under the jurisdiction of ASTM Committee E-13 on Molecular Spectroscopy and are the direct responsibility of Subcommittee E13.01 on Ultraviolet and Visible Spectroscopy.

Current edition approved Feb. 1, 1987. Published April 1987. Originally published as E 169 – 60 T. Last previous edition E 169 – 63 (81)$^{\epsilon1}$.

[2] *Annual Book of ASTM Standards,* Vol 14.01.

common practice is to purchase cells of known path length.[3] A check on path length matching, however, may be made by measuring the absorbance of a strongly absorbing solution (A~0.9) versus pure solvent, and then emptying, cleaning, reloading fresh solvent and sample into the other cells, and remeasuring the absorbance. Similarly, the absorbance of the sample in a series of cells to be used in an analysis can be measured versus pure solvent in a given reference cell.

7. Analytical Wavelengths and Photometry

7.1 Analytical wavelengths are those wavelengths at which absorbance readings are taken for use in calculations. These may include readings taken for purposes of background corrections. The analytical wavelengths are frequently chosen at absorption maxima, but this is not always necessary. For example, the use of isoabsorptive or isosbestic points is frequently useful.

7.2 On manually operated spectrophotometers, record the absorbance readings at the specified analytical wavelengths, operating the instrument in accordance with the recommendations of the manufacturer or Practice E 275. On automatic recording spectrophotometers, record the absorbance readings from the chart at the analytical wavelengths. For some newer instruments, the absorbance may be displayed on a suitable output device (for example, a CRT, DVM, etc.), or used directly in calculations.

7.2.1 The wavelength region scanned may be somewhat greater than that required to contain only the analytical wavelengths. Take care to start the chart and scanning drives exactly together when using variable slit instruments. End the scan well before the slit has opened fully.

7.3 Absorbance values should be used only if they fall within the acceptably accurate range of the particular spectrophotometer and method employed. If the absorbance is too low, either use a longer absorption cell or prepare a new solution of higher concentration. If the absorbance is too high, use a shorter cell or make a quantitative dilution. If different cells are used, a new base-line must be obtained.

7.4 The precision and bias of the wavelength and photometric scales of the instrument must be adequate for the method being used. Procedures for checking precision and accuracy of these scales are presented in Practices E 275 and E 925.

8. Spectral Band Width and Slit Width

8.1 If the analytical method specifies a spectral band width or a spectral slit width, set the spectral band width of the instrument to the specified value. If the instrument has only a mechanical slit width indicator, use the information provided in the manufacturer's literature to calculate the slit width that corresponds to the specified spectral band width.

NOTE 2—The accuracy of spectral band width and mechanical slit width indicators can be determined using the procedure given in Practice E 958.

8.2 If the analytical method specifies a mechanical slit width for a particular type of instrument and the same type of instrument is being used, set the slit width to the specified values. If a different type of instrument is being used and information is available from which the spectral slit width of both types of instruments can be calculated, adjust the instrument settings to obtain a spectral slit width equal to the one calculated from the specified mechanical slit width.

8.3 If the analytical method does not state a spectral band width or a slit width value but includes a spectrum illustrating adequate resolution, set the spectral band width or slit width of the instrument to obtain comparable resolution.

8.4 If the method neither specifies spectral band width or slit width nor provides an illustrative spectrum, use the smallest spectral band width or slit width that yields an acceptable signal-to-noise ratio. Record this value for future reference.

NOTE 3—Changes in the day-to-day values of spectral band width or slit width obtained with a given gain, or changes in gain required to obtain a given spectral band width or slit width, are indicative of present or potential problems. Increased spectral band width or gain may result from a lower output of the light source, deterioration of optical components, deposits on the windows of the cell compartment or on the inside wall of the reference cell, an absorbing impurity in the solvent, or a faulty electronic component.

9. Solvents and Solvent Effects

9.1 The ultraviolet absorption spectrum of a compound will vary in different solvents depending on the chemical structures involved. Nonpolar solvents have the least effect on the absorption spectrum. Nonpolar molecules in most instances are not affected in polar solvents. However, polar molecules in polar solvents may show marked differences in their spectra. Any interaction between solute and solvents leads to a broadening and change in structural resolution of the absorption bands. Ionic forms may be created in acidic or basic solutions. In addition, there are possible chemical reactions between solute and solvent, and also photochemical reactions arising from either room illumination or the short wavelengths in the beam of the spectrophotometer. It is important that the solvent used be specified in recording spectral data. (The change in spectra between acidic and basic conditions may sometimes be employed in multicomponent analysis.)

9.2 Common commercially available solvents of "spectroscopic purity" are listed in Table 1. The short wavelength limit is approximate, and refers to the wavelength at which a 1-cm light path length gives an absorbance of unity.

9.3 Water, and 0.1 *N* solutions of hydrochloric acid, sulfuric acid, and sodium hydroxide also are commonly used as solvents. Buffered solutions, involving nonabsorbing materials, are frequently used; both the composition of the buffer and the measured pH should be specified. Mixtures of 0.1 *N* dihydrogen sodium phosphate and 0.1 *N* hydrogen disodium phosphate are useful in the 4.5 to 8.9 pH range. A table of nonabsorbing buffers has been presented by Abbott **(8)**.[4]

10. Calculations

10.1 Quantitative analysis by ultraviolet spectropho-

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[3] A 1-cm cell having its pathlength certified to 0.005 cm is available as Standard Reference Material 932 from National Bureau of Standards (NBS), Department of Commerce, Washington, DC 20234.

[4] The **boldface** numbers in parentheses refer to the list of references at the end of this standard.

TABLE 1 Solvents[A]

Solvent	Cutoff, nm
Pyridine	305
Tetrachloroethylene	290
Benzene	280
N,N-Dimethylformamide	270
Carbon tetrachloride	265
Methyl formate	260
Chloroform	245
Dichloromethane	235
Ethyl ether	220
Acetonitrile	215
Isopropyl alcohol	210
Ethyl alcohol	210
Methyl alcohol	210
Cyclohexane	<210
*Iso*octane	<210

[A] Procedures for special purification of solvents for further improvement in the wavelength limit are given in Refs **(11, 12)**. Solvents of high purity for use in absorption spectroscopy also are available commercially as follows:

Solvents for Spectrophotometric Use, Distillation Products Industries, Division of Eastman Kodak Co., Rochester, NY 14603

Spectranalyzed^{tm} solvents, Fisher Scientific, An Allied Company, 711 Forbes Ave, Pittsburgh, PA 15219

Spectroquality Solvents, Matheson, Coleman and Bell, 11-38 31st Ave., Long Island City, NY 11106

tometry depends upon Beer's law. The terms and symbols used are those defined in Definitions E 131. According to Beer's law:

$$A = abc = (\epsilon/M) \times bc$$

where:

A = absorbance,
a = absorptivity,
b = cell length, cm,
c = concentrations, g/L,
ϵ = molar absorptivity, and
M = molecular weight.

10.1.1 In practice, a distinction must be made between c, the concentration of the absorbing material in the cell at the time of observation, and the concentration of the *absorbing material* in the *sample as received.* This is here designated as C and is in weight percent (g/100 g). The solution to be examined has a concentration of *sample* in solution, which is in units of grams per litre.

$$c = A/ab$$

$$C, \% = (c/C_3) \times 100 = (A/abC_s) \times 100$$

10.2 If one or more dilutions have been made, the quantity called the dilution factor must be included. Dilution factor, f, is the ratio of the final volume to the initial volume. If more than one dilution is performed, the dilution factor is the product of the factors from each dilution. If dilutions are made, the equation becomes the following:

$$C, \% = (c/C_s) \times 100 = (Af/abC_s) \times 100$$

Note that c and C_s, have the dimensions of grams per litre. If dilution is made, C_s is not the concentration in the cell at the time the absorbance is determined; the concentration in the cell is C_s/f.

10.3 *Reference Material*—The absorptivity of the absorbing material, the concentration of which it is desired to determine, is obtained by examination of a pure sample of this material, which is called a reference material. However, if no such pure material is available, the best available material is used, or a value of the absorptivity is taken from the literature. Take care to specify this, by reporting values as "percentage against reference material" or by noting that the accuracy of the analysis is dependent upon a published value of the absorptivity or molar absorptivity. (A reference must be cited.)

10.4 *Types of Analyses* (see Fig. 1):

10.4.1 *One Component, No Background Correction:*

$$C, \% = (Af/abC_s) \times 100$$

10.4.2 *One Component, Simple Background Correction:*

$$C, \% = \frac{(A_1 - A_2) \times f}{a_1 b C_s} \times 100$$

where the subscripts refer to analytical wavelengths. The term A_2 is the absorbance at the wavelength used for making a simple subtractive correction. It is usually selected from examination of the spectral curve of the reference material at a wavelength longer than that of A_1, preferably where a_2 is equal to or less than g/100.

10.4.3 *One Component, with Slope-Type Background Correction:*

$$C, \% = \frac{[A_1 - A_2 + S(\lambda_2 - \lambda_1)]f}{a_1 b C_s} \times 100$$

where:

S = slope between wavelengths 1 and 2 for the background.

10.4.3.1 The background absorption is usually *not* linear between the analytical wavelength and the wavelength at which a simple subtractive background correction may be obtained. When it is possible to determine the slope between wavelengths 1 and 2 by observation of the samples that do not contain the absorbing material that is to be determined, this may be used as a correction for the background absorption.

10.4.4 *One Component, With Linear Background Correction:*

10.4.4.1 The equation for the general case is as follows:

$$C, \% = \frac{A_1 - \left[A_s + [A_2 - A_3] \times \dfrac{\lambda_3 - \lambda_1}{\lambda_3 - \lambda_2}\right] f}{abC_c} \times 100$$

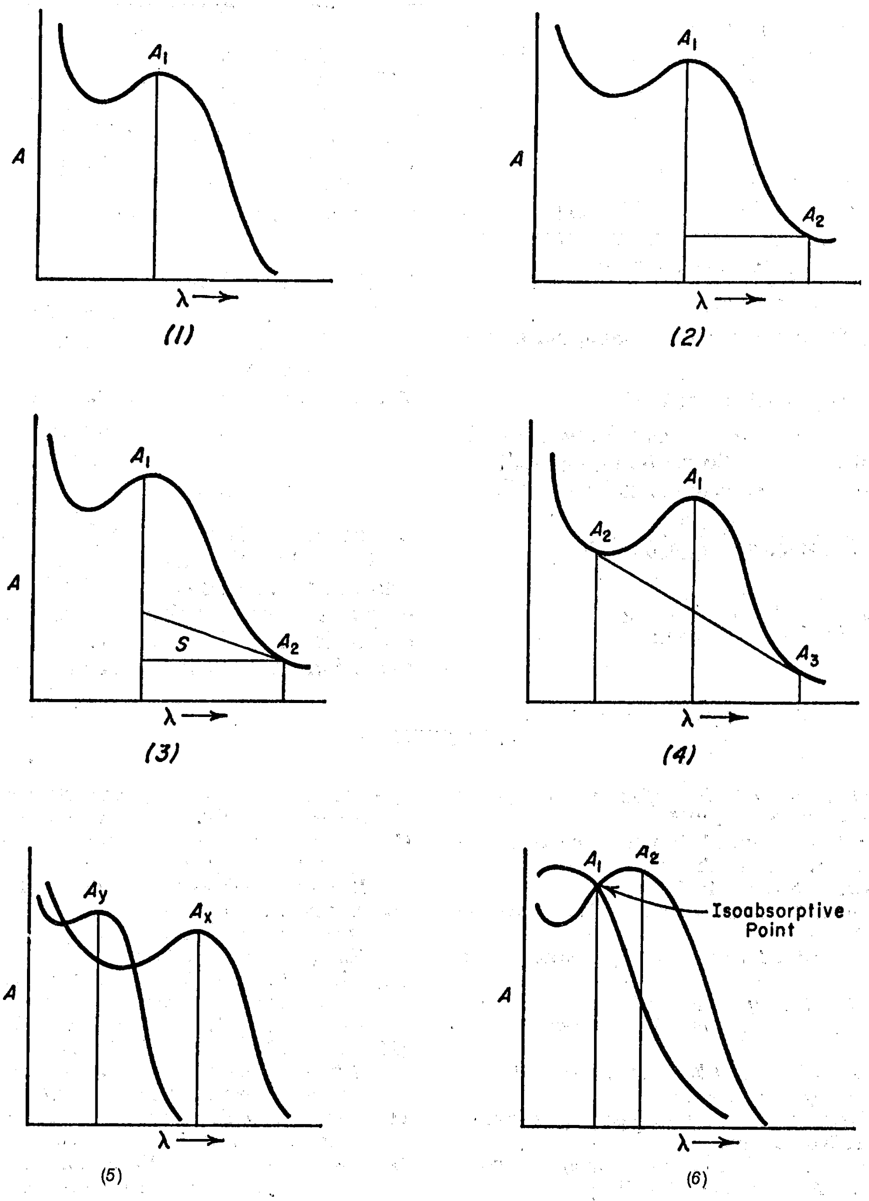
The absorptivity a is here the effective absorptivity as determined on a pure sample, using the corrections, and is somewhat lower than the true or absolute absorptivity.

10.4.4.2 This method is especially effective with materials that have sharp bands. Wavelengths 2 and 3 are selected to the long and short wavelength sides of the analytical wavelength 1, usually at absorbance minima.

10.4.4.3 For the special case where the wavelength for A_1 is exactly midway between the wavelengths for A_1 and A_3, the equation reduces to the following:

$$C. \% = f\left[\frac{A_1 - \dfrac{A_2 + A_3}{2}}{abC_s}\right] \times 100$$

10.4.5 *One Component, With Background Correction from Outside Data:*



(1) One Component, No Background Correction.
(3) One Component, with Slope-Type Background Correction.
(5) Two Components, with Overlapping Absorption for Only One Component.
(2) One Component, Simple Background Correction.
(4) One Component, with Linear Background Correction.
(6) Two Components, with Mutually Overlapping Absorption.

FIG. 1 Plot of Components

$$C, \% = \frac{(A \times X) \times f}{abC_s} \times 100$$

10.4.5.1 This is a general case in which some empirical correction may be derived from data other than spectrophotometric, and is applied as an effective absorbance which is subtracted from the observed. As an example, the concentration of a known interfering material may be determined by titration, and the absorbance due to this calculated, and then subtracted.

10.4.6 *Two Components, With No Overlapping Absorption*—Apply the method in 10.4.1 twice, at the two analytical wavelengths. This is an almost impossible case, except when the relative concentrations of the two components are such that the product of absorptivity and concentration of one component at a given wavelength is more than 100 times the product for the other component, allowing the latter to be neglected.

10.4.7 *Two Components, With Overlapping Absorption for Only One Component*—Determine the component with no interference (component x) at an analytical wavelength selected to allow no contribution from component y as follows:

$$C_x, \% = (Af/a_1bC_s) \times 100$$

10.4.7.1 Calculate the contribution of this component to the observed absorbance at the other analytical wavelength, where both components are absorbing, as follows:

$$A_2x = a_{2x}bc_x$$

10.4.7.2 Calculate the concentration of component y as follows:

$$C_y, \% = [(A_2 - A_{2x}) \times f]/a_2bC_s$$

10.4.8 *Two Components, with Mutually Overlapping Absorption*—Use the absorbance-ratio method (graphical) described in Ref **(10)** or by simultaneous equations as follows:

$$C_x, \% = \frac{[a_{x2}A_1 - a_{y1}A_2]f}{bC_s \times (a_{y2}a_{x1} - a_{y1}a_{x2})} \times 100$$

$$C_y, \% = \frac{[a_{x2}A_1 - a_{x1}A_2]f}{bC_s \times (a_{y1}a_{x2} - a_{y2}a_{x1})} \times 100$$

10.4.9 *Inverted Matrix Method, for Two or More Components, With Mutually Overlapping Absorption*—For information on the inverted matrix method, see 10.1 of Practices E 168 and Ref **(13)**.

10.5 *Computerized Calculations*—Newer instruments may perform automatically many of the calculations described in 10.4. The user should be aware of the algorithms used by the manufacturer. It is recommended that the user verify the reliability of computed results by periodically performing the calculations using the raw analytical data.

11. Presentation of Data

11.1 If absorption curves are to be presented with an analytical method, it is recommended that one of the following systems be used, with the wavelength (in nanometers) increasing linearly to the right:

$\log \epsilon$ or $\log a$ plotted against λ

A plotted against λ

$\epsilon \times 10^{-n}$ or a plotted against λ

where the symbols are as defined in Definitions and Symbols E 131. Marking the analytical wavelengths and absorptivity values on the curve is suggested for clarity, or a separate table of analytical wavelengths and absorptivities may be used. (These data are helpful for others who may wish to use the method in a somewhat modified form.)

REFERENCES

(1) Olsen, E. D., *Modern Optical Methods of Analysis*, (Chapters 1 through 3) McGraw-Hill, NY, NY, 1975.

(2) Ewing, P. J., Kolthoff, I. M., and Meehan, E. J., editors, *Treatise on Analytical Chemistry, Part I*, 7th edition, vol. 7, (Chapters 1 through 3), Interscience, NY, NY, 1981.

(3) Mellon, M. G., Editor, *Analytical Absorption Spectroscopy*, John Wiley & Sons, N. Y.

(4) Lothian, G. F., *Absorption Spectrophotometry*, Macmillan, NY, 2nd Ed., 1958.

(5) West, W., Editor, *Chemical Applications of Spectroscopy*, Vol IX or Weissberger series, *Technique of Organic Chemistry*, Interscience, NY, 1956.

(6) Friedel, R. A., and Orchin, M., *Ultraviolet Spectra of Aromatic Compounds*, John Wiley & Sons, NY, 1951.

(7) Gillam, A. E., and Stern, E. S., *Electronic Absorption Spectroscopy*, E. Arnold, London, 2nd Ed., 1957.

(8) Hershenson, H. M., *Ultraviolet and Visible Absorption Spectra: Index for 1930–1954*, Academic Press, NY, 1956.

(9) Hager, L. G. and Howell, J. A., Annual Reviews in *Analytical Chemistry*, *54*, 171R (1982) and 56 225R (1984).

(10) Abbott, R., "Ultraviolet Spectrophotometric Techniques," *Applied Spectroscopy*, Vol 4, No. 1, October 1948.

(11) Burke, R. W. and Mavrodineanu, R., *Accuracy in Analytical Spectrophotometry*, NBS Special Publication 260 – 81, National Bureau of Standards, Washington, DC, 1983.

(12) Klevens, H. B., and Platt, J. R., "Ultraviolet Transmission Limits of Some Liquids and Solids," *Journal of the American Chemical Society*, Vol 69, 1947, p. 3055.

(13) Hirt, R. C., King, F. T., and Schmitt, R. G., "Graphical Absorbance Ratio Method for Rapid Two-Component Spectrophotometric Analysis," *Analytical Chemistry*, Vol 26, 1954, p. 1270.

(14) Graff, M. M., O'Connor, R. T., and Skau, E. L., "Purification of Solvents for Absorption Spectroscopy," *Industrial and Engineering Chemistry*, Analytical Edition, IENAA, Vol 16, 1944, p. 556.

(15) Tunnicliff, D. D., "Solvents for Ultraviolet Spectrophotometry," *Talanta*, Vol 2, No. 4, 1959, p. 341.

(16) Crout, P. D., "A Short Method for Evaluating Determinants and Solvent Systems of Linear Equations with Real or Complex Coefficients," *Transactions*, Am. Inst. Electrical Engrs., Vol 60,1941, p. 1235.

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Standard Practice for
CONDUCTING SURVEILLANCE TESTS FOR LIGHT-WATER COOLED NUCLEAR POWER REACTOR VESSELS[1]

This standard is issued under the fixed designation E 185; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

$^{\epsilon}$ NOTE—Section 9.2.3 was corrected editorially and the designation date was changed July 1, 1982.

1. Scope

1.1 This practice covers procedures for monitoring the radiation-induced changes in the mechanical properties of ferritic materials in the beltline of light-water cooled nuclear power reactor vessels. This practice includes guidelines for designing a minimum surveillance program, selecting materials, and evaluating test results.

1.2 This practice was developed for all light-water cooled nuclear power reactor vessels for which the predicted maximum neutron fluence ($E > 1$ MeV) at the end of the design lifetime exceeds 1×10^{21} n/m^2 (1×10^{17} n/cm^2) at the inside surface of the reactor vessel.

2. Applicable Documents

2.1 *ASTM Standards:*

A 370 Methods and Definitions for Mechanical Testing of Steel Products[2]

E 8 Methods of Tension Testing of Metallic Materials[3]

E 21 Recommended Practice for Elevated Temperature Tension Tests of Metallic Materials[3]

E 23 Methods for Notched Bar Impact Testing of Metallic Materials[3]

E 208 Method for Conducting Drop-Weight Test to Determine Nil-Ductility Transition Temperature of Ferritic Steels[3]

E 482 Guide for Application of Neutron Transport Methods for Reactor Vessel Surveillance[4]

E 560 Recommended Practice for Extrapolating Reactor Vessel Surveillance Dosimetry Results[4]

2.2 *American Society of Mechanical Engineers Standard*: Boiler and Pressure Vessel Code, Sections III and XI[5]

3. Significance and Use

3.1 Predictions of neutron radiation effects on pressure vessel steels are considered in the design of light-water cooled nuclear power reactors. Changes in system operating parameters are made throughout the service life of the reactor vessel to account for radiation effects. Because of the variability in the behavior of reactor vessel steels, a surveillance program is warranted to monitor changes in the properties of actual vessel materials caused by long-term exposure to the neutron radiation and temperature environment of the given reactor vessel. This practice describes the criteria that should be considered in planning and implementing surveillance test programs and points out precautions that should be taken to ensure that: (*1*) capsule exposures can be related to beltline exposures, (*2*) materials selected for the surveillance program are samples of those materials most likely to limit the operation of the reactor vessel, and (*3*) the tests yield results useful for the evaluation of radiation effects on the reactor vessel.

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[1] This practice is under the jurisdiction of ASTM Committee E-10 on Nuclear Technology and Applications.

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[2] *Annual Book of ASTM Standards*, Vol 01.04.

[3] *Annual Book of ASTM Standards*, Vol 03.01.

[4] *Annual Book of ASTM Standards*, Vol 12.02.

[5] Available from the American Society of Automotive Engineers, 345 E. 47th St., New York, N. Y. 10017.

3.2 The design of a surveillance program for a given reactor vessel must consider the existing body of data on similar materials in addition to the specific materials used for that reactor vessel. The amount of such data and the similarity of exposure conditions and material characteristics will determine their applicability for predicting the radiation effects. As a large amount of pertinent data becomes available it may be possible to reduce the surveillance effort for selected reactors by integrating their surveillance programs.

4. Definitions

4.1 *adjusted reference temperature*—the reference temperature adjusted for irradiation effects by adding to RT_{NDT} the transition temperature shift (see 4.15).

4.2 *base metal (parent material)*—as-fabricated plate material or forging material other than a weldment or its corresponding heat-affected-zone (HAZ).

4.3 *beltline*—the irradiated region of the reactor vessel (shell material including weld regions and plates or forgings) that directly surrounds the effective height of the active core, and adjacent regions that are predicted to experience sufficient neutron damage to warrant consideration in the selection of surveillance material.

4.4 *EOL*—end-of-life; the design lifetime in terms of years; effective full power years; or neutron fluence.

4.5 *index temperature*—that temperature corresponding to a predetermined level of absorbed energy, lateral expansion, or fracture appearance obtained from the average (best fit) Charpy transition curve.

4.6 *fraction strength*—in a tensile test, the load at fracture divided by the initial cross-sectional area of the test specimen.

4.7 *fracture stress*—in a tensile test, the load at fracture divided by the cross-sectional area of the test specimen at time of fracture.

4.8 *heat-affected-zone* (HAZ)—plate material or forging material extending outward from, but not including, the weld fusion zone in which the microstructure of the base metal has been altered by the heat of the welding process.

4.9 *lead factor*—the ratio of the neutron flux density at the location of the specimens in a surveillance capsule to the neutron flux density at the reactor pressure vessel inside surface at the peak fluence location.

4.10 *neutron fluence*—the time integrated neutron flux density, expressed in neutrons per square metre or neutrons per square centimetre.

4.11 *neutron flux density*—a measure of the intensity of neutron radiation within a given range of neutron energies; the product of the neutron density and velocity, measured in neutrons per square metre-second or neutrons per square centimetre-second.

4.12 *neutron spectrum*—the distribution of neutrons by energy levels impinging on a surface, which can be calculated based on analysis of multiple neutron dosimeter measurements, on the assumption of a fission spectrum, or from a calculation of the neutron energy distribution.

4.13 *nil-ductility transition temperature* (T_{NDT})—the maximum temperature at which a standard drop weight specimen breaks when tested in accordance with Method E 208.

4.14 *reference temperature* (RT_{NDT})—See subarticle NB-2300 of the ASME Boiler and Pressure Vessel Code, Section III, "Nuclear Power Plant Components."

4.15 *transition temperature shift* (ΔRT_{NDT}) *or adjustment of reference temperature*—the difference in the 41-J (30-ft·lbf) index temperatures from the average Charpy curves measured before and after irradiation.

4.16 *transition region*—the region on the transition temperature curve in which toughness increases rapidly with rising temperature. In terms of fracture appearance, it is characterized by a rapid change from a primarily cleavage (crystalline) fracture mode to primarily shear (fibrous) fracture mode.

4.17 *Charpy transition curve*—a graphic presentation of Charpy data, including absorbed energy, lateral expansion, and fracture appearance, extending over a range including the lower shelf energy (< 5 % shear), transition region, and the upper shelf energy (> 95 % shear).

4.18 *upper shelf energy level*—the average energy value for all Charpy specimens (normally three) whose test temperature is above the upper end of the transition region. For specimens tested in sets of three at each test temperature, the set having the highest average may be regarded as defining the upper shelf energy.

5. Test Materials

5.1 *Materials Selection*:

5.1.1 Surveillance test materials shall be prepared from samples taken from the actual materials used in fabricating the beltline of the reactor vessel. These surveillance test materials shall include one heat of the base metal, one butt weld, and one weld heat-affected-zone (HAZ). The base metal, weld metal, and HAZ (Note 1) materials included in the program shall be those predicted to be most limiting, with regard to setting pressure-temperature limits, for operation of the reactor to compensate for radiation effects during its lifetime (Note 2). The beltline materials shall be evaluated on the basis of initial reference temperature (RT_{NDT}), the predicted changes in the initial properties as a function of chemical composition (for example, copper (Cu) and phosphorus (P)) (Note 3), and the neutron fluence during reactor operation.

NOTE 1—The base metal for the weld heat-affected-zone (HAZ) to be monitored shall correspond to one of the base metals selected for the surveillance program.

NOTE 2—The data used for the selection of surveillance test materials shall be that obtained in accordance with ASME Code Section III requirements.

NOTE 3—Other residual/alloy elements such as Ni, Si, Mn, Mo, Cr, C, S, and V may contribute to overall radiation behavior of ferritic materials.

5.1.2 The base metal and the weld with the highest adjusted reference temperature at end-of-life shall be selected for the surveillance program. If the Charpy upper shelf energy of any of the beltline materials is predicted to drop to a marginal level (currently considered to be 68 J (50 ft·lbf) at the quarter thickness (¼ T) location) during the operating lifetime of the vessel, provisions shall be made to also include that material in the surveillance program, preferably in the form of fracture toughness specimens. These additional specimens may be substituted in part for specimens of the material least likely to be limiting.

5.1.3 The adjusted reference temperature of the materials in the reactor vessel beltline shall be determined by adding the appropriate values of transition temperature shift to the reference temperature of the unirradiated material. The transition temperature shift and Charpy upper shelf energy drop can be determined from relationships of fluence and chemical composition.

5.4 *Material Sampling*—A minimum test program shall consist of the material selected in 5.1, taken from the following locations: (*1*) base metal from one plate or forging used in the beltline, (*2*) weld metal made with the same heat of weld wire and lot of flux and by the same welding practice as that used for the selected beltline weld, and (*3*) the heat-affected-zone associated with the base metal noted above.

5.5 *Archive Materials*—Representative test stock to fill at least two additional capsules with test specimens of the base metal, weld, and heat-affected-zone materials used in the program shall be retained with full documentation and identification. It is recommended that this test stock be in the form of full-thickness sections of the original materials (plates, forgings, and welds).

5.6 *Fabrication History*—The fabrication history (austenitizing, quench and tempering, and post-weld heat treatment) of the test materials shall be fully representative of the fabrication history of the materials in the beltline of the reactor vessel and shall be recorded.

5.7 *Chemical Analysis Requirements*—The chemical analysis required by the appropriate product specifications for the surveillance test materials (base metal and as-deposited weld metal) shall be recorded and shall include phosphorus (P), sulfur (S), copper (Cu), vanadium (V), and nickel (Ni), as well as all other alloying and residual elements commonly analyzed for in low-alloy steel products. The product analysis shall be verified by analyzing a minimum of three test specimens randomly selected from both the base metal and the as-deposited weld metal.

6. Test Specimens

6.1 *Type of Specimens*—Charpy V-notch impact specimens corresponding to the Type A specimen described in Methods A 370 and E 23 shall be used. The gage section of irradiated and unirradiated tension specimens shall be of the same size and shape. Tension specimens of the type, size, and shape described in Methods A 370 and E 8 are recommended. Additional fracture toughness test specimens shall be employed to supplement the information from the Charpy V-notch specimens if the surveillance materials are predicted to exhibit marginal properties.

6.2 *Specimen Orientation and Location*—Tension and Charpy specimens representing the base metal and the weld heat-affected-zone shall be removed from about the quarter-thickness (¼ T) locations. Material from the mid-thickness of the plates shall not be used for test specimens. Specimens representing weld metal may be removed at all locations throughout the thickness with the exception of locations within 12.7 mm (½ in.) of the root or surfaces of the welds. The tension and Charpy specimens from base metal shall be oriented so that the major axis of the specimen is parallel to the surface and normal to the principal rolling direction for plates, or normal to the major working direction for forgings as described in Section III of the ASME Code. The axis of the notch of the Charpy specimen for base metal and weld metal shall be oriented perpendicular to the surface of the material; for the HAZ specimens, the axis of the notch shall be as close to perpendicular to the surface as possible so long as the entire length of the notch is located within the HAZ. The recommended orientation of the weld metal and HAZ specimens is shown in Fig. 1. Weld metal tension specimens may be oriented in the same direction as the Charpy specimens provided that the gage length consists entirely of weld metal. The weldment shall be etched to define the weld heat-affected-zone. The notch roots in the HAZ Charpy specimens shall be at a standard distance of approximately 0.8 mm (1⁄32 in.) from the weld fusion line. The orientation of the HAZ samples with respect to the major working direction of the parent material shall be recorded.

6.3 *Quantities of specimens*:

6.3.1 *Unirradiated Baseline Specimens*—It is recommended that 18 Charpy specimens be provided, of which a minimum of 15 specimens shall be tested to establish a full transition temperature curve for each material (base metal, HAZ, weld metal). The three remaining Charpy specimens should be reserved to provide supplemental data in instances such as excessive data scatter. At least three tension test specimens shall be provided to establish the unirradiated tensile properties for base metal and weld metal.

6.3.2 *Irradiated Specimens*—The minimum number of test specimens for each irradiation exposure set (capsule) shall be as follows:

Material	Charpy	Tension
Base metal	12	3
Weld metal	12	3
HAZ	12	—

It is suggested that a greater quantity of the above specimens be included in the irradiation capsules whenever possible.

7. Irradiation Requirements

7.1 *Encapsulation of Specimens*—Specimens should be maintained in an inert environment within a corrosion-resistant capsule to prevent deterioration of the surface of the specimens during radiation exposure. Care should be exercised in the design of the capsule to ensure that the temperature history of the specimens duplicates, as closely as possible, the temperature experienced by the reactor vessel. Surveillance capsules should be sufficiently rigid to prevent mechanical damage to the specimens and monitors during irradiation. The design of the capsule and capsule attachments shall also permit insertion of replacement capsules into the reactor vessel if required at a later time in the lifetime of the vessel. The design of the capsule holder and the means of attachment shall (*1*) preclude structural material degradation by the attachment welds, (*2*) avoid interference with inservice inspection required by ASME Code Section XI, and (*3*) ensure the integrity of the capsule holder during the service life of the reactor vessel.

7.2 *Location of Capsules*:

7.2.1 *Vessel Wall Capsules (Required)*—Surveillance capsules shall be located within the reactor vessel so that the specimen irradiation history duplicates as closely as possible, within the physical constraints of the system, neutron spectrum, temperature history, and maximum neutron fluence experienced by the reactor vessel. It is recommended that the surveillance capsule lead factors (the ratio of the instantaneous neutron flux density at the specimen location to the maximum calculated neutron flux density at the inside surface of the reactor vessel wall) be in the range of one to three. This range of lead factors will minimize the calculational uncertainties in extrapolating the surveillance measurements from the specimens to the reactor vessel wall and maximize the ability of the program to monitor material property changes throughout the life of the reactor vessel.

7.2.2 *Accelerated Irradiation Capsules (Optional)*—Additional test specimens may be positioned at locations closer to the core than those described in 7.2.1 for accelerated irradiation.

7.3 *Neutron Dosimeters:*

7.3.1 *Selection of Neutron Dosimeters*—Neutron dosimeters for the surveillance capsules shall be selected according to Guide E 482. The group of monitors selected shall be capable of providing fast neutron fluence, fast neutron spectrum, and thermal neutron flux density information. Dosimeters shall be included in every capsule.

7.3.2 *Location of Neutron Dosimeters*—Dosimeters shall be located within the vessel wall capsules (7.2.1) and the accelerated capsules (7.2.2) if used.

7.3.3 Separate dosimeter capsules should also be used to monitor radiation conditions independent of the specimen capsules if it is expected that the withdrawal schedule will otherwise result in saturation of the dosimeter activities.

7.4 *Correlation Monitors (Optional):*

7.4.1 *Selection of Correlation Monitor Materials*—Correlation monitors[6] have been found to be useful as an independent check on the measurement of irradiation conditions for the surveillance materials. Correlation monitor materials should be well characterized in terms of irradiation behavior (transition temperature shift). The magnitude of the transition temperature shift for this material should be measureable for the selected exposures.

7.5 *Temperature Monitors:*

7.5.1 *Selection of Temperature Monitors*—Major differences between specimen irradiation temperature and design temperature, occuring as a result of capsule design features, variation in reactor coolant temperature, or both, can affect the extent of radiation induced property changes in the surveillance materials. Since it is not practical to instrument the surveillance capsules, low melting point elements or eutectic alloys are used instead as monitors to detect significant variations in exposure temperature. These monitors are used in surveillance programs to provide evidence of the maximum exposure temperature of the specimens. The monitor materials should be selected to indicate unforeseen capsule temperatures.

7.5.2 *Location of Temperature Monitors*—One set of temperature monitors shall be located within the capsule where the specimen temperature is predicted to be the maximum. Additional sets of temperature monitors may be placed at other locations within the capsule to characterize the temperature profile.

7.6 *Number of Surveillance Capsules and Withdrawal Schedule:*

7.6.1 *Number of Capsules*—A sufficient number of surveillance capsules shall be provided to monitor the effects of neutron irradiation on the reactor vessel throughout its operating lifetime. The basis for the number of capsules to be installed at beginning of life is the predicted transition temperature shift, as shown in Table 1. The decrease in the upper shelf energy may also be a factor (see 5.1, 5.2, and 5.3). Additional capsules may be needed to monitor the effect of a major core change or annealing of the vessel, or to provide supplemental toughness data for evaluating a flaw in the beltline. It is recommended that full-thickness sections of material be kept instead of loaded capsules, because the preferred type and size of test specimen may change in the intervening years. The archive material required in 5.5 is to be used for the additional capsules.

7.6.2 *Withdrawal Schedule*—The capsule withdrawal schedule should permit monitoring of long-time effects which are difficult to achieve in test reactors. Table 1 lists the recommended number of capsules and the withdrawal schedule for three ranges of predicted transition temperature shift. The withdrawal schedule is in terms of effective full-power years (EFPY) of the vessel with a design life of 32 EFPY. Other factors that must be considered in establishing the withdrawal schedule are presented in Table 1. The first capsule is scheduled for withdrawal early in the vessel life to verify the initial predictions of the surveillance material response to the actual radiation environment. It is removed when the predicted shift exceeds the expected scatter by sufficient margin to be measureable. Normally, the capsule

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[6] Information regarding the availability of correlation monitors can be obtained from ASTM Committee E-10. See also ASTM DS54, July 1974.

with the highest lead factor is withdrawn first. Early withdrawal will permit verification of the adequacy and conservatism of the reactor vessel pressure/temperature operational limits. The withdrawal schedule of the final two capsules is adjusted by the lead factor so the exposure of the second to last capsule does not exceed the peak end-of-life (EOL) fluence on the inside surface of the vessel, and so the exposure of the final capsule does not exceed twice the EOL vessel inside surface peak fluence. The decision on when to test specimens from the final capsule need not be made until the results from the preceding capsules are known.

7.6.3 *Implementation of Table 1:*

7.6.3.1 Estimate the peak vessel inside surface fluence at EOL and the corresponding transition temperature shift. This identifies the number of capsules required.

7.6.3.2 Estimate the lead factor for each surveillance capsule relative to the peak beltline fluence.

7.6.3.3 Calculate the number of EFPY for the capsule to reach the peak vessel EOL fluence at the inside surface and ¼ T locations. These are used to establish the withdrawal schedule for all but the first capsule.

7.6.3.4 Schedule the capsule withdrawals at the nearest vessel refueling date.

8. Measurement of Radiation Exposure Conditions

8.1 *Temperature Environment*—The maximum exposure temperature of the surveillance capsule materials shall be determined. If a discrepancy (> 14°C or 25°F) occurs between the observed and the expected capsule exposure temperatures, an analysis of the operating conditions shall be conducted to determine the magnitude and duration of these differences.

8.2 *Neutron Irradiation Environment:*

8.2.1 The neutron flux density, neutron energy spectrum, and neutron fluence of the surveillance specimens and the corresponding maximum values for the reactor vessel shall be determined in accordance with the guidelines in Guide E 482 and Recommended Practice E 560.

8.2.2 The specific method of determination shall be documented.

8.2.3 Neutron flux density and fluence values ($E > 0.1$ and 1 MeV) shall be determined and recorded using both a calculated spectrum and an assumed fission spectrum.

9. Measurement of Mechanical Properties

9.1 *Tension Tests:*

9.1.1 *Method*—Tension testing shall be conducted in accordance with Method E 8 and Recommended Practice E 21.

9.1.2 *Test Temperature:*

9.1.2.1 *Unirradiated*—The test temperatures for each material shall include room temperature, service temperature, and one intermediate temperature to define the strength *versus* temperature relationship.

9.1.2.2 *Irradiated*—One specimen from each material shall be tested at a temperature in the vicinity of the upper end of the Charpy energy transition region. The remaining specimens from each material shall be tested at the service temperature and the midtransition temperature.

9.1.3 *Measurements*—For both unirradiated and irradiated materials, determine yield strength, tensile strength, fracture load, fracture strength, fracture stress, total and uniform elongation, and reduction of area.

9.2 *Charpy Tests:*

9.2.1 *Method*—Charpy tests shall be conducted in accordance with Methods E 23 and A 370.

9.2.2 *Test Temperature:*

9.2.2.1 *Unirradiated*—Test temperatures for each material shall be selected to establish a full transition temperature curve. One specimen per test temperature may be used to define the overall shape of the curve. Additional tests should be performed in the region where the measurements described in 9.2.3 are made.

9.2.2.2 *Irradiated*—Specimens for each material will be tested at temperatures selected to define the full energy transition curve. Particular emphasis should be placed on defining the 41-J (30-ft·lbf), 68-J (50-ft·lbf), and 0.89-mm (35-mil) lateral expansion index temperatures and the upper shelf energy.

9.2.3 *Measurements*—For each test specimen, measure the impact energy, lateral expansion, and percent shear fracture appearance. From the unirradiated and irradiated transition temperature curves determine the 41-J (30-ft·lbf), 68-J (50-ft·lbf), and 0.89-mm (35-mil) lateral expansion index temperatures and the upper shelf energy. The index temperatures and the upper shelf energy shall be determined from the average curves.

9.2.3.1 Obtain from the material qualification test report the initial reference temperature (RT_{NDT}) as defined in the ASME Code, Section III, Subarticle NB 2300 for unirradiated materials.

9.3 *Hardness Tests (Optional)*—Hardness tests may be performed on unirradiated and irradiated Charpy specimens. The measurements shall be taken in areas away from the fracture zone or the edges of the specimens. The tests shall be conducted in accordance with Methods A 370.

9.4 *Supplemental Tests (Optional)*—If supplemental fracture toughness tests are conducted (in addition to tests conducted on tension and Charpy specimens as described in 6.1) the test procedures shall be documented.

9.5 *Calibration of Equipment*—Procedures shall be employed assuring that tools, gages, recording instruments, and other measuring and testing devices are calibrated and properly adjusted periodically to maintain accuracy within necessary limits.[7] Whenever possible calibration shall be conducted with standards traceable to the National Bureau of Standards. Calibration status shall be maintained in records traceable to the equipment.

10. Determination of Irradiation Effects

10.1 *Tension Test Data:*

10.1.1 Determine the amount of radiation strengthening by comparing unirradiated test results with irradiated test results at the temperatures specified in 9.1.2.

10.1.2 The tensile strength data can be verified using the results from the hardness test (optional) described in 9.3.

10.2 *Charpy Test Data:*

10.2.1 Determine the radiation induced transition temperature shifts by measuring the difference in the 41-J (30-ft·lbf), 68-J (50-ft·lbf), and 0.89-mm (35-mil) lateral expansion index temperatures before and after irradiation. The index temperatures shall be obtained from the average curves.

10.2.2 Determine the adjusted reference temperature by adding the shift corresponding to the 41-J (30-ft·lbf) index determined in 10.2.1 to the initial reference temperature obtained in 9.2.3.1.

10.2.3 Determine the radiation induced change in the upper shelf energy (USE) from measurements made before and after irradiation using average value curves.

10.2.4 *(Optional)*—Determine the radiation induced change in temperature corresponding to 50 % of the upper shelf energy before and after irradiation from average value curves.

10.3 *Supplemental Test Data (Optional)*—If additional, supplemental tests are performed (9.4), the data shall be recorded to supplement the information from the tensile and Charpy tests.

10.4 *Retention of Test Specimens*—It is recommended that all broken test specimens be retained until released by the owner in the event that additional analyses are required to explain anomalous results.

11. Report

11.1 The following information shall be provided. This report shall consist of the following elements. Where applicable, both SI units and conventional units shall be reported.

11.2 *Surveillance Program Description*—Description of the reactor vessel including the following:

11.2.1 Location of the surveillance capsules with respect to the reactor vessel, reactor vessel internals, and the reactor core.

11.2.2 Location in the vessel of the plates or forgings and the welds.

11.2.3 Location(s) of the peak vessel fluence.

11.2.4 Lead factors between the specimen fluence and the peak vessel fluence at the I.D. and the ¼ T locations.

11.2.5 *Surveillance Material Selection:*

11.2.5.1 Description of all beltline materials including chemical analysis, fabrication history, Charpy data, tensile data, drop-weight data and initial RT_{NDT}.

11.2.5.2 Describe the basis for selection of surveillance materials.

11.3 *Surveillance Material Characterization:*

11.3.1 Description of the surveillance material including fabrication history, material source (heat or lot), and any differences between the surveillance material history and that of the reactor vessel material history.

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[7] Standardized specimens for certification of Charpy impact machines are available from the Army Materials and Mechanics Research Center, Watertown, Mass. 02172, Attn: DRXMR-MQ.

11.3.2 Location and orientation of the test specimens in the parent material.

11.3.3 *Test Specimen Design*:

11.3.3.1 Description of the test specimens (tension, Charpy, and any other types of specimens used), neutron dosimeters, and temperature monitors.

11.3.3.2 Certification of calibration of all equipment and instruments used in conducting the tests.

11.4 *Test Results*:

11.4.1 *Tension Tests*:

11.4.1.1 Trade name and model of the testing machine, gripping devices, extensometer, and recording devices used in the test.

11.4.1.2 Speed of testing and method of measuring the controlling testing speed.

11.4.1.3 Complete stress-strain curve (if a group of specimens exhibits similar stress-strain curves, a typical curve may be reported for the group).

11.4.1.4 Test data from each specimen as follows:

(*1*) Test temperature;

(*2*) Yield strength or yield point and method of measurement;

(*3*) Tensile strength;

(*4*) Fracture load, fracture strength, and fracture stress;

(*5*) Uniform elongation and method of measurement;

(*6*) Total elongation;

(*7*) Reduction of area; and

(*8*) Specimen identification.

11.4.2 *Charpy Tests*:

11.4.2.1 Trade name and model of the testing machine, available hammer energy capacity and striking velocity, temperature conditioning and measuring devices, and a description of the procedure used in the inspection and calibration of the testing machine.

11.4.2.2 Test data from each specimen as follows:

(*1*) Temperature of test;

(*2*) Energy absorbed by the specimen in breaking, reported in joules (and foot-pound-force);

(*3*) Fracture appearance;

(*4*) Lateral expansion; and

(*5*) Specimen identification.

11.4.2.3 Test data for each material as follows:

(*1*) Charpy 41-J (30-ft·lbf), 68-J (50-ft·lbf), and 0.89-mm (35-mil) lateral expansion index temperature of unirradiated material and of each set of irradiated specimens, along with the corresponding temperature increases for these specimens;

(*2*) Upper shelf energy (USE) absorbed before and after irradiation;

(*3*) Initial reference temperature; and

(*4*) Adjusted reference temperature.

11.4.3 *Hardness Tests (Optional)*:

11.4.3.1 Trade name and model of the testing machine.

11.4.3.2 Hardness data.

11.4.4 *Other Fracture Toughness Tests*:

11.4.4.1 If additional tests are performed, the test data shall be reported together with the procedures used for conducting the tests and analysis of the data.

11.4.5 *Temperature and Neutron Radiation Environment Measurements*:

11.4.5.1 Temperature monitor results and an estimate of maximum capsule exposure temperature.

11.4.5.2 Neutron dosimeter measurements, analysis techniques, and calculated results including the following:

(*1*) Neutron flux density, neutron energy spectrum, and neutron fluence in terms of neutrons per square metre and neutrons per square centimetre (> 0.1 and 1 MeV) for the surveillance specimens using both calculated spectrum and assumed fission spectrum assumptions.

(*2*) Description of the methods used to verify the procedures including calibrations, cross sections, and other pertinent nuclear data.

11.5 *Application of Test Results*:

11.5.1 Extrapolation of the neutron flux and fluence results to the surface and ¼ T locations of the reactor vessel at the peak fluence location.

11.5.2 Comparison of fluence determined from the dosimetry analysis with original predicted values.

11.5.3 Extrapolation of fracture toughness properties to the surface and ¼ T locations of the reactor vessel at the peak fluence location.

11.6 *Deviations*—Deviations or anomalies in procedure from this practice shall be identified and described fully in the report.

TABLE 1 Minimum Recommended Number of Surveillance Capsules and Their Withdrawal Schedule (Schedule in Terms of Effective Full-Power Years of the Reactor Vessel)

	Predicted Transition Temperature Shift at Vessel Inside Surface		
	≤ 56°C (≤ 100°F)	> 56°C (> 100°F) ≤ 111°C (≤ 200°F)	> 111°C (> 200°F)
Minimun Number of Capsules	3	4	5
Withdrawal Sequence:			
First	6[A]	3[A]	1.5[A]
Second	15[B]	6[C]	3[D]
Third	EOL[E]	15[B]	6[C]
Fourth		EOL[E]	15[B]
Fifth			EOL[E]

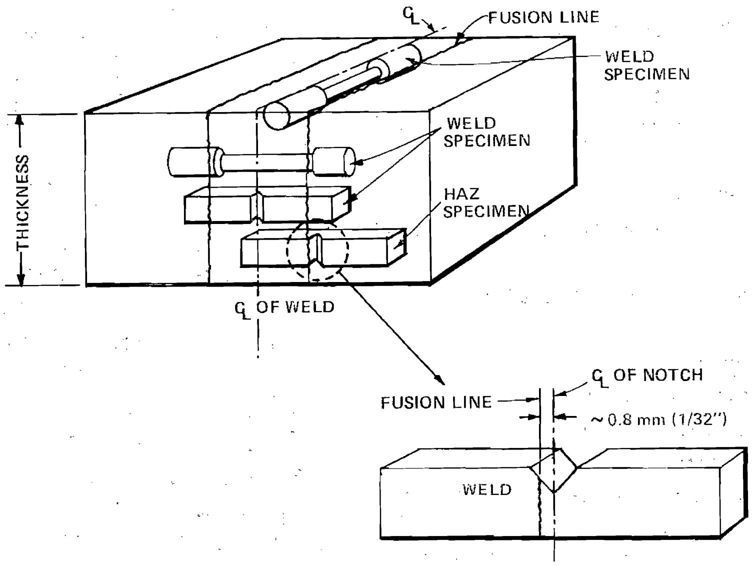
[A] Or at the time when the accumulated neutron fluence of the capsule exceeds 5×10^{22} n/m^2 (5×10^{18} n/cm^2), or at the time when the highest predicted ΔRT_{NDT} of all encapsulated materials is approximately 28°C (50°F), whichever comes first.

[B] Or at the time when the accumulated neutron fluence of the capsule corresponds to the approximate EOL fluence at the reactor vessel inner wall location, whichever comes first.

[C] Or at the time when the accumulated neutron fluence of the capsule corresponds to the approximate EOL fluence at the reactor vessel ¼ T location, whichever comes first.

[D] Or at the time when the accumulated neutron fluence of the capsule corresponds to a value midway between that of the first and third capsules.

[E] Not less than once or greater than twice the peak EOL vessel fluence. This may be modified on the basis of previous tests. This capsule may be held without testing following withdrawal.



(NOT DRAWN TO SCALE)

FIG. 1 Location of Test Specimens Within Weld and Heat-Affected-Zone (HAZ) Test Material.

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Standard Practice for Packed Column Gas Chromatography[1]

This standard is issued under the fixed designation E 260; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice is intended to serve as a general guide to the application of gas chromatography (GC) with packed columns for the separation and analysis of vaporizable or gaseous organic and inorganic mixtures and as a reference for the writing and reporting of GC methods.

NOTE 1—This practice excludes any form of gas chromatography associated with open tubular (capillary) columns.

1.2 *This standard does not purport to address all the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 7 and 9.1.3.

2. Referenced Documents

2.1 *ASTM Standards:*

E 355 Practice for Gas Chromatography Terms and Relationships[2]

E 516 Practice for Testing Thermal Conductivity Detectors Used in Gas Chromatography[2]

E 594 Practice for Testing Flame Ionization Detectors Used in Gas Chromatography[2]

E 697 Practice for Use of Electron Capture Detectors in Gas Chromatography[2]

E 840 Practice for Using Flame Photometric Detectors in Gas Chromatography[2]

E 1140 Practice for Testing Nitrogen/Phosphorus Thermionic Ionization Detectors for Use in Gas Chromatography[2]

2.2 *CGA Publications:*

CGA P-1 Safe Handling of Compressed Gases in Containers[3]

CGA G-5.4 Standard for Hydrogen Piping Systems at Consumer Locations[3]

CGA P-9 The Inert Gases: Argon, Nitrogen and Helium[3]

CGA V-7 Standard Method of Determining Cylinder Valve Outlet Connections for Industrial Gas Mixtures[3]

CGA P-12 Safe Handling of Cryogenic Liquids[3]

HB-3 Handbook of Compressed Gases[3]

3. Terminology

3.1 Terms and relations are defined in Practice E 355 and references therein.

4. Summary of Practice

4.1 A block diagram of the basic apparatus needed for a gas chromatographic system is as shown in Fig. 1. An inert, pressure or flow-controlled carrier gas flowing at a measured rate passes to the injection port or gas sample valve. A sample is introduced into the injection port, where it is vaporized, or if gaseous, into a gas sample valve, and then swept into and through the column by the carrier gas. Passage through the column separates the sample into its components. The effluent from the column passes to a detector where the response of sample components is measured as they emerge from the column. The detector electrical output is relative to the concentration of each resolved component and is transmitted to a recorder, or electronic data processing system, or both, to produce a record of the separation, or chromatogram, from which detailed analysis can be obtained. The detector effluent must be vented to a hood if the effluent contains toxic substances.

4.2 Gas chromatography is essentially a physical separation technique. The separation is obtained when the sample mixture in the vapor phase passes through a column containing a stationary phase possessing special adsorptive properties. The degree of separation depends upon the differences in the distribution of volatile compounds, organic or inorganic, between a gaseous mobile phase and a selected stationary phase that is contained in a tube or GC column. In gas-liquid chromatography (GLC), the stationary phase is a nonvolatile liquid or gum coated as a thin film on a finely-divided, inert support of a relatively large surface area, and the distribution is based on partition. The liquid phase should not react with, and should have different partition coefficients for, the various components in the sample. In gas-solid chromatography (GSC), the stationary phase is a finely divided solid adsorbent (see 4.4).

4.2.1 After separation in the analytical column, the components are detected, and the detector signal is related to the concentration of the volatile components. Tentative identifications can be made by comparison with the retention times of known standards under the same conditions, either on a single column or preferably by injecting the sample onto two columns of different selectivity. Ancillary techniques, such as mass spectrometry or infrared spectrophotometry, are generally necessary for positive identification of components in samples.

4.2.2 Prior to performing a GC analysis, the following parameters must be considered:

4.2.2.1 Sample preparation.

4.2.2.2 Stationary phase and loading on support.

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[1] This practice is under the jurisdiction of ASTM Committee E-19 on Chromatography and is the direct responsibility of Subcommittee E19.03 on Methods and Specifications, Gas Chromatography.

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[2] *Annual Book of ASTM Standards*, Vol 14.02.

[3] Available from Compressed Gas Association, Inc., 1725 Jefferson Davis Highway, Arlington, VA 22202-4100.

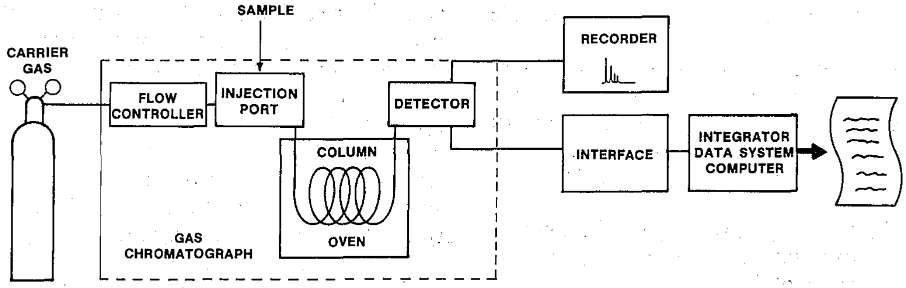

FIG. 1 Block Diagram of a Basic Gas Chromatographic System

4.2.2.3 Column material required.

4.2.2.4 Solid support and mesh size.

4.2.2.5 Column length and diameter.

4.2.2.6 Instrument and detector type that will be needed.

4.2.2.7 Injector, column oven, and detector temperatures required for analysis.

4.2.2.8 Injection techniques, such as flash volatilization, on-column technique, purge and trap, pyrolysis, etc.

4.2.2.9 Carrier gas and flow rate.

4.2.2.10 Data handling and presentation.

4.3 In gas-liquid chromatography, the degree of separation possible between any two compounds (solutes), is determined by the ratio of their partition coefficients and the separation efficiency. The partition coefficient, *K*, is the ratio of the solute concentration in the liquid phase to the solute concentration in the vapor phase at equilibrium conditions. The partition coefficient is affected by temperature and the chemical nature of the solute (sample) and solvent (stationary phase).

4.4 Another mechanism for separation is gas-solid chromatography. With this technique there is no liquid phase, only a porous polymer, molecular sieve, or solid adsorbent. Partition is accomplished by distribution between the gas phase and the solid phase.

4.5 After the sample is resolved into individual components by the chromatographic column, the concentration or mass flow of each component in the carrier gas can be measured by an appropriate detector which sends an electrical signal to a recording potentiometer or other readout device. The curve obtained by plotting detector response against time is referred to as a chromatogram. For flame ionization and thermal conductivity detectors, either the peak areas or the peak heights are proportional to the concentration of the components in the sample within the linear range of the detector system. However, response fractors are not necessarily the same for all compounds, and linearity of detector response may depend on operating conditions. (Testing of detector performance is discussed in ASTM Standard Practices for the appropriate detector, see 2.1).

4.6 Components in a mixture may be tentatively identified by retention time. Ideally, each substance has a unique retention time in the chromatogram for a specific set of operating conditions. However, caution is required because the GC separation may be incomplete and a single peak may represent more than one compound. This is especially true of unknown mixtures and complex mixtures because of the very large number of possible compounds in existence and the finite number of peaks that a chromatograph might resolve. Additional characterization data may be provided by ancillary techniques, such as spectrometry.

5. Significance and Use

5.1 This practice describes a procedure for packed-column gas chromatography. It provides general comments, recommended techniques, and precautions. A recommended form for reporting GC methods is given in Section 14.

6. Apparatus

6.1 *Carrier Gas System*—Common carrier gases are helium and nitrogen. Paragraph 7.6 provides more details on carrier gases. Means must be provided to measure and control the flow rate of the carrier gas. Any flow or pressure control and measurement combination may be used that will give an accurately known and reproducible flow rate over the desired range.

6.1.1 The main gas supply is regulated with a two-stage regulator which must have a stainless steel diaphragm. Rubber or plastic diaphragms permit oxygen or water to diffuse into the carrier gas. In addition, instruments will have a flow controller between the pressure regulator and column inlet to maintain a constant flow during temperature programming. Copper or stainless steel carrier gas lines, not plastic tubing, should be used to avoid diffusion of oxygen (air) into the carrier gas. When using the thermal conductivity detector, variations in the flow will change retention and response. The carrier gas line pressure must be higher than that required to maintain the column flow at the upper temperature limit for the flow controller to operate properly. A pressure of 40 to 60 psi is usually sufficient.

6.2 *Column Temperature Control*—Precise column temperature control is mandatory if reproducible analyses are to be obtained. Temperature control must be within 0.1°C if retention times are to be compared with another instrument.

6.2.1 *Air Bath*—The thermostated forced-air bath is generally accepted as the best practical method of temperature regulation for most applications. Temperatures can be controlled by regulators or proportionally controlled heaters using a thermocouple or platinum-resistance thermometer as a sensing element. The advantage of a forced-air bath is the speed of temperature equilibration. Air bath ovens are readily adaptable to temperature programming and are capable of operating over a range of 35 to 450°C. This range can be extended down to −100°C by using cryogenic equipment.

6.2.2 *Other Devices*—Liquid baths, drying ovens, incubators, or vapor jacket enclosures are less stable, less convenient means of providing a source of heat to maintain or raise the temperature of a chromatographic column. These devices are not recommended for precision chromatographic applications.

6.3 *The Injection Port*—The purpose of the injection port is to introduce the sample into the gas chromatographic column by instantaneous volatilization following injection into the gas chromatographic system. Two sample inlet types are in common use in gas chromatography: the flash vaporization and the on-column injection inlets.

6.3.1 The temperature of the flash vaporization inlet should be above the boiling points of the sample components and is limited by the amount of septum bleed generated and the temperature stability of sample components. It should be set at that temperature above which no improvement in peak shape occurs but should be determined by the nature of the sample and the volume injected, not by the temperature of the column. If the inlet temperature is too low, broad peak with a slowly rising front edge will result from slow vaporization of the sample. If the temperature is set far above what is necessary to produce fast vaporization, thermal decomposition of the sample, decreased septum life, and ghost peaks due to septum bleed may be observed. Generally, a good guideline is to maintain the inlet temperature 25 to 30°C higher than the highest boiling point of any sample component.

6.3.2 A glass liner placed inside the injection port will eliminate sample contact with hot metal inner walls of the inlet, which can catalyze thermal decompositions. Any debris left in the liner, especially from biological samples, can be a source of excessive sample adsorption. If a liner is used, the debris can easily be removed by replacing the liner. Deactivation of the glass liner by treatment with dimethyldichlorosilane may be necessary for some compounds.

6.3.3 With on-column injection technique, the sample is deposited in the liquid state directly on the column packing. The sample must be small enough to preclude flooding of the column, with possible detrimental effects to peak shape and column life. Ideally, the on-column inlet is a part of the column, so its temperature may be controlled as the column temperature is controlled. In practice, because an on-column inlet usually has a somewhat higher thermal mass than an equivalent sector of the rest of the column, the inlet must be heated somewhat above the maximum analysis temperature of the column oven. The criteria of good peak shape and quantitation should be used to determine the maximum required temperature for the inlet. One should consider the temperature limit of the column packing when heating the injection inlet and detector. With some samples, a nonheated injection port is adequate, especially with temperature-programmed operation.

6.3.4 *Injection Port Septum:*

6.3.4.1 The septum is a disc, usually made of silicone rubber, which seals one end of the injection port. It is important to change the septum frequently after two to three dozen injections, or preferably at the end of the working day. The best technique is to change the septum when the column is relatively cool (below 50°C) to avoid contact of stationary phase in a hot column with air (danger of oxidation). After the septum is changed, return the inlet temperature to that which was originally set. The inlet temperature should be the optimum for the particular analysis, as well as within the recommended operating temperature of the septum. If the septum is punctured too many times, it will leak air into the gas chromatographic system, even though it is under pressure. At high temperatures, above 150 to 200°C, air (oxygen) in the carrier gas from a septum leak will degrade the stationary phase. An excessive septum leak will also produce a change in carrier gas flow rate (a change in retention time) and loss of sample (irreproducible peak heights) due to outflow from the leak. When installing the septum, do not overtighten the retaining nut. The septa will swell at high temperature and extrude out of the injection port. A snug fit at room temperature is sufficient. It is important for septum life to make sure the injection needle is sharp with no bent tip. Fine emery cloth, or a fine sharpening stone, can be used to sharpen the point.

6.3.4.2 Ghost peaks may be observed in temperature programmed runs due to septum bleed. Septum bleed is due to the thermal decomposition, 300°C or higher, of the septum that produces primarily lower molecular weight cyclic dimethylsiloxanes. It contributes to baseline response and is frequently observed as evenly spaced peaks in a temperature programmed run in which no sample has been injected. This situation can be demonstrated by the disappearance of ghost peaks after placing aluminum foil (precleaned with solvents such as methylene chloride or toluene) over the inner face of the septum or by turning off the injector temperature and making several blank runs. Septum bleed can be decreased by using either air- or water-cooled septum retaining nuts, by using a septum flush head, or by using special high-temperature septa which are available from a number of gas chromatographic supply houses.

6.4 *Detector Temperature Control*—The detector temperature should always be above that of the maximum operating analytical temperature, to prevent the possibility of condensation of sample components or stationary phase bleed in the detector and connecting line. Because there is usually some temperature gradient across a detector, the temperature should be set at 30 to 50°C above the maximum analysis temperature to ensure that the entire detector is hot enough to prevent condensation. Usually, it is neither necessary nor desirable to use an excessively high temperature since this can result in reduced sensitivity, increased noise level, frequent need to clean the detector, and thermal decomposition of sample or stationary phase.

6.5 *Measurement of Temperature*—The choice of sensing elements used to measure temperature depends on the desired accuracy (control about a set point) and precision of the measurements. Instrument read-outs should be verified periodically. Some common temperature measurement devices are as follows:

6.5.1 *Standardized Mercury Thermometer:*

Range, °C	Accuracy, °C
0 to 100	±0.02
100 to 200	±0.05
200 to 400	±0.50

6.5.2 *Calibrated Platinum Resistance Thermometer:*

Range, °C	Accuracy, °C
−140 to 500	±0.01

6.5.3 Thermocouple (iron – constantan, or other).

6.6 *Analytical Column:*

6.6.1 The analytical column is a length of tubing (glass, metal, or plastic) that is filled with a packing material. It is discussed thoroughly in Section 8.

6.6.1.1 *Column Characteristics*—Specified by method.

6.6.1.2 *Carrier Gas*—Specified by method.

6.6.1.3 *Sample Size*—Variable within limits.

6.6.1.4 *Flow Rates of Carrier Gas and Detector Gases*—Variable within limits.

6.6.1.5 *Column Temperature*—Usually specified by method, and

6.6.1.6 *Physical or Chemical Characteristic of Compound Analyzed*, or both.

6.6.2 *Detector Characteristics*—Desirable detector characteristics should include the following:

6.6.2.1 Good stability (low noise level, minimum response to changes in temperature and flow rate).

6.6.2.2 Ruggedness and simplicity.

6.6.2.3 Sensitivity to the components for which analysis is desired. Use either a selective detector for materials of interest or one with a similar response for all components.

6.6.2.4 Linearity of response versus sample concentration. Wide linear range.

6.6.2.5 Rapid response to changes in column effluent composition (small internal volume or flow-through design, or both).

6.6.2.6 Detectors, which are nondestructive and do not contribute to band broadening may be used in series with other detectors.

6.7 *Types of Detectors*—The detector is located at the outlet end of the chromatographic column and both senses and measures the amount of components that have emerged from the column. The optimum detector should have high sensitivity, low noise level, a wide linearity of response, a response to all compounds of interest, and yet be insensitive to changes in flow and temperature. Selective detectors are characterized as having selective, or greatly enhanced response to certain components. Linearity is decreased for all detectors by column bleed. As many as forty detection systems have been reported, yet only about a dozen are commonly used. Table 1 shows some of the more commonly used detectors. Of these, the thermal conductivity, the flame ionization, the electron capture, the nitrogen-phosphorus, and the flame photometric detectors are the most popular. Nondestructive detectors should be vented to a hood to remove any toxic effluents from the workplace. The effluent from destructive detectors may also be toxic. Details on detectors can be found in the applicable methods in Practices E 516, E 594, E 697, E 840, and E 1140.

6.8 *Programmed Temperature Operation*—The apparatus used in programmed temperature gas chromatography differs in some respects from that normally used for isothermal work. Basically, the column temperature is varied with time (program rate) to enhance speed of separations. The advantages of using programmed temperature operation include better resolution of lower boiling components because of lower starting temperature and greater sensitivity because of sharper peaks for the higher boiling components.

6.8.1 *Column Heater and Temperature Programmer*—It is of utmost importance that the column temperature program be reproducible, and that the difference between the set (desired) temperature and the true average column temperature be as small as possible. However, these requirements are difficult to achieve at high heating rates and with columns of large diameter. The mass of the column and its heater should be kept as small as possible. This will minimize thermal lag and will give proportionately small variations around the set temperature at any time. Proportional temperature controllers supply almost full power to the heater until the set point is very closely approached.

6.8.2 The recirculating air bath is the recommended method of heating in programmed temperature gas chromatography (PTGC). The obvious advantages are extremely rapid heating (and cooling after an analysis is completed) with very little temperature lag.

6.8.3 Liquid baths may be used for very low heating rates. They are commonly contained in taped Dewar flasks.

6.8.4 No matter what type of heating device is used, accurate control of the temperature program is necessary. This is usually accomplished by appropriate electronic systems that develop linear (or other) programming rates as desired.

6.8.5 Detectors for programmed temperature gas chromatography should be relatively insensitive to minor temperature and flow fluctuations and insensitive to stationary-phase bleed. These difficulties can be overcome by operating the detector at or near the upper temperature limit for the analysis and by using adequate flow controllers. If stationary-phase bleeding is excessive during PTGC runs, a second conditioning procedure (Section 9) might improve the situation. Alternately, a duplicate analysis column can be used on the reference side of the detector. By equalizing substrate bleed on both sides of the detector, the baseline drift can be substantially compensated. However, this technique does not improve column life and is detrimental to detector linearity. If at all possible, operate the column within its recommended temperature range.

6.8.6 When using the temperature programming technique, the resistance to carrier gas flow in the gas chromatographic column increases with increasing temperature. The flow controllers need a positive pressure of 10 psi to operate properly. By setting the second stage of the regulator to 40 to 60 psi, there will usually be sufficient excess pressure to maintain a constant gas flow through the column. Higher pressures might be required to maintain flow when using relatively long columns of 10 ft, or longer, or packings finer than 120 mesh.

7. Hazards

7.1 *Gas Handling Safety*—The safe handling of compressed gases and cryogenic liquids for use in chromatography is the responsibility of every laboratory. The Compressed Gas Association, a member group of specialty and bulk gas suppliers, publishes the following guidlines to assist the laboratory chemist to establish a safe work environment: CGA P-1, CGA G-5.4, CGA P-9, CGA V-7, CGA P-12, and HB-3.

TABLE 1 Applicability of Commonly Used Gas Chromatographic Detectors

Detector[A]	Applicability (Type of Compound)	Range of Minimal Detectable Amounts (grams)
Thermal Conductivity	All	10^{-6} to 10^{-7}
Flame Ionization	Organic	10^{-12}
Electron Capture	Halogenated and Oxygenated	10^{-12} to 10^{-15}
Flame Photometric	Sulfur, Phosphorus	10^{-11}
Alkali Flame	Nitrogen, Phosphorus	10^{-12} (even lower for phosphorus)

[A] Further information can be found in Practices E 516, E 594 and E 697.

8. Materials

8.1 *Stationary Phases*—The stationary phases (partitioning agents) that have been successfully used for specific separations are found most quickly by a literature search. Many phases are listed in ASTM publications AMD-25A and AMD-25A-51.[4] The most desirable stationary phases do not volatilize (bleed) significantly from the solid supports at temperatures required to elute the sample.

8.1.1 The polarity of the stationary phases is currently best characterized by McReynolds Constants.[5] The higher the McReynolds Constant, the more polar the phase. Rohrschneider constants can also be used to measure the polarity of stationary phases.[6]

8.1.2 The effects of using polar and nonpolar stationary phases are summarized as follows:

8.1.2.1 Nonpolar stationary phases separate compounds primarily by order of relative volatility or boiling point.

8.1.2.2 Polar stationary phases separate compounds by order of both relative volatility and relative polarity. With polar phases, nonpolar compounds will elute before polar compounds of the same boiling point.

8.1.2.3 Polarity alone is insufficient to describe the separation power of a column. One must consider the overall selectivity of a column towards a set of analytes. This selectivity is a summation of the effects of dispersive interactions, acid-base interactions and the dipole interactions offered by the various pendent groups in the stationary phase.

8.1.3 The stationary phases used in gas chromatographic columns have both minimum and maximum temperature limits. The chromatographer must be aware of the limits for the phase being used. Below the minimum temperature, the phase will behave as either a viscous liquid or a solid. Less efficient separation will be observed, and the chromatographic results will be exhibited as broader peaks in the gas chromatogram due to poor mass transfer of components in the stationary phase.

8.1.3.1 Above the maximum temperature limit, the phase will begin to bleed off the column at an accelerated rate, and the observed results will include a drifting baseline or excessive spiking on the baseline. Under these conditions, the liquid phase will decompose or volatilize, and thus be removed from the column. This situation will eventually lead to decreased retention times with broader peaks resulting in poorer resolution of very close peaks. Peak tailing will also be observed as the uncoated surface becomes exposed by removal of liquid phase, thus shortening column life. Bleeding also can expose bare support surface that can adsorb molecules being analyzed and reduce column efficiency. In extreme cases, phase bleeding will result in fouling the detector and connecting lines. The observed maximum temperature will depend upon many experimental variables, such as type of liquid phase column, conditioning, phase-loading level, column temperature, sensitivity setting of the detector, and purity of the carrier gas. In programmed temperature runs, the column can sometimes be operated for short periods about 25°C above maximum temperature. However, column bleed should be minimized for quantitative results since it decreases the linear range of all detectors.

8.2 *Active Solids:*

8.2.1 *Molecular Sieves*—The synthetic zeolite molecular sieve sorbents separate molecules by size and structural shape. Isomers with a more round shape, as branched versus straight chain molecules, diffuse in and out of the zeolite structure more easily than isomers with the long chain structures. Separations are affected by the differences in times required for molecules of different sizes to find their way into and out of the sieve-like structure of the adsorbent. Molecular sieves are most useful for separating H_2, O_2, N_2, CO, and CH_4. Carbon molecular sieves are also available, and can be used to separate O_2, N_2, CO, CO_2, H_2O, and C_1 to C_4 hydrocarbons.

8.2.2 *Porous Polymers:*

8.2.2.1 One type of porous polymer used in gas chromatography is available in the form of microporous cross-linked, polymer beads produced by copolymerizing styrene and divinylbenzene or more polar copolymers, or both. These materials are generally used as received without coating with any liquid phase. They provide symmetrical peaks for polar, hydrogen-bonding compounds such as water, alcohols, free acids, amines, ammonia, hydrogen sulfide, etc., and organic compounds up to molecular weights corresponding to about 170.

8.2.2.2 Another porous polymer is poly(2,6-diphenyl-*p*-phenylene oxide). This material is useful for the analysis of amines, alcohols, and hydrogen-bonding compounds. It is also used as an adsorbent for trapping trace organic compounds in water and air.

8.2.3 *Silica Gel, Alumina, and Carbon*—Among the active solid adsorbents are silica gel, alumina, and activated carbon. They are useful for low-boiling hydrocarbons.

8.2.4 Solid adsorbents modified by low concentrations of liquid phases may retain the advantageous properties of both. Some solid adsorbents can be modified by the addition of surface activating compounds such as wetting agents, silver nitrate, and the metal salts of fatty acids.

8.3 *Diatomaceous Earth Supports*—The most popular gas chromatographic supports are those prepared from diato-

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[4] *Gas Chromatographic Data Compilation*, ASTM, 1981.

[5] McReynolds, W. O., *Journal of Chromatography Science*, Vol 8, 1970, p. 685.

[6] Supina, W. R., and Rose, L. P., *Journal of Chromatography*, Vol 8, 1970, p. 214.

maceous earth, also called diatomaceous silica or kieselguhr. The two main types are white and pink in color. The white supports are recommended over the pink supports because of their more inert surface. The former are, however, very friable and must be handled very carefully when preparing packings and loading into gas chromatographic columns. Before using these supports, check the manufacturer's literature for comments on their use.

8.3.1 The white-colored supports are produced by calcination of diatomaceous earth with sodium carbonate as a flux. In this process, the diatomaceous earth fuses, due to formation of a sodium silicate glass. The product is white in color due to conversion of iron oxide into a colorless complex of sodium iron silicate. These white materials are used to prepare the more inert gas chromatographic supports. However, they are fragile and subject to abrasion from excessive handling in the course of sieving, packing, or shipping. Abrasion will produce finer particles, or fines, which will decrease column efficiency.

8.3.2 The pink-colored supports are prepared by crushing diatomaceous earth firebrick that has been calcined with a clay binder. The metal impurities remaining form complex oxides that contribute to the pink color of the support. These pink supports are denser than the white supports because of the greater destruction of the diatomite structure during calcination. They are harder and less friable than the white supports and are capable of holding larger amounts of liquid phase (up to 30 %) without becoming too sticky to flow freely. Their surface is generally more adsorptive than white supports. For this reason, they are not recommended for use in the gas chromatographic analysis of polar compounds. However, pink supports provide excellent efficiencies for the analysis of hydrocarbons and organic compounds of low polarity.

8.3.3 *Chemical Treatment of Diatomaceous Earth Supports*—Neither the pink nor the white materials give generally acceptable analysis of more polar compounds without further treatment. With these compounds, severe peak tailing is often observed, especially with the dense pink supports. This tailing is due to the presence of adsorptive and catalytic centers on all diatomaceous earth supports. The adsorptive sites are attributed to metal oxides (Fe, Al) and surface silanol groups, -SiOH, on the support surface. The latter are capable of forming hydrogen bonds with polar compounds.

8.3.3.1 Metal impurities are removed by washing with hydrochloric acid, which leaches out iron and aluminum and renders the surface both less adsorptive and less catalytically active. However, even with acid washing, the pink supports are still more adsorptive toward polar compounds than the white-type supports. Acid washing is sometimes followed by base washing, which seems to remove only minor amounts of metal impurities, but is a good pretreatment for supports that are to be used for the analysis of basic compounds.

8.3.3.2 Neither acid or base washing is effective in reducing peak tailing due to hydrogen bonding with the surface silanol groups, -SiOH. These groups are most effectively masked by treatment with dimethyldichlorosilane.

8.3.4 Acid-washed silanized grades of white diatomaceous earths are recommended as supports for nonpolar and medium polarity liquid phases. Because of the hydrophobic character of a silanized diatomaceous earth, even coating of the most polar liquid phases is difficult to achieve. Acid-washed, silanized grades of white diatomaceous earths are recommended as supports for the polar liquid phases, such as polyesters and silicones of high cyano-group content.

8.3.5 If the column is 6 ft (2 m), or less, use particle size of 100 to 120 mesh (125 to 149 μm) for highest efficiency under isothermal conditions. If the column is longer than 6 ft, use 80 to 100 mesh (149 to 177 μm) particles. If temperature programming is used, 80 to 100 mesh particles should be used to lessen resistance to carrier gas flow.

8.3.6 Further information concerning the liquid phase loading is given in 9.3.

8.4 *Halocarbon Supports*—The two types of halocarbon supports are those prepared from poly(tetrafluoroethylene) and poly(chlorotrifluoroethylene). These supports are relatively inert and are nonpolar. They eliminate peak tailing observed in the analysis of organic compounds capable of hydrogen bonding, such as water, alcohols, amines, etc. They are the preferred supports in the analysis of corrosive halogen compounds such as HF, BCl_3, UF_6, $COCl_2$, F_2, and HCl.

8.4.1 Poly(tetrafluoroethylene) supports require special handling procedures. When used as received, they are soft and tend to form gums upon handling. They can also build up a static charge and spray out of the column during the packing operation. These problems can be virtually eliminated by cooling the support to 0°C before coating with liquid phase and by avoiding the use of glass vessels. Rinsing poly(tetrafluoroethylene) with methanol and drying before use is another way to eliminate the static-charge problem.

8.4.2 Supports prepared from poly(chlorotrifluoroethylene) are structurally harder and are much easier to handle and to pack into a column.

8.5 *Tubing Materials*—Tubing materials should be chosen on the basis of the following criteria:

8.5.1 They should be nonreactive with the stationary phase, sample solvent, and carrier gas.

8.5.2 They should possess physical properties to withstand temperature and pressure of operating conditions, and

8.5.3 They can be shaped to fit in the column oven of the chromatograph.

8.5.4 Satisfactory materials include glass, nickel, stainless steel, and glass-lined stainless steel. Glass is the material of choice, unless conditions prohibit its use. Nickel tubing is more inert than stainless steel in most applications. Less frequently used column materials are poly(tetrafluoroethylene), aluminum, and copper.

8.6 *Carrier Gas*—The use of an impure carrier gas will produce problems in gas chromatography. Trace water and oxygen can cause decomposition of the liquid phase coated on the support. The common carrier gases, helium and nitrogen, should contain less than 5 ppm water and less than 1 to 2 ppm oxygen by volume. An oxygen adsorption trap can be used to remove trace oxygen, while trace amounts of water and hydrocarbons with molecular weights higher than methane, can be trapped on a molecular sieve trap. Place the molecular sieve drier nearest the gas supply. Calcium sulfate has been used in drying tubes, but cannot dry carrier gas to the same level as molecular sieve.

8.6.1 For some applications, hydrogen may be the preferred carrier gas. However, additional safety precautions are required due to hydrogen's explosive nature.

8.6.2 Air (oxygen) can leak into the gas chromatographic system through loose fittings or a septum, that has been punctured too many times, even though the carrier gas is under a pressure of 40 to 60 psi. Keep all fittings on the gas delivery lines tight, and check them at periodic intervals. Change the septum in the injection port frequently. Plastic tubing should never be used for carrier gas, hydrogen fuel (for FID), or make-up gas lines due to the possibility of oxygen or moisture diffusing through the tubing wall.

8.6.3 Each cylinder of carrier gas has its own impurity level. Occasional tanks contain large amounts of impurities which might overcome a low-capacity oxygen adsorption trap and destroy a gas chromatographic column at high temperature. A new tank or a fresh oxygen adsorption unit, or both will improve this situation.

8.6.4 Always change the tank when the pressure is less than 200 psi. As the total pressure in the cylinder decreases, there is an increase in the partial pressure of the water and other impurities adsorbed on the inner walls of the gas cylinder. As a result, the last amounts of gas delivered from the gas cylinder contain high levels of impurities.

8.6.5 *Carrier Gas for Instruments with Thermal Conductivity Detectors*—A major factor in sensitivity is the difference in thermal conductivity of the compound being analyzed and the thermal conductivity of the carrier gas. Helium (thermal conductivity = 33.60 cal/cm-s-°C) is usually the carrier gas of choice.

8.6.6 *Carrier Gas for Instruments with Flame Ionization Detectors*—The most commonly used carrier gases are nitrogen or helium. A maximum impurity level of 0.05 volume % does not generally interfere with most applications. Hydrogen is less commonly used in the US but is more popular in Europe because of availability and relatively low cost.

NOTE 2—If hydrogen is used, special precautions must be taken due to its explosive nature, to ensure that the system is free from leaks and that the effluent is properly vented.

8.6.7 *Carrier Gas for Instruments with Electron-Capture Detectors*—Users should follow the manufacturers' recommendations for the choice of carrier gas. Some common ones are nitrogen or 95 % argon/5 % methane. When using a tritium source in the detector, do not use hydrogen as the carrier gas. Hydrogen will replace tritium in the source.

9. Preparation of Packed Gas Chromatographic Columns

9.1 *Preparation of the Tubing Material:*

9.1.1 Glass columns should be cleaned and deactivated, first by rinsing with 30 mL acetone and then 30 mL toluene. Next, fill the column with 10 volume % solution of dimethyldichlorosilane in toluene. Allow the solution to stand in the column for 30 min. Finally, rinse the column with anhydrous toluene and then anhydrous methanol to cap unreacted DMDCS CL groups. Dry the column by passing a stream of dry nitrogen through it. Cap both ends of the column until such time that it can be packed.

9.1.2 Metal columns should be cleaned thoroughly before packing by rinsing with methanol, acetone, and chloroform. The column should be dried by passing nitrogen or dry air through it. Do not blow house air through the column since this compressed air usually contains an oil aerosol from the pump.

NOTE 3—Most chromatographic supply houses provide metal tubing that has been washed with solvents and is ready for use.

9.1.3 An alternative procedure is recommended for nickel tubing and can be used to clean stainless steel tubing. Rinse the nickel tubing with ethyl acetate, methanol, and distilled water. Then fill the tube with 20 volume % nitric acid and let it stand for 10 min. (**CAUTION:** Work in a hood and wear safety equipment when using nitric acid.) Next, rinse the tube with distilled water to neutrality and then rinse with methanol and acetone. Finally, dry the column by blowing nitrogen or helium through it.

9.1.4 The column length is generally 3 to 6 ft (1 to 2 m). Shorter columns can be used to decrease the time of analysis or to separate high boiling compounds. Longer columns are used to improve resolution, but have longer analysis times. (Columns longer than 20 ft (6.1 m) require excessive pressures to maintain the proper carrier gas flow.) A compromise is usually made between analysis time and resolution. As a general rule, an increase or decrease of column length by a factor of 3 to 4 is necessary to see a significant change in peak separation.

9.1.5 The diameter of the column can be $\frac{1}{8}$ in. (3.2 mm) or $\frac{1}{4}$ in. (6.4 mm) outside diameter. The $\frac{1}{8}$-in. column has less sample capacity, but greater efficiency, and is the most common type. Glass columns are generally 2 mm or 4 mm inside diameter. Some analysts have found that $\frac{3}{16}$ in. (4.8 mm outside diameter) metal columns are the ideal combination between the capacity of $\frac{1}{4}$ in. (6.4 mm outside diameter) columns and the efficiency of $\frac{1}{8}$ in. (3.2 mm) outside diameter columns.

9.2 *Choice of Diatomaceous Earth Support for Packed Columns*—See 8.3.

9.3 *Phase Loading on Diatomaceous Supports*—For preparative work and analysis of substances boiling below room temperature, use 15 wt % loadings for white-type supports and 30 wt % for pink-type supports. For general work, use loadings of the range of 3 to 15 wt %. For highest efficiency, shortest retention times, and the least amount of bleed during high-temperature operation, use 3 wt. % loadings. The lower phase loadings have lower sample capacity and elute components more rapidly and at lower temperatures. Always check the manufacturers' literature for suggested phase loadings for a particular support. For some applications (especially headspace analysis) loadings as low as 0.2 wt. % are used which result in very narrow peaks and short analysis times. High phase loadings tend to produce less reactive packings.

9.4 *Preparation of the Gas Chromatographic Packing*—The following procedures describe the coating of a solid support with stationary phase. The following four methods are commonly used to prepare gas chromatographic packings: (*a*) Filtration or Solution Coating Method, (*b*) Rotating Evaporator Method (*c*) Evaporative Method, and (*d*) Vacuum Evaporative Method. When preparing packings with loadings in the range of less than 5 wt %, the Filtration or Solution Coating Method is recommended. This method is preferred because it provides minimum handling of the friable white-type supports. For loadings of more than 5 wt. %, other methods can be used. The Rotating Evaporator

Method is recommended, but should only be used if a rotating evaporator is available, which turns very slowly at 20 to 30 rev per min.

NOTE 4—A 5 wt. % loading of stationary phase consists of 5 g stationary phase added to 95 g of support.

9.4.1 *Filtration or Solution Coating Method*—Prepare 100 mL of a solution of the desired phase in a vacuum filter flask. Use a suitable high boiling solvent (boiling point more than 60°C). The actual loading of the liquid phase on the support will depend upon both the viscosity of the phase solution and the density and mesh size of the support.

9.4.1.1 Add 20 g of support to the filter flask. Reduce the pressure in the filter flask for a few minutes with a water aspirator, then release the vacuum. Repeat this procedure for several cycles in order to remove air bubbles from the pores of the support particles. Be prepared to release the vacuum if the slurry foams excessively.

9.4.1.2 Allow the slurry to stand for several minutes. Pour the slurry into a coarse-frit sintered-glass filter funnel, and allow the solvent to drain freely until the support settles.

9.4.1.3 Apply vacuum cautiously and stop instantly when the solvent stops dripping. Dump the support into a flat borosilicate glass dish, and allow it to dry. Do not scrape the particles out of the funnel, since this might crush the particles. Do not resieve before use.

9.4.1.4 The actual phase loading will depend upon the viscosity of the phase solution and both the density and particle size of the support. For example, a 2 % solution of dimethyl silicone gum liquid phase will give a 3.8 wt % loading on white-type supports. A 10 wt % solution of a less viscous liquid phase will give a 5.5 wt % loading on white-type supports and 7.5 wt % on pink-type supports. Loadings obtained with other phases on other supports are best determined by experimentation.

9.4.1.5 The best way to determine the percent loading is to extract it from the support by extraction in a Soxhlet apparatus and determine the weight loss. Alternatively, measure the volume of solution recovered and calculate the volume of solution held up by the support. Calculate the approximate percent loading on the support by assuming that the concentration of the solution does not change.

9.4.2 *Evaporative Method:*

9.4.2.1 Weigh out the desired amounts of support and phase. Use a larger amount than that required to account for attrition, spills, etc. Dissolve the liquid phase in a chemically inert, low-boiling solvent contained in a filtration flask (see Table 2). (Most catalogs of gas chromatography equipment suppliers contain lists of suitable solvents.)

9.4.2.2 Gradually add the support to the solution with gentle swirling or agitation but with no mechanical stirring. (Suggested solvents are given in Table 2.) The amount of solution should be just enough to wet the solid support and form a slurry with little excess solvent.

9.4.2.3 Evacuate the flask briefly several times to remove air bubbles from the pores of the support. Be prepared to release the vacuum if the slurry foams excessively.

9.4.2.4 Transfer the slurry to a large flat borosilicate glass dish, and slowly evaporate the solvent in a hood with no further handling. The dish must be of a size that the packing is spread on the bottom in a thin layer, no more than about ¼-in. thick. A borosilicate glass baking dish makes a suitable container.

TABLE 2 Solvent for Liquid Phases

Liquid Phase Type^A	Solvent
Dimethyl Silicone	Toluene
Phenylmethyl Silicone	Ethyl Acetate
Cyanopropylphenyl Silicone	Methylene Chloride
Trifluoropropyl Silicone	Ethyl Acetate
Polyethylene Glycol	Methylene Chloride
Cyanopropyl Silicone	Methylene Chloride
Other Liquid Phases	Use solvent as recommended by supply house.

9.4.2.5 The critical stage occurs when excess solvent has evaporated, but the bed is still quite damp with a slight excess of solvent. Break up the damp bed by gently raking it with a spatula. As the solvent evaporates from the surface of a static bed of support, it leaves a higher concentration of phase at the bed surface. Therefore, the bed must be broken up frequently during the final stages of solvent evaporation to prevent formation of an unevenly coated support.

9.4.2.6 Continue to air-dry the material in the hood until the last traces of solvent are gone. Avoid excessive handling of the particles to prevent formation of fines due to abrasion, especially in the case of the white-type supports.

9.4.3 *Rotating Evaporator Method*—Prepare the slurry of support and phase as described in 9.4.2.1 to 9.4.2.3, except in an indented, round-bottom flask. Connect the flask to a rotating evaporator. Rotate the flask very slowly (less than 20 to 30 revolutions per minute) and evaporate the solvent under reduced pressure (water aspirator). Very slow rotation is necessary to prevent the particles from abrading against each other. Use of a heat lamp increases the evaporation rate. This method is not recommended for fluorocarbon supports.

9.4.4 *Vacuum Evaporative Method*—Prepare a slurry of support and phase in a filtration flask of sufficient capacity. (Suitable solvents are given in Table 2.) Attach the flask to a vacuum source (water aspirator) and apply vacuum briefly. (Be prepared to release the vacuum if the slurry foams excessively.) Repeat this procedure several times in order to remove the air bubbles from the pores in the support.

9.4.4.1 Apply the vacuum for a longer period, and swirl the contents of the flask occasionally until all the solvent is almost evaporated. This is the critical stage.

9.4.4.2 Now shake the contents of the flask by gently bumping the flask on a wood or plastic board. This will break up the bed of packing. Do not allow the solvent to evaporate from the surface of the support bed. Otherwise, the solvent will evaporate and leave a higher concentration of phase at the bed surface.

9.4.4.3 Continue to apply vacuum until the packing is a freely flowing powder, then transfer it to a tray for air-drying in a hood.

9.4.5 *Fluidized Drying Technique*—This technique has been used to produce efficient, uniformly coated packings. During the drying stages of methods 9.4.1 to 9.4.4, when the packing has reached the consistency of a wet sand, add it to a fluidizer. Then dry the packing by passing a flow of inert, warmed gas (nitrogen or helium) through the bed of packing.

9.5 *Packing the Gas Chromatographic Column*—The purpose in packing a gas chromatographic column is to fill the column with packing as completely as possible, leaving no empty spaces. Two variations are noted in 9.5.3 and 9.5.4 (a pressure-fill procedure and a vacuum fill procedure).

9.5.1 It is preferable to coil the column before packing to prevent crushing of the support particles. Metal columns can be coiled after loading to meet equipment requirements. Bends in the packed region must never be made with radii less than those specified in 9.5.2, to avoid crushing the packing in the column.

9.5.2 Right-angle bends are often necessary to make connections to injection and detection systems, and must be made before packing the column since some tubing deformation will occur, which will crush some of the solid support. Bends for such purposes should be within 4 in. (10 cm) of the column ends. For coiled columns, minimum diameter mandrels should be as follows: for ⅛ in. (3.2 mm) OD column use a 1½-in. (38-mm) mandrel; for ¼ in. (6.4 mm) OD column use a 2-in. (51-mm) mandrel. These configurations do not preclude the use of U- or W-shaped columns. If a U- or W-shaped column is to be used, the minimum 180° bend diameter must be at least that given for the above mandrel sizes.

9.5.3 *Pressure Fill Procedure*—To each end of the column to be filled, fit a nut, a back ferrule, and a suitable front ferrule. Place a small plug of silanized glass wool into the detector end of the column, and cap the column by screwing in a metal cap with a 1⁄16-in. vent hole drilled into it. When analyzing trace acidic compounds, as organic acids and phenols, adsorption can be decreased by using phosphoric acid-treated glass wool to plug the column ends. Wear safety glasses when pressure-packing columns.

9.5.3.1 Attach the end of the empty column to an apparatus similar to that shown in Fig. 2A. Add to the reservoir sufficient packing material to fill the column, plus about 30 %. Attach the upper end of the reservoir to a nitrogen supply line controlled to provide approximately 40 psi. Check that all connections are tightened, place a safety shield in front of the setup, and apply 40 psi to the system.

9.5.3.2 As the stationary phase starts to fill the column, gently tap the column with a wood rod (handle of spatula or screwdriver) or an electrical vibrator set at a very low vibration level. Continue tapping until the packing shows no voids and the level of packing in the reservoir remains constant.

9.5.3.3 Shut off the nitrogen supply and wait for the pressure to dissipate. Disconnect the column from the reservoir. Do not disconnect the column while it is under pressure. Have a clean beaker available to collect excess packing material that will fall from the opened reservoir. Tap out about ⅛ in. (3 mm) of column packing, and replace it with a silanized glass wool plug. Affix a metal column tag engraved with a description of the stationary phase, loading, support, and the assigned column number.

9.5.4 *Vacuum-Fill Procedure:*

9.5.4.1 Clamp the column so that the detector and injector ends point upward. Plug the detector end of the column with a ¼-in. plug of silanized glass wool. Use phosphoric-acid treated glass wool when analyzing for trace organic acids and phenols.

9.5.4.2 Attach a small funnel to the injection port end of the column. Attach the detector end of the column to a vacuum source, either a vacuum pump (preferably) or a water aspirator. (If a water aspirator is used, a 500-mL filter flask, or the device shown in Fig. 2B, should be placed in the line between the pump and the column.) Do not turn on the vacuum yet.

9.5.4.3 Add 1 to 2 mL of packing to the funnel, and tap the column gently to settle the packing. A pencil or a wooden spatula handle can be used. Alternatively, the column can be stroked with a plastic saw. The use of an electric vibrator is not recommended. Excessive vibration will cause the particles to abrade against each other, producing fines and newly fractured surfaces that are not coated with stationary phase.

9.5.4.4 Turn on the vacuum source. Continue to add the packing in small increments with tapping until the column is full. Finally, apply pressure to the head of the column to pack it a little tighter. However, take care to make sure the pressure is equalized slowly, because packing will be blown out of the column if the pressure is released too suddenly.

9.5.4.5 Next, tap out enough packing to create a ⅛ in. (3 mm) void space at the injector port end of the column. Plug this end with a silanized glass wool plug. Do not pack the plug too tightly. This will either impede the carrier gas flow or crush the packing particles.

9.5.4.6 Higher efficiencies are always observed if the column is packed for on-column injection. In this technique, the column is packed so that there is space at the injection port end of the column, which is then placed inside the injection port. This void space should be of such a length that the injection needle just reaches, or slightly penetrates, only the glass wool plug, not the packing, when the column is installed. Thus the sample is injected almost directly onto the column.

9.6 *Conditioning of Packed GC Columns:*

9.6.1 The purpose of the conditioning process is to remove extraneous material (solvent and adsorbed material) from the column before analytical usage. Since the column is heated, the liquid phase also redistributes itself over the support surface to provide a more even coating.

9.6.2 Install the column into the gas chromatograph at the injection side only. Do not connect the column to the detector during the conditioning stage. Any column bleed might foul the detector and the connection lines between the column and detector. Turn on the normal analytical carrier gas flow and flush air out of the column at ambient temperature for 30 min.

9.6.3 Heat the column at a rate of 2°C/min to the conditioning temperature. The latter temperature should be at least 25°C higher than the analytical temperature but 25°C lower than the maximum operating temperature recommended for the liquid phase. Maintain this temperature overnight with carrier gas flow.

9.6.4 The next day cool the column and connect it to the detector. Detectors operated in very sensitive modes, particularly the electron capture detector, might require two or more days of conditioning at the higher temperatures before a satisfactory baseline is obtained. (Other sources for baseline drift and noise are impurities in the carrier gas, a dirty detector, air leaks in the gas-line fittings, insufficient carrier gas pressure, a much-punctured septum, chemical decomposition of the phase (due to presence of traces of acid or base on the support, in the phase, or on the inner column walls, and incorrect fuel gas ratios to the flame ionization detector.)

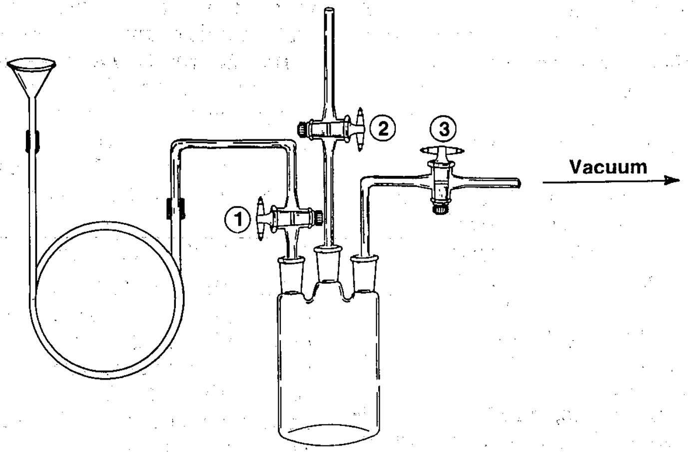

FIG. 2A Vacuum Fill Apparatus

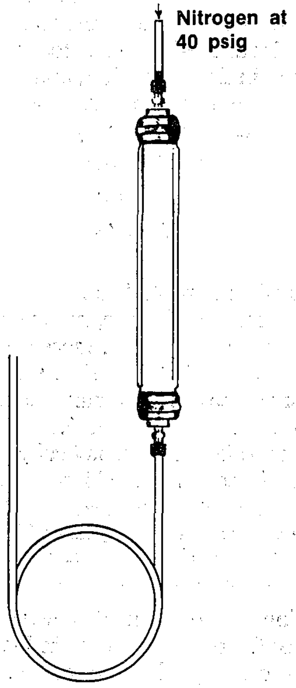

FIG. 2B Pressure Fill Apparatus

9.6.5 There is a special "no-flow" conditioning procedure which can be used with certain silicone phases, as methyl and methylphenyl silicones with or without low vinyl content. It has been reported to improve analysis for drug compounds. The procedure starts by conditioning the column for ½ h as described in 9.6.2. Turn off the carrier gas flow, cap the free end of the column with a metal cap, and heat at 310°C for 1.5 h with no carrier gas flow. Cool the column to 100°C. Uncap the oven, turn on the carrier gas flow, and continue the regular conditioning procedures listed in paragraph 8.6.3.

NOTE 5—This no flow conditioning procedure may damage or destroy non-silicone containing stationary phase or silicone containing functional groups other than phenyl or methyl.

9.6.6 Many of the liquid phases are commercial-grade material, and conditioning might require several days before the noise level is low enough to provide usable baselines at high sensitivity. The use of gas chromatographic-grade phases is recommended since they have been carefully purified and long periods of conditioning are usually not necessary.

10. Sample Injection Procedures

10.1 *Injection Technique*—Useful analyses are obtained only by injecting representative samples into the gas chromatograph. Since chromatographic samples are small, the best practices and procedures must be followed.

10.1.1 If elution is to be otherwise, the sample injection must be almost instantaneous in order to introduce the sample as a plug. Avoid unnecessary sample dilution and inadvertent trapping.

10.2 *Sample Size*—Sample sizes used for analysis with 2- to 5-mm ID packed columns are in the range of 0.1 to 10 µL multi-component liquids. Gas samples usually range from 10 µL to 2 mL.

10.2.1 There is frequently a reduction in retention times with sample size when the column is overloaded. Therefore, when identifying by comparison with a known sample, the same amount of each component must be used in an amount that does not overload the column. The sample size that overloads the column is that size which decreases the efficiency (Section 11) by 10 % compared to a smaller sample size. Sample overload is sometimes shown by peaks with sloping fronts and backs that are almost perpendicular to the baseline. (Another reason for this peak shape is insufficient vaporization of the sample. Either the injection port or the column temperatures, or both, are too low.)

10.3 *Sample Injection Devices*—Samples may be introduced by syringes, automatic sample injectors using syringes, or sample valves. (There are also devices that introduce capsules containing sample into the injection port.) For rigorous quantitative work, any sample introduction device should be flushed and filled at least three times with the sample immediately before use.

10.3.1 The most common method for liquids is the use of a syringe injection technique through a self-sealing septum. In the usual 10-µL syringe, there is approximately 0.8 µL of dead volume in the syringe needle. This volume is additional to the volume in the syringe barrel and can be measured by withdrawing the entire sample volume into the syringe barrel.

10.3.1.1 First, pump the sample back and forth vigorously in the syringe to wet the plunger and to remove air bubbles. Then withdraw the sample back into the syringe so that the entire volume can be read on the volume marks of the syringe barrel. When preparing the syringe for injection, never leave the sample solution in the needle. This technique will minimize sample boiling out of the needle when it is inserted into the hot injection port.

10.3.1.2 In an alternative procedure, called the solvent-flush technique, load the syringe in the following order: solvent, air, sample solution, and air, with only air remaining inside the needle. When the sample is injected, the solvent is the last to leave the syringe, and it rinses out sample residue in the needle.

10.3.1.3 Wipe the syringe needle off before injection. Insert the needle carefully through the GC septum, inject the sample at once, and withdraw the needle in one continuous motion.

10.3.1.4 Often the tip of the needle becomes bent, forming a fish hook that will tear the septum on subsequent injections. This can be detected by brushing the end of the needle gently over the end of a finger. A few strokes on a sharpening stone will remove the fish hook.

10.3.1.5 Syringes should be cleaned with a solvent that will remove all traces of contamination. Consult the manufacturer's literature for further information. Many chromatographic supply vendors sell suitable cleaning solutions and kits. Liquid sample valves, in both automated and manual versions, are also available.

10.3.1.6 Gas samples are most conveniently injected using gas-tight syringes. These devices are quite satisfactory for survey work because the sample size can easily be verified. However, the syringe needle is more likely to clog with pieces of septum material when gas samples are injected than when liquids are injected. If no chromatographic peaks are observed, test the syringe by injecting air into a liquid. If no bubbles are seen, unclog the needle with a wire or by filling the syringe without the needle and forcing solvent through the needle. However, repeatable results in gas analysis will only be obtained using gas sample valves that have a fixed sample loop. The latter valves can easily be automated.

10.3.1.7 A sealed, friable, or puncturable ampule containing a weighed amount of sample may be placed in the injection chamber. The ampule is physically broken to release the sample, which is then swept into the gas chromatographic column by the carrier gas.

TABLE 3 n-Alkanes Used for Column Evaluation at 200°C

1 m 10 % Dimethyl silicone C_{18}, C_{20}, C_{22}
3 m 10 % Dimethyl silicone C_{14}, C_{16}, C_{18}
1 m 10 % Phenylmethyl silicone C_{20}, C_{22}, C_{24}
3 m 10 % Phenylmethyl silicone C_{16}, C_{18}, C_{20}
1 m 10 % Cyanopropyl silicone C_{24}, C_{26}, C_{28}
3 m 10 % Cyanopropyl silicone C_{20}, C_{22}, C_{24}
1 m 10 % Polyethylene glycol C_{24}, C_{26}, C_{28}
3 m 10 % Polyethylene glycol C_{18}, C_{20}, C_{22}

10.4 *Sample Container*—Care must be taken in the design and construction of sample containers so that none of the components of interest are in any way changed or removed from the sample by reaction, diffusion, or adsorption. Stopcock grease and material desorbed from plastic cap liners are frequent sample contaminants.

11. Evaluation of Column Performance

11.1 Make a test mixture which contains a normal aliphatic hydrocarbon and the compound being analyzed, or one similar in structure. The aliphatic hydrocarbon peak should appear in the chromatogram near the second component in the test mixture. (Suggested aliphatic hydrocarbon mixtures are shown in Table 3.) The peaks of both components should be about the same size. The analytical conditions should be chosen so that both chromatographic peaks will appear about 4 to 6 times the retention time (distance) of the solvent peak. (If a selective detector is being used, choose a compound that will give a response to that detector.)

11.2 The different methods of determining column efficiency are shown in Equations 1, 2, and 3. Fig. 3 shows the measurements made on a chromatographic peak which are used to determine column efficiency.

$$N = 16\,(t_R/w_b)^2 \tag{1}$$

$$N = 5.54\,(t_R/w_h)2 \tag{2}$$

$$H = L/N \tag{3}$$

where:

N = number of theoretical plates,

t_R = retention time, or distance, measured in mm,

w_b = width of the peak at base, measured in mm, (Determined by extrapolating, as shown in Figs. 3a and 3b.)

w_h = width of the peak at one-half the peak height, h, all measured in mm, (See Note 6.)

H = height equivalent to a theoretical plate, HETP, and

L = length of the column in millimetres, or in centimetres.

NOTE 6—The peak width may be measured to the nearest 0.1 mm by a magnifying loupe fitted with a scale graduated in 0.1 mm increments. The peak to be so measured should be at least 10 mm wide; this is arranged by choosing an adequately large chart speed.

11.3 Equation 1 is often used. However, it involves an extrapolation of the baseline, shown in Fig. 3, which can be in error. The use of Eq 2 is preferred because the term, w_h can be determined directly from the chromatogram without extrapolation. However, the width of the recorder pen line can be variable which can lead to difficulties in determining the true value of the peak width. Use a pen that writes with a sharp, thin line. The peak width should be measured from the leading edge of one line to the leading edge of the other line, as shown in Fig. 3a. For accuracy, determine the peak width twice (see Fig. 3a), and use the average value to calculate "N". To further minimize errors in determining the

TABLE 4 General Optimum Values of Carrier Gas Flow Rates

Column Dimensions	General Optimum Carrier Gas Flow	
	Helium	Nitrogen
⅛ in. OD or 2 mm ID	30 mL/min	20 mL/min
¼ in. OD or 4 mm ID	60 mL/min	40 mL/min

(3a)

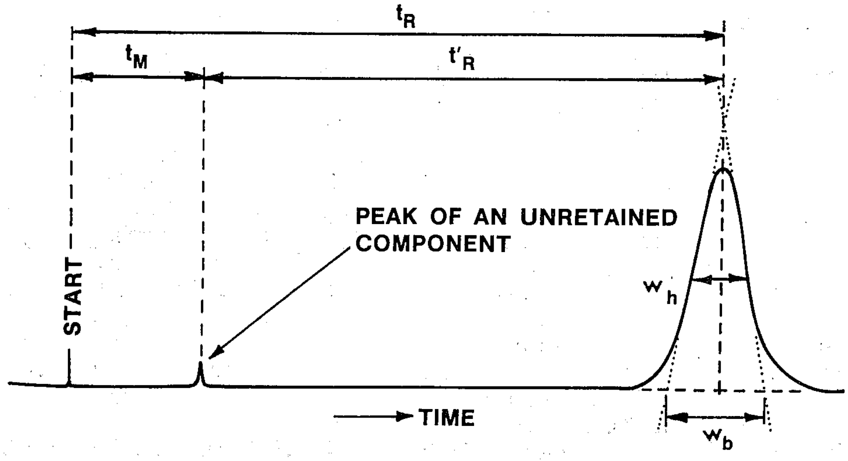

FIG. 3a Use of the Chromatogram to Calculate Column Efficiency

(3b)

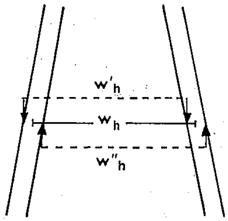

FIG. 3b Shows the Procedure to Measure the Peak Width, at w_h, to Account for the Thickness of the Pen. Use the Average of the Two Determinations, w'_h and w''_h, shown here

peak width, use a recorder chart speed that will give a minimum peak width of 4 to 5 cm. The peak height should be about 40 to 80% full-scale chart deflection. This can be achieved by either changing the detector sensitivity, or changing the sample size. However, be sure that the sample size does not exceed the capacity of the column.

11.4 The optimum efficiency occurs at the optimum carrier gas flow which can be determined by plotting the efficiency of the hydrocarbon peak versus carrier gas flow. General optimum values of the carrier gas flow rates are shown in Table 4, but should be determined for the particular column.

11.5 Calculate the efficiency of the second component. The results should be about the same for both components. If the efficiency of the second component is about 25 % lower than that of the hydrocarbon, the column might not be the best choice for analyzing the compound(s) of interest. A strong indication of nonsuitability is shown by comparatively greater tailing of the nonhydrocarbon component. Tailing can indicate strong interaction with the column packing (phase or support, or both), column tubing, or adsorption in the injection port or in lines leading from the column to the detector.

11.6 Typical efficiencies are shown in Table 5. Not all gas chromatographic packings are capable of excellent efficiencies. For example, porous polymers, Teflon supports, and some liquid phases, such as trifluoropropyl silicone can give efficiencies less than 500 plates per foot.

TABLE 5 Typical Efficiencies and Ratings of Gas Chromatographic Columns

Plates/ft	Plates/metre	HETP	Rating
500	1640	0.6 mm	Excellent
400–500	1300–1640	0.60–0.76 mm	Good
300–400	980–1300	0.76–1.0 mm	Fair
300	980	1.0	Poor

12. Use of the Gas Chromatographic Packed Column

12.1 Certain precautions and preventive maintenance are necessary to obtain the best column performance. Some of these points have been made before and will be referred to in this section. Further precautions will also be discussed.

12.2 *Carrier Gas Purity*—Trace, or adventitious, oxygen and water in the carrier gas can produce degradation of liquid phases on the support. Purification of the carrier gas and necessary precautions are discussed in 8.6.

12.3 *The Injection Port*—The injection port is often a source of trouble. The temperature can be either too low or too high. See 6.3. Another problem is a leaking septum. See 6.3.4.

12.4 *Column Care:*

12.4.1 Never heat a gas chromatographic column with air in it. When any column is first placed in a gas chromatograph, flush any air (oxygen) out of it by flushing with carrier gas at normal flow rates for 15 to 30 min. at ambient temperatures. Then heat the column to the desired operating temperature. Always cool the column to room temperature before removing it from the gas chromatograph. If the column is to be used again, cap the ends with metal caps to prevent diffusion of air (oxygen) into the column during storage. Contact of the stationary phase with oxygen when hot or for prolonged periods of time at room temperature cause degradation of the stationary phase. This is particularly the case with polyglycols and polyester type phases and to a lesser extent cyanosilicones. Other phases are affected to a varying degree.

12.4.2 After long periods of use, column performance may degrade as shown by peak broadening, tailing, or gradual merging of adjacent peaks. Often the problem lies in the front end of the column. The injection port temperature might have been too high and destroyed the initial section of the liquid phase on the column packing. Residues or decomposition products might have built up on the glass wool plug. These problems can be remedied by repacking the first few inches of a glass column, or cutting off the first few inches of a metal column. Use fresh silanized glass wool to close the end of the column in both cases.

13. Methods of Qualitative Analysis

13.1 Identification of compounds by gas chromatography alone cannot be absolute, and the results must be considered with care. Elution of a compound is dependent upon carrier gas flow rate, column temperature, support size, amount and type of liquid phase, column dimensions, instrument dead volume, and column pressure drop. These parameters must be stable to obtain reproducible results. The recommended format for a gas chromatographic method is given in Section 15.

13.2 Tentative identification of a compound can be made by comparing its adjusted retention time against those of known standards using exactly the same chromatographic parameters.

13.2.1 The retention time is the time interval measured from the point of injection to maximum peak height of the sample. Adjusted retention time, t'_R, is derived by subtracting the time required for an unabsorbed gas, like air, or methane, t_M, to traverse the column (also called the gas holdup time) from the retention time, t_R.

NOTE 7—On some solid adsorbent columns, such as molecular sieves, there is no nonadsorbed component.

$$t'_R = t_R - t_M \tag{4}$$

13.2.2 Retention times are affected by all chromatographic parameters. As a result, direct comparison of retention times of the same components on different instruments or between laboratories should be done with caution. Use of relative retention time is an easy practical technique for providing elution data. The retention of a component is expressed relative to the retention of a known reference standard. The reference standard should possess structural or chemical similarity to the compounds being analyzed.

13.3 The retention of a given weight of compound is usually independent of its concentration if the compound does not overload the column producing skewed peaks. The retention of the compound is also independent of other substances present if there is no appreciable overlap with another compound. Substances that exhibit positive, or Langmuir-type, skewing (tailing) during elution will produce a decrease in retention as the concentration increases; while negative, or anti-Langmuir-type fronting will produce an increase in retention time with increased concentration.

13.3.1 The logarithm of the retention time of members of a homologous series run isothermally is usually a linear function of the number of carbon atoms of a molecule. Using this characteristic, two or three reference compounds can provide sufficient information to prepare a plot of the logarithm of the retention time versus carbon number, and they can identify other members of the series. Retention time, adjusted or not, is of little value in comparing the results from various instruments. The use of Kovats retention index, based on the relative retention of a compound to the retention of normal paraffins, provides a more reliable means of comparing the results obtained from different instruments (see Practice E 355).

13.4 Absolute compound identification or characterization, must be made with ancillary techniques such as mass or infrared spectrometry, nuclear magnetic resonance, chemical analysis of the effluent, or spot tests for functional groups.

13.4.1 The samples for the analyses in 13.1 through 13.4 may be obtained by trapping components as they emerge from the chromatograph. A trap, glass capillary, or U-tube, is cooled with ice or dry ice, and placed in the effluent stream of the column. Several collections may be required to obtain a sufficiently large sample.

13.4.2 The collection of effluent is easiest with nondestructive detectors, see 6.7. In the case of destructive detectors, a split is made for the collection just before the detector.

13.4.3 Apparatus is also available so that the effluent from the gas chromatographic column can be analyzed directly by mass spectrometers or infrared spectrophotometers.

14. Methods of Quantitative Analysis

14.1 Gas chromatography can be used to determine quantitatively the composition of complex samples. There are several factors that must be considered before the sample is analyzed. The recommended format for gas chromatographic methods is given in Section 15.

14.1.1 *The Chemistry of the Sample*—The chemistry of the sample, if known, allows a chromatographer to select more accurately a column compatible with the sample and to anticipate potential interferences from reaction byproducts.

14.1.2 *The Choice of a Detector*—A detector must be chosen with the needed selectivity and sensitivity. If components will be analyzed at low levels, an electrolytic conductivity electron capture, nitrogen phosphorus, microcoulometric, ionization, or flame photometric detector should be selected. The detector may be limited to these lower concentrations and not applicable to high concentrations.

14.1.3 *Initial Separation of Components*—Next, a column must be chosen that will resolve the components of interest in the sample within a reasonable amount of time. First, a rough separation should be achieved with known standards. Next, actual samples should be analyzed to determine if there are any interferences. A second column, or an ancillary technique (GC/mass spectrometry, GC/infrared spectrometry, etc., should be used to verify that additional components are not eluting with the component of interest. Each new sample adds the possibility of an interference eluting with the component of interest; therefore this should be checked often. If an interference is detected, the chromatographer must change the method to remove it. The several options for doing this are as follows:

14.1.3.1 Select a column stationary phase with a greater selectivity for either the interference or the component of interest.

14.1.3.2 Choose a different type of detector that would detect the component of interest but not the interference. Examples would be water not being detected by a flame-ionization detector, or hydrocarbons not detected by an electrolytic conductivity or electron capture detector.

14.1.3.3 Consider other types of chromatographic separation such as capillary gas chromatography for more efficient separation of volatile compounds, liquid chromatography for separation of non-volatile compounds, or another appropriate separation technique.

14.1.4 *Detector Sensitivity and Linearity*—Once the chromatographic separation has been optimized, the detector can be optimized and calibrated. Gas flows should be adjusted to the optimum levels to get peak sensitivity at the concentration range of the components of interest. The detector must also be clean and leak-tight. (See the manufacturer's manual for suggested procedures.)

14.1.4.1 The linearity of the detector over the desired concentration range of the component(s) of interest is determined using prepared standards. This step will determine what the response is to increasing amounts of component. The peak area or height should be plotted versus the concentration for about five concentrations near the expected sample concentration. There should be a linear correlation. Nonlinearity may be caused by reactivity, adsorptivity, thermal sensitivity, or excessive column bleed. If the latter is the cause, change to a more thermally stable column or one of different polarity. Column reactivity can be characterized by skewed, misshaped peaks. This can be corrected by installing a fresh column of the same type that does not have reactive sites. Test mixtures can be used to demonstrate nonreactivity. Other sources of adsorptivity or reactivity with the sample are the injection port, connecting lines to the detector, or glass wool. Each of these sources can be detected by carefully troubleshooting the system.

14.1.4.2 The detector performance should be checked periodically throughout the analysis. This can be done by injecting one of the linearity standards and comparing it to the linearity plot.

14.1.5 *Peak Area or Height Measurement*—Many types of peak area and height measurement techniques exist. The oldest methods for calculating the peak area are manual measurement with a ruler of the peak area using one of the following equations:

$$\text{peak area} = w_h \times h \tag{5}$$

where:

w_h = peak width at half height, and

h = peak height

or

$$\text{peak area} = \tfrac{1}{2}\, w_b \times h \tag{6}$$

where:

w_b = peak width at the base of the peak, and

h = peak height.

Another precise measurement defines the peak area as retention distance (in millimetres) times the peak height (also in millimetres). For peak height, this distance is simply measured from the baseline to the apex of the peak. However, these techniques now, for the most part, have been replaced by electronic integration, which is much faster. The proper use of these devices is crucial for accurate quantitative analysis. The instruction manual for the particular integrator should be studied and understood thoroughly before attempting to use electronic integration for peak area or peak height measurement.

14.1.6 *Data Handling:*

14.1.6.1 All manufacturers supply an integral electrometer to allow the small electrical current changes to be coupled to recorders/integrators/computers. The preferred system will incorporate one of the newer integrators or computers that converts an electrical signal into clearly defined peak area counts in units such as microvolt-seconds. These data can then be readily used to calculate the linear range.

14.1.6.2 Another method uses peak height measurements. This method yields data that are very dependent on column performance and, therefore, not recommended.

14.1.6.3 Regardless of which method is used to calculate linear range, peak height is the only acceptable method for determining minimum detectability.

14.1.7 *Calibration*—It is essential to calibrate the measuring system to ensure that the nominal specifications are acceptable and particularly to verify the range over which the output of the device, whether peak area or peak height, is linear with respect to input signal. Failure to perform this calibration may introduce substantial errors into the results. Methods for calibration will vary for different manufacturers' devices but may include accurate constant voltage supplies or pulse generating equipment. The instruction manual should be studied and thoroughly understood before attempting to use electronic integration for peak area or peak height measurements.

14.2 *Types of Calculations:*

14.2.1 Each method of quantitative analysis has advantages and disadvantages. The four methods of quantitative analysis are as follows:

14.2.1.1 Internal standardization,

14.2.1.2 External standardization,

14.2.1.3 Normalization, and

14.2.1.4 Corrected area.

14.2.2 *Internal Standardization*—In this technique, a pure component (the internal standard) is added to a sample in a known amount. The peak area, or height, of all components of interest is compared to the peak area, or height, of the internal standard. These comparisons are referred to as response factors:

$$R_F = A_C/A_{IS} \times W_{IS}/W_C \tag{7}$$

where:

R_F = response factor,

A_C = peak area of component,

A_{IS} = peak area of internal standard,

W_{IS} = mass of internal standard, and

W_C = mass of component.

The amount of the component can be calculated from the weights of the sample and internal standard, the response factor, and the peak areas (or heights) as follows:

$$\%\ \text{Conc}_C = W_{IS}/W_S \times A_C/A_{IS} \times 1/R_F \times 100\ \% \tag{8}$$

where:

Conc_C = concentration of component in sample,

W_{IS} = mass internal standard,

W_S = mass sample,

A_C = peak area of component,

A_{IS} = peak area of internal standard, and

R_F = response factor.

This technique provides a correction for the relatively high variability of syringe injection and, therefore, yields a more precise method of analysis. Neither the quantity of solution injected, nor change in detector response, will alter the area ratio of the analyte and the internal standard. To achieve optimum performance, the internal standard must meet the following criteria.

14.2.2.1 The internal standard must elute in an area of the chromatogram that is free of sample components, or possible sample components.

14.2.2.2 The internal standard must not react with the sample or any of its components.

14.2.2.3 The internal standard and the sample must be homogeneous. A cosolvent may be used to produce a homogeneous mixture.

14.2.2.4 The internal standard must be easily and accurately added.

14.2.2.5 The internal standard must be pure.

14.2.2.6 The internal standard should elute near the component of interest.

14.2.2.7 The concentration of the internal standard, relative to that of the analyte, should be such that these two peaks are within 50 to 100 % of full scale deflection with the same electronic attenuation and sensitivity setting in order to allow manual measurements and calculations of parameters, if desired.

14.2.2.8 The most common use for the internal standard technique in chromatography is to correct for quantitative variations in the injection, particularly when using syringes. For this purpose, the internal standard need not be chemically related to the analyte, but must possess the criteria cited above and may be added in the final solution.

14.2.2.9 In certain applications, an internal standard with functional groups similar to the analyte may be desirable. For instance, those with a labile proton can be expected to exhibit similar adsorption isotherm behavior and to undergo similar physico-chemical transformations during such processes, as extraction from a complex matrix or derivatization, or both. Likewise, similar electronegative functional groups are likely to behave similarly towards an electron capture detector.

14.2.3 *External Standardization:*

14.2.3.1 This method compares peak areas or heights of components in a sample chromatogram to those in a standard solution injected separately. It is critical that accurate amounts of sample and standard be injected for the method to be valid. Generally, the solvent flush injection technique (see 10.3.1.2) or a sample valve of fixed volume is preferred.

14.2.3.2 The advantages of this method are as follows:

(*a*) Nondetected components do not bias the results.

(*b*) It can be used where several known components must be determined in a very complex sample.

(*c*) It can quantitate relatively reactive components.

(*d*) A single sample can be analyzed where maximum accuracy is not required.

(*e*) Nonlinearity has a minimal effect if the external standard is near the concentration of the sample.

14.2.3.3 The critical part of this method is the injection. The volume of sample in the injection syringe and standard must be accurately measured, allowing no bubbles in the slug of sample or standard solution. If the sample and standard have different densities, a correction must be made. Densities are easily determined by filling a 50-µL syringe to about 30 µL, wiping the needle, weighing it, expelling the sample, wiping the needle again, and reweighing it.

14.2.3.4 The peak areas or heights of the component in the sample and the standard compound are measured and the concentration calculated as follows:

$$\%\ \text{Conc}_C = A_C/A_{ES} \times W_{ES}/W_S \times \%\ \text{Conc}_{ES} \tag{9}$$

where:
Conc_C = concentration of component,
A_C = peak area of component in sample,
A_{ES} = peak area of external standard,
W_{ES} = mass of external standard injected,
W_S = mass of sample injected, and
Conc_{ES} = concentration of external standard in solution.

14.2.4 *Normalization*—This calculation assumes that every component elutes and that each has similar response factors. It is a fast procedure that requires no weighing. The sample is injected, and the peak areas or heights of all components are measured. The concentration of the component of interest is calculated as follows:

$$\%\ \text{Conc}_C = A_C/A_{\text{ALL}} \times 100 \tag{10}$$

where:
Conc_C = concentration of component of interest,
A_C = area of component, and
A_{ALL} = sum of areas of all components.

Severe errors result if the components have different response factors or do not all elute.

14.2.5 *Corrected Area*—This method corrects for differences in response but still assumes that all components elute and are observed by the detector. Response factors are used to correct for response differences as follows:

$$\%\ \text{Conc}_C = A_C/(A \times R_F)_{\text{ALL}} \times R_{F_C} \times 100\ \% \tag{11}$$

where:
Conc_C = concentration of component of interest,
A_C = peak area of component,
$(A \times R_F)_{\text{ALL}}$ = sum of peak areas times their respective response factors relative to a standard, and
R_{F_C} = response factor of component to the same standard.

15. Recommended Form for Writing Gas Chromatographic Methods

15.1 *General*—Not all of the steps outlined in this section may be needed to describe adequately a method. A number of variations in procedure format are shown in the publication, ASTM Standards in Chromatography.[7] Ideally, the procedure should be written so that it can be followed by a person with the equivalent of a high school-chemistry understanding or six to twelve months of practical laboratory experience. Critical steps should be identified along with any reasons that show why this step is necessary to achieve a successful analysis. Any involved procedures should be written in an Appendix so that the main points in the procedure can be read more easily.

15.2 *Recommended Form:*

15.2.1 *Title*—The title should be concise, but complete

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[7] *ASTM Standards on Chromatography*, ASTM, 1981.

enough to identify the component(s) analyzed, the nature of the method (gas chromatography), the detector, and the materials to which it is applicable. Select words that easily lend themselves to indexing.

15.2.2 *Scope*—State as clearly as possible the range of application of method. In a separate paragraph, note interferring substances or any significant limitations of the method. This material could be placed in a later section (15.2.5), if an involved description is necessary.

15.2.3 *Pertinent Documents or References:*

15.2.3.1 *ASTM Standards.*

15.2.3.2 *Other Standard Methods*—Include any standard methods.

15.2.4 *Summary of the Method*—Describe the method in a general way, without going into details of the procedure. It may be appropriate to touch briefly on the following points: sample introduction technique, column dimensions and type of tubing material, nature of the packing material, mesh size of support or adsorbent, liquid phase loading (if a liquid phase was used), isothermal or programmed temperature and detector type (thermal conductivity, flame ionization, electron capture, *etc.*).

15.2.5 *Significance and Application*—Use this section for a more detailed discussion than can be fitted in the Scope.

15.2.6 *Definitions*—Include special definitions in this section. General chromatographic definitions are already available in Practice E 355, to which reference can be made.

15.2.7 *Interferences*—Use this section for a more detailed discussion than can be fitted into the Scope.

15.2.8 *Special Comments*—Use this section to include a description of special requirements needed to achieve a successful analysis.

15.2.9 *Safety Precautions*—If the method involves hazards, insert a warning to this effect. Point out the nature of the hazards, and describe precautionary measures which must be taken. Refer to the latest OSHA regulations regarding all materials used in this procedure.

15.2.10 *Gas Chromatographic System*—List and describe the apparatus. Describe the essential features of the apparatus that are necessary to achieve the desired analysis. Avoid the use of trade names. Include schematic drawings or photographs if they are needed to clarify or supplement the text. The gas chromatographic conditions can be either summarized in a table, as in Table 6, or in the text as follows:

15.2.10.1 *Sample Injection Port*—Construction: stainless steel, glass liner, fitted for on-column injection with a glass or metal column, etc. Temperature at which used.

15.2.10.2 *Column Oven*—Isothermal or temperature programmed operation: give temperatures and programming rates required.

15.2.10.3 *Detector*—Type (flame ionization, thermal conductivity, etc.), temperature of operation, sensitivity required. Detector gases used and flow rates.

15.2.10.4 *Recorder*—Operating range (in millivolts), chart speed, time for full-scale deflection of pen.

15.2.10.5 *Integrator*—Note operating characteristics of integrator and parameters used.

15.2.11 *Preparation and Installation of the Chromatographic Column:*

15.2.11.1 *Tubing Material*—Note the type of material, as stainless steel, nickel, glass, or glass-lined tubing, as well as the dimensions (outer diameter and inner diameter, or wall thickness and length). Any pretreatment of the column material, solvent washing, or silanization should be mentioned.

15.2.11.2 *Partitioning Phase*—Solid adsorbent, if used (type and mesh size). Coated support if used (liquid phase, percent loading and coating procedure, support type and pretreatment, and mesh size). Note sources for special materials in footnotes. Provide preparation and purification method for materials not commercially available. In an Appendix, note other liquid phases that have been successfully used in this analysis.

15.2.11.3 *Column Preparation*—Describe the procedure used to pack the column. Note the amount of packing in the column.

15.2.11.4 *Column Installation*—Note if the column is set up for back-flushing, if a sample fraction is removed on a pre-column, or other special column arrangement.

15.2.11.5 *Column Conditioning*—Provide the column conditioning procedure.

15.2.11.6 *Column Evaluation*—Give the procedure for evaluating the column. Calculation of the resolution between two components in a standard mixture will often be sufficient. Provide some estimate of column life and signs of degrading column performance (loss of resolution, peak broadening, or tailing). Provide examples of good and bad chromatograms.

15.2.12 *General Apparatus*—Volumetric flasks and pipets, microsyringes for sample introduction, balance (capacity and sensitivity), heat lamps, hot plates, etc.

15.2.13 *Reagents and Materials:*

15.2.13.1 *Chemicals and Reagents*—Include derivatizing reagents. Note purity, or purification methods, if required.

15.2.13.2 *Calibration Standards*—Note purity required.

15.2.13.3 *Gas* (*or Gases*)—Carrier gas, fuel gases for flame ionization detector, special gas for electron capture detector, etc. Note purity required.

15.2.14 *Calibration*—Describe in detail the calibration procedure. State whether pure components or standard mixtures are used and the basis of measurement. Include equations and describe the preparation of any calibration charts. Show the calibration curve. If a trace method is described, provide a chromatogram of the lowest detectable amount. Lengthy procedures, such as the development of complex equations, or the preparation of standard mixtures, should be placed in a section of an Appendix.

15.2.15 *Procedure*—Include, in proper sequence, directions for carrying out the method. Refer to the pertinent parts of the calibration procedure in 15.2.14. Do not repeat these steps here. Possible subheadings are as follows:

15.2.15.1 *Final Conditioning and Adjustment of the Gas Chromatographic System*—This section is intended to include adjustment and verification of the state of the chromatographic system before analytical use.

15.2.15.2 *Sampling*—Careful attention must be given to the sampling techniques since representative samples are essential to achieve successful analysis. Include special directions that may be required for taking samples, for preservation of samples, and for special treatment of samples prior to injection.

TABLE 6 Summary of Gas Chromatographic Conditions

Column	Length, outside diameter, and internal diameter (or outside diameter and wall thickness); weight percent of specified stationary phase; specified solid support (mesh size and treatment; for example, acid-washed, silanized); approximate amount of total column packing in a given column length.
Temperatures:	
Sample inlet system	°C
Detector	°C
Column	
If isothermal	°C
If programmed	
Initial	°C (note any hold time at initial temperature)
Final	°C
Heating rate	°C/min
(State specific temperatures and times if isothermal operation and temperature programming are combined)	
Carrier gas	(specify)
Flow rate	cm^3/min^A
Detector	TCD, FID, ECD
Detector Sensitivity	Flame ionization detector—amps full scale deflection, AFS^B Detector voltage or bridge current, if applicable.
Recorder characteristics	mV range, speed of full-scale deflection
Chart speed	cm (or in.)/min
Sample size	µL (or cm^3, if gas)

[A] Flow rate is best measured at the column or detector outlet, at the analytical temperature and flow rate.

[B] A flame ionization detector is assumed to be operated under optimum hydrogen and air flow rates, unless otherwise specified.

15.2.15.3 The remainder of the steps leading to the chromatogram.

15.2.15.4 *Typical Chromatogram*—Show, in a figure, a plot of the retention time (in minutes) versus the detector response. Label the known peaks (including the dead volume or unadsorbed gas peak) and indicate in parentheses the attenuation for each peak.

NOTE 8—When determining the retention time of the unadsorbed peak, the retention time of air is used for thermal conductivity detectors, methane for flame ionization detectors, and methylene chloride lead space vapors for ECDs.

15.2.15.5 *Retention Time Data*—Include a table listing retention times and relative retention times for all compounds of interest, for all recommended columns. Identify the unadsorbed peak and the reference material used for relative retention time calculations.

15.2.16 *Calculation*—State the reference point on which the calculations are based (for example, sample as received), the terms in which the results are finally obtained (weight, volume, or mole percent), and whether or not these values are normalized. Present the calculations in equation form, using letter symbols to designate variable values and numerical values of constants. Define the letter symbols in a legend immediately following the calculation equation.

15.2.17 *Report*—Show limits to be reported.

15.2.18 *Precision*—Limiting values for precision should be based on cooperative test results. Judgment as to the acceptability of results (95 % probability) should be based on the following criteria.

15.2.18.1 *Repeatability*—The following wording should be used: Duplicate results by the same operator should not be considered suspect unless they differ by more than the following amounts: (insert determined limits in tabular form).

15.2.18.2 *Reproducibility*—The following wording should be used: The result submitted by each of two laboratories should not be considered suspect unless the two results differ by more than the following amounts: (insert determined amounts in tabular form).

15.2.19 *Appendixes*—Supplementary information may be included in one or more Appendixes to the report. Examples of such information are: technique to improve column life, directions to clean the apparatus, leak check procedures, procedures to optimize column performance, development of equations used in the calculations, and precautions to avoid common causes of errors, etc.

16. Keywords

16.1 gas chromatography; GC; packed columns

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

Document Name: ASTM E283: Standard Test Method for Determining the Rate of Air Leakage Through Exterior Windows, Curtain Walls, and Doors

CFR Section(s): 10 CFR 434.402.2

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:

THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: E 283 – 91 (Reapproved 1999)

An American National Standard

Standard Test Method for Determining Rate of Air Leakage Through Exterior Windows, Curtain Walls, and Doors Under Specified Pressure Differences Across the Specimen

This standard is issued under the fixed designation E 283; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method provides a standard laboratory procedure for determining the air leakage rates of exterior windows, curtain walls, and doors under specified differential pressure conditions across the specimen. The test method described is for tests with constant temperature and humidity across the specimen.[1]

1.2 This laboratory procedure is applicable to exterior windows, curtain walls, and doors and is intended to measure only such leakage associated with the assembly and not the installation. The test method can be adapted for the latter purpose.

NOTE 1—Performing tests at non-ambient conditions or with a temperature differential across the specimen may affect the air leakage rate. This is not addressed by this test method.

1.3 This test method is intended for laboratory use. Persons interested in performing field air leakage tests on installed units should reference Method E 783.

1.4 Persons using this procedure should be knowledgeable in the areas of fluid mechanics, instrumentation practices, and shall have a general understanding of fenestration products and components.

1.5 Throughout this test method, SI units are listed first in accordance with E-6 metric policy, and shall be considered the primary units. Non-SI units are provided in parenthesis.

1.6 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statement see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*

E 631 Terminology of Building Constructions[2]

E 783 Test Method for Field Measurement of Air Leakage Through Installed Exterior Windows and Doors[2]

3. Terminology

3.1 *Definitions*—Terms used in this standard are defined in Terminology E 631.

3.2 *Descriptions of Terms Specific to This Standard:*

3.2.1 *air leakage rate* (q_A or q_L), $m^3/s \cdot m^2$ ($ft^3/min \cdot ft^2$), or $m^3/s \cdot m$ ($ft^3/min \cdot ft$)—the air leakage per unit of specimen area (A) or per unit length of operable crack perimeter (L).

3.2.2 *extraneous air leakage* (Q_e), m^3/s (ft^3/min)—the volume of air flowing per unit of time through the test chamber and test apparatus, exclusive of the air flowing through the test specimen, under a test pressure difference and test temperature difference, converted to standard conditions.

3.2.2.1 *Discussion*—Extraneous leakage is the sum of all leakage other than that intended to be measured by the test.

3.2.3 *specimen*—the entire assembled unit submitted for test as described in Section 7.

3.2.4 *specimen air leakage* (Q_s), m^3/s (ft^3/min)—the volume of air flowing per unit of time through the specimen under a test pressure difference and test temperature difference, converted to standard conditions.

3.2.5 *specimen area* (A), m^2 (ft^2)—the area determined by the overall dimensions of the frame that fits into the rough opening.

3.2.6 *standard test conditions*—in this test method, dry air at:

Pressure—101.3 kPa (29.92 in. Hg)
Temperature—20.8°C (69.4°F)
Air Density—1.202 kg/m^3(0.075 lbm/ft^3)

3.2.7 *test pressure differences, Pa (lbf/ft^2)*—the specified differential static air pressure across the specimen.

3.2.8 *total air flow* (Q_t), m^3/s (ft^3/min)—the volume of air flowing per unit of time through the test chamber and test apparatus, inclusive of the air flowing through the test specimen, under a test pressure difference and test temperature difference, converted to standard conditions.

3.2.9 *unit length of operable crack perimeter* (L), m (ft)—the sum of all perimeters of operable ventilators, sash, or doors contained in the test specimen, based on the overall dimensions of such parts. Where two such operable parts meet the two adjacent lengths of perimeter shall be counted as only one length.

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[1] This test method is under the jurisdiction of ASTM Committee E-6 on Performance of Buildings and is the direct responsibility of Subcommittee E06.51 on Component Performance of Windows, Curtain Walls, and Doors.

Current edition approved Aug. 15, 1991. Published October 1991. Originally published as E 283 – 65 T. Last previous edition E 283 – 84.

[2] *Annual Book of ASTM Standards*, Vol 04.11.

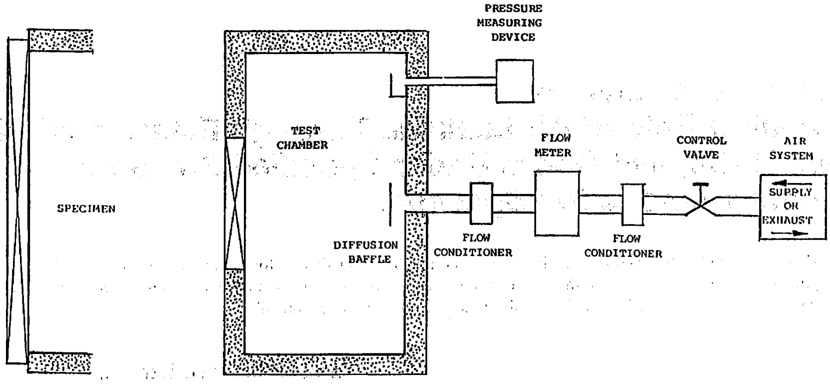

FIG. 1 General Arrangement of the Air Leakage Apparatus

4. Summary of Test Method

4.1 The test consists of sealing a test specimen into or against one face of an air chamber, supplying air to or exhausting air from the chamber at the rate required to maintain the specified test pressure difference across the specimen, and measuring the resultant air flow through the specimen.

5. Significance and Use

5.1 This test method is a standard procedure for determining the air leakage characteristics under specified air pressure differences at ambient conditions.

NOTE 2—The air pressure differences acting across a building envelope vary greatly. The factors affecting air pressure differences and the implications of the resulting air leakage relative to the environment within buildings are discussed in the literature.[3,4,5] These factors should be fully considered in specifying the test pressure differences to be used.

5.2 Rates of air leakage are sometimes used for comparison purposes. Such comparisons may not be valid unless the components being tested and compared are of essentially the same size, configuration, and design.

6. Apparatus

6.1 The description of the apparatus in this section is general in nature. Any suitable arrangement of equipment capable of maintaining the required test tolerances is permitted.

6.2 *Test Chamber*—A well sealed box, wall, or other apparatus into or against which the specimen is mounted and secured for testing. An air supply shall be provided to allow a positive or negative pressure differential to be applied across the specimen without significant extraneous losses. The chamber shall be capable of withstanding the differential test pressures that may be encountered in this procedure. At least one static air pressure tap shall be provided on each side of the specimen to measure the test pressure differences. The pressure tap shall be located in an area of the chamber in which pressure readings will not be affected by any supply air. The air supply opening to the chamber shall be located in an area in which it does not directly impinge upon the test specimen.

6.2.1 *Supply Air System*—A controllable blower, exhaust fan, or reversible blower designed to provide the required air flow at the specified test pressure difference. The system should provide essentially constant air flow at the specified test pressure difference for a time period sufficient to obtain readings of air flow.

6.2.2 *Pressure Measuring Apparatus*—A device to measure the differential test pressures to ± 2 % of setpoint or ± 2.5 Pa (± 0.01 in. of water column), whichever is greater.

6.2.3 *Air Flow Metering System*—A device to measure the air flow into the test chamber or through the test specimen. The air flow measurement error shall not exceed ± 5 % when the air flow equals or exceeds 9.44×10^{-4} m^3/s (2 ft^3/min) or ± 10 % when the air flow is less than $9.44^{-4} \times 10$ m^3/s (ft^3/min).

NOTE 3—At lower flows a greater percentage of errors will be acceptable. If higher precision is required, special flow metering techniques are necessary. The accuracy of the specimen air leakage flow measurement is affected by the accuracy of the flowmeter and the amount of extraneous air leakage. (See Annex A1.)

7. Hazards

7.1 **Precaution**—Glass breakage may occur at the test pressure differences applied in this test. Adequate precautions should be taken to protect personnel.

8. Test Specimen

8.1 The test specimen for a wall shall be of sufficient size to determine the performance of all typical parts of the wall system. For curtain walls or walls constructed with prefabricated units, the specimen width shall be not less than two typical units plus the connections and supporting elements at both sides, and sufficient to provide full loading on at least one typical vertical joint or framing member, or both. The height shall be not less than the full building story height or the height of the unit, whichever is greater, and shall include at least on full horizontal joint, accommodating vertical expansion, such joint being at or near the bottom of the specimen, as well as all connections at top and bottom of the units.

8.1.1 All parts of the wall test specimen shall be full size

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[3] Available from American Society of Heating, Refrigeration, and Air-Conditioning Engineers, 1791 Tullie Circle N.E., Atlanta, GA 30329. ASHRAE Handbook of Fundamentals, 1989.

[4] Fluid Meters—Their Theory and Application, 5th Edition, 1959.

[5] Available from American Society of Mechanical Engineers, 345 E. 47th St., New York, NY 10017. Power Test Code, 2nd Edition, 1956, Part 5, Chapter 4, "Flow Measurements."

using the same materials, details, and methods of construction and anchorage as used on the actual building.

8.1.2 Conditions of structural support shall be simulated as accurately as possible.

8.2 The test specimen for a window, door, or other component shall consist of the entire assembled unit, including frame and anchorage as supplied by the manufacturer for installation in the building. If only one specimen is to be tested the selection shall be determined by the specifying authority.

NOTE 4—The air leakage rate is likely to be a function of size and geometry of the specimen.

9. Calibration

9.1 Specific procedures for calibration of the total air flow measurement system are being developed in a separate ASTM document. When complete, that document will be referenced. However, all test apparatus shall be calibrated at a minimum of every 6 months to the tolerances established in Section 6. The procedures for this calibration are, at this time, the responsibility of the testing agency. Calibration should be conducted at or near the environmental conditions (temperature, relative humidity, and so forth) under which the tests are to be conducted and to which the test apparatus is to be exposed.

10. Test Conditions

10.1 The specifying authority shall supply the following information:

10.1.1 Specimen test size,

10.1.2 Test pressure difference (if no value is designated, 75 Pa (1.57 lb/ft^2)), and

10.1.3 Direction of air flow, exfiltration or infiltration. (If none is specified, the test shall be infiltration.)

10.2 *Air Leakage Rate*—Basis for reporting air leakage rate shall be total air leakage m^3/h (ft^3/min), per unit length of operable crack perimeter, m^3/h – m (ft^3/min·ft), and per unit area of outside frame dimension, m^3/h – m^2 (ft^3/min·ft ^{2}).

11. Procedure

11.1 Remove any sealing material or construction that is not normally a part of the assembly as installed in or on a building. Fit the specimen into or against the chamber opening. Installation should be such that no parts or openings of the specimen are obstructed.

NOTE 5—Nonhardening mastic compounds or pressure sensitive tape can be used effectively to seal the test specimen to the chamber, and to achieve air tightness in the construction of the chamber. These materials can also be used to seal a separate mounting panel to the chamber. Rubber gaskets with clamping devices may also be used for this purpose, provided that the gasket is highly flexible and has a narrow contact edge.

11.2 Without disturbing the seal between the specimen and the test chamber, adjust all hardware, ventilators, balances, sash, doors, and other components included as an integral part of the specimen so that their operation conforms to test method requirements.

11.3 To ensure proper alignment and weather seal compression, fully open, close, and lock each ventilator, sash, or door five times prior to testing.

11.4 Adjust the air flow through the test chamber to provide the specified test pressure difference across the test specimen. When the test conditions are stabilized, record the air flow through the flowmeter and the test pressure difference. This measured air flow is designated the total air flow, Q_t. Measure the barometric pressure, and temperature of the air at the test specimen.

11.5 Eliminate extraneous chamber leakage, or, if this is impractical, measure the amount of such leakage with the specimen sealed, at the air pressure differences to be exerted during the air leakage tests. Designate this measured air flow as the extraneous air flow, Q_e.

12. Calculation

12.1 Express the total air flow, Q_t, and the extraneous leakage, Q_e, in terms of flow at standard conditions, Q_{st}, using the Eq 1 and 2.

$$Q_{st} = Q(W/W_s)^{ln\$} \quad (1)$$

$$W = 3.485 \times 10^{-3}\,(B/(T + 273)) \quad (2)$$

where:

Q = airflow at non-standard conditions,
Q_{st} = airflow corrected to standard conditions,
W_s = density of air at reference standard conditions—1.202 kg/m^3 (0.075 lb/ft^3),
W = density of air at the test site, kg/m^3(lb/ft^3),
B = barometric pressure at test site corrected for temperature, Pa (in. Hg), and
T = temperature of air at flowmeter, °C (°F).

NOTE 6—Use the equation $W = 1.326\,(B/(T + 460))$ for calculating in inch-pound units.

12.2 Express the air leakage through the test specimen as follows:

$$Q_s = Q_t - Q_e \quad (3)$$

where:

Q_s = air leakage through the test specimen, m^3/s (ft^3/min), at standard conditions.

12.3 Calculate the rate of air leakage for the test specimen according to 12.3.1 and 12.3.2.

12.3.1 To calculate q_L rate of air leakage per unit of length of operable crack perimeter use Eq 4:

$$q_L = Q_s/L,\ \mathrm{m^3/h \cdot m}\ (\mathrm{ft^3/min \cdot ft}) \quad (4)$$

12.3.2 To calculate q_A rate of air leakage per unit area:

$$q_A = Q_s/A,\ \mathrm{m^3/h \cdot m^2}\ (\mathrm{ft^3/ min \cdot ft^2}) \quad (5)$$

13. Report

13.1 Report the following information:

13.1.1 *General*—Testing agency, date and time of test, and date of report.

13.1.2 *Sample Description*—Manufacturer, model, operation type, materials, and other pertinent information; description of the locking and operating mechanisms if applicable; glass thickness, type and method of glazing; weather seal dimensions, type, and material; and crack perimeter and specimen area.

13.1.3 *Drawings of Specimen*—Detailed drawings of the specimen showing dimensioned section profiles, sash or door dimensions and arrangement, framing location, panel arrangement, installation and spacing of anchorage, weatherstripping, locking arrangement, hardware, sealants, glazing details, and any other pertinent construction details. Any modifications made on the specimen to obtain the reported test values shall be noted.

13.1.4 *Test Parameters*—List or describe the specified test pressure difference(s), whether the tests were conducted for infiltration or exfiltration, and whether a positive or negative test pressure was used.

13.1.5 *Pressure Differences and Leakage*— A statement or tabulation of the pressure differentials exerted across the specimen during the test and the corresponding specimen air leakage (Q_s) and the two air leakage rates (q_L and q_A).

13.1.6 *Compliance Statement*—A statement that the tests were conducted in accordance with this test method, or a complete description of any deviation from this test method. When the tests are conducted to check for conformity of the specimen to a particular performance specification, the specification shall be identified.

13.2 If several identical specimens are tested, the results for each specimen shall be reported, each specimen being properly identified, particularly with respect to distinguishing features or differing adjustment. A separate drawing for each specimen shall not be required if all differences between the specimens are noted on the drawings provided.

14. Precision and Bias

14.1 The precision and bias of this test method has not been determined.

15. Keywords

15.1 air leakage; curtain walls; doors; fenestration; laboratory method; static pressure chamber; windows

ANNEX

(Mandatory Information)

A1. ERRORS IN WINDOW AIR LEAKAGE MEASUREMENT

A1.1 Terminology

A1.1.1 *Symbols*:

A1.1.1.1 Q_s = air flow through specimen.

A1.1.1.2 Q_{ts} = total air flow.

A1.1.1.3 Q_{es} = extraneous air flow.

A1.1.1.4 Δ = delta.

NOTE A1.1—Symbols A1.1.1.1-A1.1.1.4 have been converted to standard conditions.

A1.2 In the apparatus using a supply air system, $Q_s = Q_{ts} - Q_{es}$, the extraneous air leakage (Q_{es}) represents all the air leakage leaving the chamber which does not pass through the specimen proper. This includes leakage passing through the chamber walls and around the specimen mounting. When the mounting panel is used, leakage between the chamber and the panel contributes to extraneous leakage. The extraneous leakage flow is a function of the pressure difference between the chamber and the room, which is also the test specimen difference.

A1.3 The total error in the specimen flow determination (neglecting errors in the air density determination) is as follows:

$$\Delta Q_s/Q_s = [\Delta Q_{ts}/(Q_{ts} \cdot Q_{es})] \pm [\Delta Q_{es}/(Q_{ts} \cdot Q_{es})] \quad \text{(A1.1)}$$

A1.3.1 According to 6.2.3, the air flow through the test specimen is to be determined with an error no greater than $\Delta Q_s/Q_s = \pm 5$ % if the extraneous leakage is accurate to $\Delta Q_{es}/Q_{es} = \pm 10$ % and Q_{es} is 10 % of Q_s, then the contribution of the extraneous leakage to the overall error in Eq 2 is ± 1 %. (Note that the error attributed to the extraneous leakage determination is a function not only of the accuracy of the flow meter used in the determination, but also of the constancy of the leakage from the time of determination to the time of test.) The error contributed by the flow meter to the total error is then limited to 4 %, but because $Q_{ts} = Q_s + Q_{es} = 1.10\ Q_s$ the accuracy required of the flowmeter is:

$$\Delta\left(\frac{Q_{ts}}{Q_t}\right) = \frac{4}{1.1}\ \% = 3.6\ \% \quad \text{(A1.2)}$$

A1.3.2 It is seen that the major factor affecting the accuracy required of the flowmeter is the proportion of Q_{es} to Q_s. If $\Delta Q_{es}/Q_{es}$ remains at ±10 %, but Q_{ts} is 50 % of Q_s, the error contributed by the extraneous leakage becomes 5 % and no error can be tolerated in the flowmeter if the conditions of 6.2.5 are to be met—with Q_{es} in excess of 50 % it is impossible to achieve the required overall limit of error. Likewise, if the extraneous leakage is eliminated, the flowmeter error can be as great as 5 %.

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Document Name: ASTM E408: Standard Test Methods for Total Normal Emittance of Surfaces Using Inspection-Meter Techniques

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WASHINGTON, D.C.

Designation: E 408 – 71

Standard Methods of Test for
TOTAL NORMAL EMITTANCE OF SURFACES USING INSPECTION-METER TECHNIQUES[1]

This Standard is issued under the fixed designation E 408; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 These methods cover determination of the total normal emittance (Note 1) of surfaces by means of portable, inspection-meter instruments.

NOTE 1—Total normal emittance (ϵ_N) is defined as the ratio of the normal radiance of a specimen to that of a blackbody radiator at the same temperature. The equation relating ϵ_N to wavelength and spectral normal emittance [$\epsilon_N(\lambda)$] is

$$\epsilon_N = \int_0^\infty L_b(\lambda,T)\epsilon_N(\lambda)d\lambda / \int_0^\infty L_b(\lambda,T)d\lambda$$

where:

$L_b(\lambda,T)$ = Planck's blackbody radiation function = $c_1\pi^{-1}\lambda^{-5}(e^{c_2/\lambda T} - 1)^{-1}$,
c_1 = 3.7415×10^{-16} W·m^2,
c_2 = 1.4388×10^{-2} m·K,
T = absolute temperature, K,
λ = wavelength, m,
$\int_0^\infty L_b(\lambda,T)d\lambda$ = $\Delta\pi^{-1}T^4$, and
Δ = Stefan-Boltzmann constant = 5.66961×10^{-8} W·m^2·K^{-4}

1.2 The methods are intended for measurements on large surfaces when rapid measurements must be made and where a nondestructive test is desired. They are particularly useful for production control tests.

2. Summary of Methods

2.1 At least two different types of instruments are commercially available for performing this measurement. One type measures radiant energy reflected from the specimen (Method A),[2] and the other type measures radiant energy emitted from the specimen (Method B).[3] A brief description of the principles of operation of each method follows.

2.1.1 *Method A*—The theory employed in Method A has been described in detail by Nelson et al[4] and therefore is only briefly reviewed herein. The surface to be measured is placed against an opening (or aperture) on the portable sensing component. Inside the sensing component are two semi-cylindrical cavities which are maintained at different temperatures, one at near ambient and the other at a slightly elevated temperature. A suitable drive mechanism is employed to rotate the cavities alternately across the aperture. As the cavities rotate past the specimen aperture, the specimen is alternately irradiated with infrared radiation from the two cavities. The cavity radiation reflected from the specimen is detected with a vacuum thermocouple. The vacuum thermocouple views the specimen at near normal incidence through an optical system which transmits radiation through slits in the ends of the cavities. The thermocouple receives both radiation emitted from the specimen and other surfaces, and cavity radiation which is reflected from the specimen. Only the reflected energy varies with this alternate irradiation by the two rotating cavities, and the detection-amplifying system is made to respond only to the alternating signal. This is accomplished by ro-

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[1] This method is under the jurisdiction of ASTM Committee E-21 on Space Simulation.
Effective May 19, 1971.

[2] A satisfactory instrument for this type of measurement is the Infrared Reflectometer Model DB 100, manufactured by Gier-Dunkle Instruments, Inc., Santa Monica, Calif.

[3] A satisfactory instrument for this type of measurement is the Model 25A Emissometer, manufactured by the Lion Research Corp., Cambridge, Mass.

[4] Nelson, K. E., Leudke, E. E., and Bevans, J. T., *Journal of Spacecraft and Rockets*, JSCRA, Vol 3, No. 5, 1966, p. 758.

tating the cavities at the frequency to which the amplifier is tuned. Rectifying contacts coupled to this rotation convert the amplifier output to a d-c signal, and this signal is read with a millivoltmeter. The meter reading must be suitably calibrated with known reflectance standards to obtain reflectance values on the test surface. The resulting data can be converted to total normal emittance by subtracting the measured reflectance from unity.

2.1.2 *Method B*—The theory of operation of Method B has been described in detail by Gaumer et al[5] and is briefly reviewed as follows: The surface to be measured is placed against the aperture on the portable sensing component. Radiant energy which is emitted and reflected from the specimen passes through a suitable transmitting vacuum window and illuminates a thermopile. The amount of energy reflected from the specimen is minimized by cooling the thermopile and the cavity walls which the specimen views. The output of the thermopile is amplified and sensed by a suitable meter. The meter reading must be calibrated with standards of known emittance.

3. Limitations

3.1 Both methods are limited in accuracy by the degree to which the emittance properties of calibrating standards are known and by the angular emittance characteristics of the surfaces being measured.

3.2 Method A is normally subject to a small error caused by the difference in wavelength distributions between the radiant energy emitted by the two cavities at different temperatures, and that emitted by a blackbody at the specimen temperature. Method B also has nongray errors since the detector is not at absolute zero temperature. The magnitude of this type of error is discussed by Nelson et al.[4]

3.3 Method A is subject to small errors which may be introduced if the orientation of the sensing component is changed between calibration and specimen measurements. This type of error results from minor changes in alignment of the optical system.

3.4 Method A is subject to error when curved specular surfaces of less than about 300-mm radius are measured. These errors can be minimized by using calibrating standards that have the same radius of curvature as the test surface.

3.5 Method A can measure reflectance on specimens which are either opaque or semi-transparent in the wavelength region of interest (about 4 to 50 μm). However, if emittance is to be derived from the reflectance data on a semi-transparent specimen, a correction must be made for transmittance losses.

3.6 Method B is subject to several possible significant errors. These may be due to (*1*) variation of the test surface temperature during measurements, (*2*) differences in temperature between the calibrating standards and the test surfaces, (*3*) changes in orientation of the sensing component between calibration and measurement, (*4*) errors due to irradiation of the specimen with thermal radiation by the sensing component, and (*5*) errors due to specimen curvature. Variations in test surface temperature severely limit accuracy when specimens that are thin or have low thermal conductivity are being measured. Great care must be taken to maintain the same temperature on the test surface and calibrating standards. Meter readings are directly proportional to the radiant flux emitted by the test surface, which in turn is proportional to the fourth power of temperature. Changes in orientation of the sensing component between calibration and test measurement introduces errors due to temperature changes of the thermopile. The relatively poor vacuum around the thermopile results in variations in convection heat transfer coefficients which are affected by orientation.

3.7 Method B is limited to emittance measurements on specimens that are opaque to infrared radiation in the wavelength region of interest (about 4 to 50 μm).

3.8 The emittance measured by Method B is an intermediate value between total-normal and total-hemispherical emittance because of the relationship between the thermocouple sensing elements and the test surface. The close proximity of the thermopile to the relatively large test surface allows it to receive radiation emitted over a significant angle (up

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[5] Gaumer, R. E., Hohnstreiter, G. F., and Vanderschmidt, G. F., "Measurement of Thermal Radiation Properties of Solids," *NASA SP-31*, 1963, p. 117.

to 80 deg). This error (the difference between total-normal and total-hemispherical emittance can be as large as 10 percent on certain types of specimens (such as specular metal surfaces).

4. Procedure

4.1 Calibration procedures for both methods of measurement are jointly discussed because of their similarity. In Method A infrared reflectance properties of calibrating standards must be known, and for Method B emittance values of standards are utilized. Following an appropriate warm-up time, calibrate the readout meter. Adjust the meter to give the correct reading when measuring both high and low emittance (or reflectance) standards. Repeat calibration of the meter several times at short time intervals until the correct readings can be obtained near each end of the scale. Typical high and low emittance (low and high reflectance) standards may consist of black paint (or preferably a blackbody cavity) and polished high-purity aluminum, respectively. Measure the thermal radiation properties of the standards independently with an absolute instrument, and maintain the standards in a clean condition thereafter.

4.2 In Method B care must be taken to prevent stray radiant energy from entering the sensor. This can occur if the test surface is not sufficiently flat or is not opaque.

4.3 In Method B the test surfaces and calibrating standards must be maintained at the same temperature. If thin (less than about 0.7 mm thick) conducting specimens are to be measured, they should be bonded to a thick metallic substrate. Specimen temperature changes can be noted by observing whether the indicated meter reading drifts with time.

4.4 In Method B the orientation of the sensor must be the same for both calibration and test surface measurements.

4.5 After the meter has been properly calibrated, place the test surface over the aperture of the measuring instrument. The resulting meter reading of Method A is then the infrared reflectance for blackbody radiant energy at near room temperature, or in Method B, a meter reading that can be converted to emittance using the manufacturers emittance/meter reading conversion data. In Method A, obtain the emittance by subtracting the reflectance from unity. It is recommended that the instrument be recalibrated as soon as possible after measuring the test surface. If the meter calibration has changed, repeat the entire calibration and readout procedure. It is recommended that at least three readings be taken for each test specimen, and the results averaged, to minimize statistical errors. It is also recommended that both laboratory and working emittance (or reflectance) standards be maintained, and that they be kept clean.

5. Report

5.1 The report shall include the following:

5.1.1 Name and pertinent other identification of the test material,

5.1.2 Name and pertinent other identification or traceability of the surfaces used for calibration,

5.1.3 Emittance (or reflectance) values assumed for calibration surfaces,

5.1.4 Locations on the surface area at which emittance (or reflectance) measurements were performed. (Not applicable for small individual test specimens.),

5.1.5 Ambient temperature,

5.1.6 For Method A the indicated meter reading (reflectance) shall be recorded for three successive measurements. An average of the three values shall than be calculated and subtracted from one to obtain the emittance,

5.1.7 For Method B the indicated meter reading shall be recorded for three successive measurements. These meter readings shall be converted to emittance using the manufacturer's data, and then averaged, and

5.1.8 Date and time the measurements were taken.

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Document Name: ASTM E424: Test for Solar Energy Transmittance and Reflectance (Terrestrial) of Sheet Materials

CFR Section(s): 24 CFR 200, Subpart S

Standards Body: American Society for Testing and Materials

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Designation: E 424 – 71

Standard Methods of Test for
SOLAR ENERGY TRANSMITTANCE AND REFLECTANCE (TERRESTRIAL) OF SHEET MATERIALS[1]

1. Scope

1.1 These methods cover the measurement of solar energy transmittance and reflectance (terrestrial) of materials in sheet form. Method A, using a spectrophotometer, is applicable for both transmittance and reflectance and is the referee method. Method B is applicable only for measurement of transmittance using a pyranometer in an enclosure and the sun as the energy source. Specimens for Method A are limited in size by the geometry of the spectrophotometer while Method B requires a specimen 0.61 m (2 ft) square. For the materials studied by the drafting task group, both methods give essentially equivalent results.

2. Applicable Documents

2.1 *ASTM Standards:*

E 259 Recommended Practice for Preparation of Reference White Reflectance Standards[2]

E 275 Recommended Practice for Describing and Measuring Performance of Spectrophotometers[2]

E 284 Definitions of Terms Relating to Appearance of Materials[2]

E 308 Recommended Practice for Spectrophotometry and Description of Color in CIE 1931 System[2]

3. Summary of Methods

3.1 *Method A*—Measurements of spectral transmittance, or reflectance *versus* a magnesium oxide standard, are made using an integrating sphere spectrophotometer over the spectral range from 350 to 2500 nm. The illumination and viewing mode shall be normal-diffuse or diffuse-normal. The solar energy transmitted or reflected is obtained by integrating over a standard solar energy distribution curve using weighted or selected ordinates for the appropriate solar-energy distribution. The distribution at sea level, air mass 2, is used.

3.2 *Method B*—Using the sun as the source and a pyranometer as a detector the specimen is made the cover of an enclosure with the plane of the specimen perpendicular to the incident radiation; transmittance is measured as the ratio of the energy transmitted to the incident energy. (The apparatus of Method B has been used for the measurement of solar-energy reflectance but there is insufficient experience with this technique for standardization at present.)

4. Significance

4.1 Solar-energy transmittance and reflectance are important factors in the heat admission through fenestration, most commonly through glass or plastics. (See Appendix A3.) These methods provide a means of measuring these factors under fixed conditions of incidence and viewing. While the data may be of assistance to designers in the selection and specification of glazing materials, the solar-energy transmittance and reflectance are not sufficient to define the rate of heat transfer

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[1] These methods are under the jurisdiction of ASTM Committee E-12 on Appearance of Materials.
Effective April 15, 1971.

[2] *Annual Book of ASTM Standards*, Part 30.

without information on other important factors. The methods have been found practical for both transparent and translucent materials as well as for those with transmittances reduced by highly reflective coatings. Method B is particularly suitable for the measurement of transmittance of inhomogeneous, patterned, or corrugated materials since the transmittance is averaged over a large area.

5. Definitions

5.1 *solar absorptance*—the ratio of absorbed to incident radiant solar energy (equal to unity minus the reflectance and transmittance).

5.2 *solar admittance*—solar heat transfer taking into account reradiated and convected energy.

5.3 *solar energy*—for these methods the direct radiation from the sun at sea level over the solar spectrum as defined in 4.2, its intensity being expressed in watts per unit area.

5.4 *solar reflectance*—the percent of solar radiation (watts/unit area) reflected by a material.

5.5 *solar spectrum*—for the purposes of these methods the solar spectrum at sea level extending from 350 to 2500 nm.

5.6 *solar transmittance*—the percent of solar radiation (watts/unit area) transmitted by a material.

6. Method A—Spectrophotometric Method

6.1 *Apparatus:*

6.1.1 *Spectrophotometer*—An integrating sphere spectrophotometer, by means of which the spectral characteristics of the test specimen or material may be determined throughout the solar spectrum. For some materials the spectrum region from 350 to 1800 nm may be sufficient. The design shall be such that the specimen may be placed in direct contact with the sphere aperture for both transmission and reflection, so that the incident radiation is within 6 deg of perpendicularity to the plane of the specimen.[3]

6.1.2 *Standards:*

6.1.2.1 For transmitting specimens, incident radiation shall be used as the standard relative to which the transmitted light is evaluated. Paired reflecting standards are used, prepared in duplicate as described below.

6.1.2.2 For reflecting specimens, use smoked magnesium oxide (MgO) as a standard as the closest practicable approximation of the completely reflecting, completely diffusing surface for the region from 300 to 2100 nm. The preferred standard is a layer (at least 2.0 mm in thickness) freshly prepared from collected smoke of burning magnesium (Recommend Practice E 259). Pressed barium sulfate ($BaSO_4$) or MgO are not recommended because of poor reflecting properties beyond 1000 nm.

6.1.3 *Specimen Backing for Reflectance Measurement*—Transparent and translucent specimens shall be backed by a light trap or a diffusing black material which is known to absorb the near infrared. The backing shall reflect no more than 1 percent at all wavelengths from 350 to 2500 nm as determined using the spectrophotometer.[4]

6.2 *Test Specimens:*

6.2.1 Opaque specimens shall have at least one plane surface; transparent and translucent specimens shall have two surfaces that are essentially plane and parallel.

6.2.2 Comparison of translucent materials is highly dependent on the geometry of the specific instrument being used. It is recommended that the specimen be placed in direct contact with the sphere to minimize and control loss of scattered radiation.

6.2.3 For specularly reflecting specimens the sphere conditions, especially where the reflected beam strikes the sphere wall, shall be known to be highly reflecting (95 percent or higher). It is recommended that a freshly coated sphere be used especially when measuring translucent or specularly reflecting specimens.

6.3 *Calibration:*

6.3.1 *Photometric*—The calibration of the photometric scale shall be done as recommended by the manufacturer. It shall be carefully executed at reasonable time intervals to ensure accuracy over the entire range.

6.3.2 *Wavelength*—Periodic calibrations should be made of the wavelength scales. Pro-

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[3] The Beckman DK-2 Recording Spectrophotometer available from Beckman Instruments, Inc., Fullerton, Calif., and the Cary 14 and 17 Recording Spectrophotometer available from Varian Assoc., Palo Alto, Calif., have been found satisfactory for this purpose. For additional apparatus specifications see Recommended Practice E 308.

[4] A piece of velvet sprayed with Nextel velvet coating 101-C10 Black available from 3M Company; Parson's Black available from Eppley Laboratories, Newport, R. I.: or Krylon Flat Black have been found satisfactory for this purpose.

cedures for wavelength calibration may be found in Recommended Practice E 275. A didymium filter has also been used for this purpose. Although the absorption peaks have been defined for specific resolution in the visible spectrum it also has peaks in the near infrared; however, the wavelength of the peaks must be agreed upon, using a specific instrument.

6.4 *Procedure:*

6.4.1 *Transmittance*—Obtain spectral transmittance data relative to air. For measurement of transmittance of translucent specimens, place freshly prepared matched smoked MgO surfaces at the specimen and reference ports at the rear of the sphere (Note 1). The interior of the sphere should be freshly coated with MgO and in good condition.

NOTE 1—Magnesium oxide standards may be considered matched if on interchanging them the percent reflectance is altered by no more than 1 percent at any wavelength between 350 and 1800 nm.

6.4.2 *Reflectance*—Obtain spectral directional reflectance data relative to MgO. Include the specular component in the reflectance measurement. Back the test specimen with a black diffuse surface if it is not opaque. Depending on the required accuracy, use the measured values directly or make corrections for instrumental 0 and 100 percent lines (see Recommended Practice E 308).

6.5 *Calculation*—Solar energy transmittance or reflectance is calculated by integration. The distribution of solar energy as reported by Parry Moon[5] for sea level and air mass 2 shall be used.

6.5.1 *Weighted Ordinates*—Obtain the total solar energy transmittance, T_{se}, and reflectance, R_{se}, in percent, by integrating the spectral transmittance (reflectance) over the standard solar energy distribution as follows:

$$T_{se} \text{ or } R_{se} = \sum_{\lambda = 350 \text{ nm}}^{\lambda = 2100 \text{ nm}} T_\lambda \text{ (or } R_\lambda) \times E_\lambda$$

E_λ for air mass 2, at 50-nm intervals, normalized to 100, is given in Appendix A1.

6.5.1.1 This integration is easily programmed for automatic computation.

6.5.2 *Selected Ordinates*—Integration is done by reading the transmittance or reflectance at selected wavelengths and calculating their average. Appendix A2 lists 20 selected ordinates for integration.[6]

6.6 *Report*—The report shall include the following:

6.6.1 Complete identification of the material tested, and whether translucent, clear, or specularly reflecting,

6.6.2 Solar *T* percent or Solar *R* percent, or both, to the nearest 0.1 percent,

6.6.3 Specimen thickness,

6.6.4 Identification of the instrument used, and

6.6.5 Integration method.

7. Method B—Pyranometer Method

NOTE 2—The pyranometer is used to measure total global (sun and sky) radiation (previously designated a 180 deg pyroheliometer; presently the latter word refers to a normal incidence measurement of direct solar radiation). See IGY Instruction Manual, Part VI, *Radiation Instruments*, Pergamon Press, New York, N. Y.

7.1 *Apparatus:*

7.1.1 *Enclosure*—The apparatus that has been used successfully is a box capable of supporting a 0.61-m (24-in.) square specimen. The box, which would normally be about 0.66-m (26-in.) square outside, should be capable of being faced in any direction, as on a universal mount. The inside of the box should be painted flat black.[4] A typical unit is shown in Fig. 1.

7.1.2 *Sensor:*

7.1.2.1 The sensing element of this instrument is a pyranometer consisting of concentric rings, or wedges of thermopiles, colored alternately black and white.[7] The voltage output of this sensor is proportional to the intensity of the total incident solar irradiation. The spectral sensitivity of this instrument extends from the ultraviolet to infrared wavelengths (280 to 2800 nm), thus encompassing all the solar spectrum. The pyranometer should be located inside the box so that the sensing thermopile is approximately 50 mm (2 in.) from the center of the bottom plane of the sample.

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[5] *Journal of the Franklin Institute*, Vol 230, 1940, p. 583, or *Smithsonian Physical Tables*, Table 1, Vol 815, 1954, p. 273.

[6] Olson, O. H., "Selected Ordinates for Solar Absorptivity Calculations," *Applied Optics*, Vol 2, No. 1, January 1963.

[7] An Eppley 50-Junction Pyranometer, Serial No. 9624 (6.66 MV/cal·cm²·min) and an Eppley 10-Junction Pyranometer, Serial No. 8553 (2.21 MV/cal·cm²·min) available from Eppley Laboratories, Inc., Newport, R. I., have been found satisfactory for this purpose. Various other Eppley Pyranometers have also been successfully used.

7.1.2.2 The pyranometer has a viewing area of 180 deg. An Eppley pyranometer with its 25-mm (1-in.) diameter sensing disk, when placed in the center of the box, views the midpoint of the edges of the test specimen as a cone of 160 deg; the diagonal of the specimen is viewed as a cone of 166 deg when the thermopile is 50 mm (2 in.) below the bottom of the specimen.

7.2.3 *Read-Out Instrumentation*—A recorder, or a nonrecording meter capable of indicating in the 0.2 to 15-mV range are permissible for use. The output voltage of the pyranometer will be affected by the input impedance of the meter to which it is connected. Thus, the meter used to indicate solar intensity should have a very high input impedance, such as a precision vacuum-tube voltmeter, or a meter which has been calibrated for one particular sensing element, thus compensating for any loading effects on that element.

7.2 *Specimens*—The test specimens should be not less than 0.61 by 0.61 m (24 by 24 in.). If the cross-sectional shape of the specimen is not flat, care must be taken to prevent the possibility of light leaks at the edges such as are caused by the use of oversize specimens.

7.3 *Procedure:*

7.3.1 Conduct the tests on a clear sunny day with no cloud cover interruptions during the individual tests. Conduct testing between the hours of 9 a.m. and 3 p.m. local standard time; this is when the solar radiation is at least 80 percent of the value obtained at solar noon for that day. In the Northern hemisphere take readings between November and February only between 10 a.m. and 2 p.m. Expose the test specimen approximately normal to the sun for 15 min prior to testing. Next, align the box normal to the sun's rays and take the average incident solar-energy reading over a period of time (normally several minutes) until a steady trace, or reading is obtained. Then place the test specimen on the box and again record the average solar energy reaching the sensor. When the test specimen has a corrugated or irregular surface move it across the sensing element, and take readings at 10-mm (½-in.) intervals for the width of one corrugation or irregularity, and average the readings. Also measure corrugated specimens with the corrugations in the North - South direction and in the East - West direction.

7.3.2 The solar energy transmittance of the test specimens is the ratio of the energy measured when the test specimen is placed between the sun and the sensor and the energy measured by the sensor with no test specimen in place.

7.4 *Report*—The report shall include the following:

7.4.1 The source and identity of the test specimen,

7.4.2 A complete description of the test specimen, that is, thickness, cross-sectional shape, color, size, translucent or transparent, type of material,

7.4.3 The percent solar energy transmittance to the nearest 1 percent,

7.4.4 The place, date, and time of the test,

7.4.5 The intensity of the solar radiation,

7.4.6 Type of sensing unit used, and

7.4.7 Ambient air temperature.

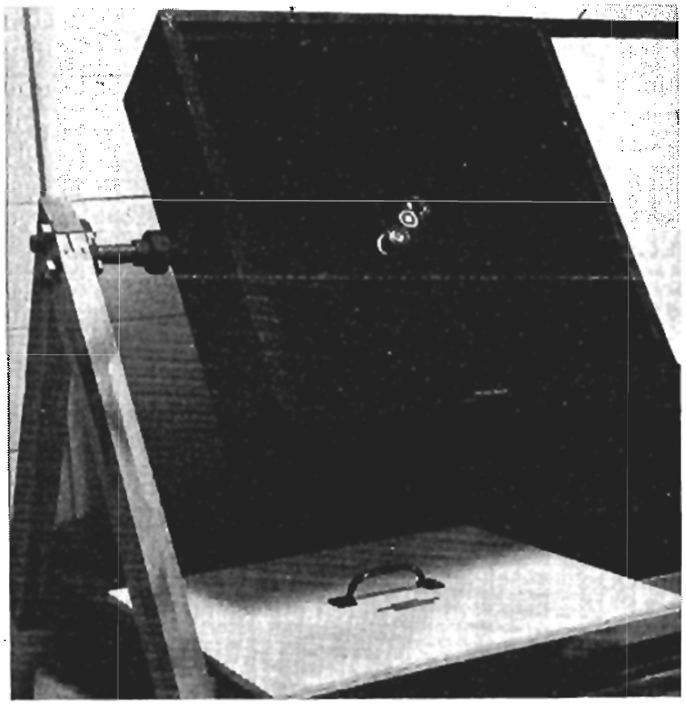

FIG. 1 Typical Unit with Pyranometer Mounted in Black Box.

APPENDIXES

A1. SOLAR ENERGY TRANSMITTANCE OR REFLECTANCE USING WEIGHTED ORDINATES (NORMALIZED TO $\Sigma = 100.00$)

$$T_{se}\text{ (percent)} = \sum_{\lambda = 350\text{ nm}}^{\lambda = 2100\text{ nm}} T_\lambda \times E_\lambda$$

Wavelength, nm	Relative Energy	Wavelength, nm	Relative Energy	Wavelength, nm	Relative Energy
350	1.27	950	3.29	1550	1.49
400	3.18	1000	4.25	1600	1.36
450	6.79	1050	3.72	1650	1.17
500	8.20	1100	1.70	1700	0.89
550	8.03	1150	1.46	1750	0.54
600	7.88	1200	2.52	1800	0.01
650	7.92	1250	2.21	1850	0.00
700	7.48	1300	1.78	1900	0.00
750	5.85	1350	0.12	1950	0.12
800	5.79	1400	0.00	2000	0.02
850	5.66	1450	0.16	2050	0.26
900	3.24	1500	1.06	2100	0.58
				Total	100.00

A2. TWENTY SELECTED ORDINATES FOR EVALUATION OF SOLAR TRANSMITTANCE OR REFLECTANCE AT SEA LEVEL

No.	Wavelength, nm	No.	Wavelength, nm
1	390	11	745
2	444	12	786
3	481	13	831
4	511	14	877
5	543	15	959
6	574	16	1026
7	606	17	1105
8	639	18	1228
9	669	19	1497
10	705	20	1722

A3. SOLAR ADMITTANCE PARAMETERS

A3.1 Solar energy poses a complex problem to architects and engineers concerned with maintaining a comfortable indoor space condition. The problem exists when solar energy is admitted into a space which must be thermally and optically controlled, that is, temperature, humidity, and brightness.

A3.2 The amount of solar-energy admitted into a space can be calculated with the solar-admittance parameters, total solar-energy transmittance (TSET), and total solar-energy reflectance (TSER) of the materials surrounding the space.

A3.3 With homogenous materials the percent of solar energy reflected, R, absorbed, A, and transmitted, T, can be determined by the following equation:

$$100 \text{ percent} = R + A + T$$

A3.4 For transparent materials, such as glass and clear plastics, the total solar energy transmittance is significant and environmental control systems must be designed to handle the changing solar load.

A3.5 Space environmental engineers use the total solar-energy transmittance and total solar-energy reflectance parameters of materials to determine the solar energy admitted into a space.

A3.5.1 *For example:*

A ¼-in. bronze-tinted glass has the following typical solar energy admittance properties:

$$\begin{aligned} \text{TSET} &= 46 \text{ percent} = T \\ \text{TSER} &= 6 \text{ percent} = R \\ \text{TSEA} &= 48 \text{ percent} = A \end{aligned}$$

For the following conditions:

Design day—Sept. 21, 40 deg North latitude, 4 p.m., West elevation (*ASHRAE Handbook of Fundamentals*, 1967, Table 4, p. 472).[8] (All solar energy rates are per hour, square foot of glazing area.)

Direct normal solar irradiation:	230 Btu
Recommended outdoor wind velocity: (Table 9, Item 3, p. 477)[8]	7.5 mph
Corresponding outdoor surface coefficient:	4.0 Btu/deg F
Recommended indoor air velocity: (Table 9, Item 3, p. 477)[8]	Still
Corresponding indoor surface coefficient:	1.46 Btu/ deg F

Total solar energy admitted indoors:

Total solar heat gain indoors:

$$= 0.46\,(230) + 1.46/[(1.46 + 4)\,(0.48 \times 230)]$$

where:

$$\begin{aligned} 0.46\,(230) &= \text{transmitted solar energy} \\ &= 106 \text{ Btu} \end{aligned}$$

and:

$$1.46/[(1.46 + 4.0)\,(0.48 \times 230)]$$

(*ASHRAE Handbook*, 1967, p. 480) Eq 19

= portion of absorbed solar energy reradiated and convected indoors
= 30 Btu (Note A1)

Total solar energy admitted indoors
= 136 Btu (Note A2)

A3.5.2 The 1967 *ASHRAE Handbook of Fundamentals*[8] reviews this procedure on pages 477 through 480.

NOTE A1—The amount of absorbed solar energy which is reradiated and convected indoors is a direct function of the air movement over the indoor and outdoor glazing surfaces.

NOTE A2—Cooling loads used for design also include conduction resulting from out-in temperature differences; heat capacity of building materials may introduce a delay in peak load timing.

A3.6 The Handbook[8] also illustrates a more commonly used method on pages 470 through 476 with the use of shading coefficients for the glazing under consideration and solar heat gain factors (SHGF) for ⅛-in. clear glass (Tables 2 to 6, pp. 470–474).

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[8] *ASHRAE Handbook of Fundamentals*, American Society of Heating, Refrigerating, and Air Conditioning Engineers, 345 E. 47th St., New York, N. Y. 10017, 1967, pp. 470–480.

A3.6.1 *For example:*

A ¼-in. bronze-tinted glass typical shading coefficient = 0.67.

If, SHGF = 205 Btu (Table 4, Sept. 21, p. 472, West elevation, 4 p.m.),

Solar energy admitted indoors = 0.67 × 205 = 137 Btu.

A3.7 Double glazing computations become more complex and the solar energy admitted indoors requires considerably more calculation as the RAT formula does not apply directly. For this type of glazing, the shading coefficient technique is more applicable.

A3.8 Representative shading coefficients are available from glass and plastic manufacturers.

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Standard Recommended Practice for
CONSTANT-AMPLITUDE LOW-CYCLE FATIGUE TESTING[1]

This standard is issued under the fixed designation E 606; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This recommended practice covers the determination of low-cycle fatigue properties of nominally homogeneous metallic materials by the use of uniaxially loaded test specimens. It is intended as a guide for low-cycle fatigue testing performed in support of such activities as materials research and development, mechanical design, process and quality control, product performance, and failure analysis.

1.2 The use of this recommended practice is limited to metallic specimens and does not cover testing of full-scale components, structures, or consumer products.

1.3 This recommended practice is applicable to temperatures and strain rates for which the magnitudes of time-dependent inelastic strains are negligible relative to the magnitudes of time-independent inelastic strains. No restrictions are placed on environmental factors, such as temperature, pressure, humidity, medium, etc., provided they remain constant throughout the test and do not cause loss of or change in dimensions with time.

NOTE 1—The term *inelastic* is used herein to refer to all nonelastic strains. The term *plastic* is used herein to refer only to the time-independent (that is, noncreep) component of inelastic strain.

NOTE 2—Appendix X4 has been added to cover those test conditions where time dependent strains may be present.

1.4 This recommended practice is restricted to the testing of axially loaded uniform and hour-glass profile test specimens in the low-cycle fatigue regime. Testing is limited to constant-amplitude stress or strain cycling in which stress and strain are to be continuously varying (that is, without hold times).

2. Applicable Documents

2.1 *ASTM Standards:*

A 370 Methods and Definitions for Mechanical Testing of Steel Products[2]

E 3 Methods of Preparation of Metallographic Specimens[3]

E 4 Practices for Load Verification of Testing Machines[4]

E 8 Methods of Tension Testing of Metallic Materials[5]

E 9 Methods of Compression Testing of Metallic Materials at Room Temperature[6]

E 83 Practice for Verification and Classification of Extensometers[6]

E 111 Test Method for Young's Modulus, Tangent Modulus, and Chord Modulus[6]

E 112 Methods for Determining Average Grain Size[3]

E 132 Test Method for Poisson's Ratio at Room Temperature[6]

E 157 Method of Assigning Crystallographic Phase Designations in Metallic Systems[3]

E 209 Practice for Compression Tests of Metallic Materials at Elevated Temperatures with Conventional or Rapid Heating Rates and Strain Rates[6]

E 337 Test Method for Measuring Humidity with a Psychrometer (The Measurement of Wet-Bulb and Dry-Bulb Temperatures)[7]

E 384 Test Method for Microhardness of Materials[8]

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[1] This recommended practice is under the jurisdiction of ASTM Committee E-9 on Fatigue, and is the direct responsibility of Subcommittee E09.08 on Fatigue Under Cyclic Strain.

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[2] *Annual Book of ASTM Standards*, Vols 01.01, 01.02, 01.03, 01.04, 01.05, and 03.01.

[3] *Annual Book of ASTM Standards*, Vol 03.03.

[4] *Annual Book of ASTM Standards*, Vols 03.01, 04.02, 07.01, and 08.03.

[5] *Annual Book of ASTM Standards*, Vols 02.01, 02.02, 02.03, and 03.01.

[6] *Annual Book of ASTM Standards*, Vol 03.01.

[7] *Annual Book of ASTM Standards*, Vols 07.01, 11.03, and 15.09.

[8] *Annual Book of ASTM Standards*, Vols 03.01, 03.03, and 03.04.

E 399 Test Method for Plane-Strain Fracture Toughness of Metallic Materials[6]

E 467 Practice for Verification of Constant Amplitude Dynamic Loads in an Axial Load Fatigue Testing Machine[6]

E 513 Definitions of Terms Relating to Constant-Amplitude Low-Cycle Fatigue Testing[6]

3. Significance

3.1 Although low-cycle fatigue is a phenomenon that is influenced by many of the same variables that influence high-cycle fatigue, the nature of low-cycle fatigue imposes distinctive requirements on fatigue testing methods. In particular, cyclic total strain should be measured and cyclic plastic strain should be determined. Furthermore, either of these strains typically are used to establish cyclic limits; total strain usually is controlled throughout the cycle. The uniqueness of this recommended practice and the results it yields is the determination of cyclic stresses and strains throughout the tests.

3.2 Low-cycle fatigue can be an important consideration in the design of industrial products. It is important for situations in which components or portions of components undergo either mechanically or thermally induced cyclic plastic strains that cause failure within relatively few cycles. Information obtained from low-cycle fatigue testing may be an important element in the establishment of design criteria to protect against component failure by fatigue.

3.3 Low-cycle fatigue test results are useful in the areas of mechanical design as well as materials research and development, process and quality control, product performance, and failure analysis. Results of a low-cycle fatigue test program may be used in the formulation of empirical relationships between the cyclic variables of stress, total strain, plastic strain, and fatigue life. They are commonly used in data correlations such as curves of cyclic stress or strain versus life and cyclic stress versus cyclic plastic strain. Examination of the cyclic stress - strain curve and its comparison with monotonic stress - strain curves gives useful information regarding the cyclic stability of a material, for example, whether the values of hardness, yield point, yield strength, and strain hardening exponent will increase or decrease (that is, whether a material will harden or soften) due to cyclic plastic straining. Results of the uniaxial tests on specimens of simple geometry can be applied to the design of components with notches or other complex shapes provided that the strains can be determined and multiaxial states of stress or strain and their gradients are correctly correlated with the uniaxial strain data.

4. Definitions

4.1 The definitions in this recommended practice are in accordance with Definitions E 513.

5. Functional Relationships

5.1 Empirical relationships that have been used commonly for description of low-cycle fatigue data are given in Appendix X1.

5.2 Relationships for conversion of diametral strain to axial strain are given in Appendix X2.

6. Apparatus

6.1 *Testing Machine*—Testing should be conducted with a tension - compression fatigue testing machine that conforms to the requirements of Practices E 4 and Practice E 467. The machine should be one in which specific measures have been taken in its design to minimize backlash in the loading train.

NOTE 3—Load capacity of 45 kN (approximately 10 kips) or greater would be sufficient for the recommended specimens (Section 7) and most test materials. The machine load capacity used for these specimens ordinarily would not exceed 110 kN (approximately 25 kips).

6.2 *Stress or Strain Control*—Testing machine controls should permit cycling between constant stress or strain limits. If material behavior permits (for example, aging effects do not hinder), control stability should be such that the respective stress or strain limit is repeatable from cycle-to-cycle to within 0.5 % of the average control limit and repeatable over the test duration to within 2 % of the average control limit.

6.3 *Fixtures*—Fixtures should be provided for rigidly gripping specimen ends and accurately transmitting cyclic loads (without backlash) along the longitudinal specimen axis.

6.3.1 To minimize bending strains, specimen fixtures should be aligned such that the major axis of the specimen closely coincides with the load axis throughout each loading cycle. It is important that the accuracy of alignment be kept consistent from specimen to specimen. Alignment should be checked by means of a trial test specimen with longitudinal strain gages placed at three or four equidistant locations around the minimum diameter. The trial test specimen should be turned about its axis, installed, and checked for each of three or four orientations within the fixtures. The maximum bending strains so determined should not exceed 5 % of the minimum axial strain range imposed during any test program. For specimens having a uniform gage length, it is advisable to place a similar set of gages at two or three axial positions within the gage section. One set of strain gages should be placed at the center of the gage length to detect misalignment that causes relative rotation of the specimen ends about axes perpendicular to the specimen axis. An additional set of gages should be placed away from the gage-length center to detect relative lateral displacement of the specimen ends.

6.3.2 Several commonly used fixturing techniques are shown schematically in Fig. 4. The selection of any one fixturing technique depends primarily upon the user's specimen design. The fixtures should be constructed of hardened steel for high strength and abrasion resistance. Chrome or nickel plating may be needed for corrosion resistance. Fixtures may be coupled with the Wood's metal pot **(1, 2)**[10] of Fig. 3 to permit initially zero specimen stress during fixturing. Placement of the fixtures within a die-set or flex bars alleviates relative lateral motion of specimen ends and provides rigidity that is important in preventing compressive buckling of the test specimen. Initially, zero fixturing stress combined with precise and rigid alignment may be achieved by installing fixtures and a Wood's metal pot within the die-set or flex bars.

6.4 *Extensometers*—Extensometers should be employed for the purpose of measuring deformation in the gage section. They should be suitable for dynamic measurements over long periods of time.

6.4.1 The extensometers may be of two major types: electromechanical (for example, the more frequently used strain gage or LVDT type as shown in Fig. 5) or optical (for example, using image orthicon tubes **(2)** or lasers). In all cases, they should generate information suitable for readout and recordings. The extensometers should qualify as Class B-2 or better in accordance with Practice E 83; however, error in measured value of strain should not exceed ± 2.5 % of strain amplitude.

6.4.2 Extensometers should measure longitudinal deformation when a uniform-gage specimen, such as shown in Fig. 1(*a*), is tested. Generally, these extensometers are attached as shown in Fig. 5(*a*). Either the strain-gage or LVDT systems of Figs. 5(*c*) and 5(*d*) may be used with the probes shown in Fig. 5(*a*).

NOTE 4—Care should be exercised when installing the longitudinal extensometer so as to prevent damage to the specimen surface and consequential premature fatigue failure at the gage points. Small strips of cellophane tape can be applied to the specimen surface where the extensometer knife edges make contact so that the knife edges imbed in the tape rather than in the specimen. However, use care to ensure that no creep or slippage occurs in the tape adhesive.

6.4.3 Extensometers should measure diametral deformations when specimens having hourglass profiles are tested. A typical method of diametral displacement measurement is shown in Fig. 5(*b*). Curved extensometer tips, convex in the longitudinal plane, can provide point contact during testing. Either of the strain-gage or LVDT systems of Figs. 5(*c*) or 5(*d*) may be used with the probes shown in Fig. 5(*b*). Care should be exercised during installation of the diametral extensometer to prevent damage to the test specimen surface. Extensometer tips should be adjusted properly to minimize the force they impose on the specimen. When installing the extensometer, gently move its tip longitudinally along the specimen while watching the gage readout to find the minimum diameter. Calibration of extensometers should be conducted before and after each test program.

NOTE 5—Care should be taken in the measurement of diametral strains for materials such as cast materials that possess large crystals and a large degree of preferred orientation. These, as well as hexagonal close-packed materials, tend to be anisotropic and may require special methods of strain measurement and interpretation.

6.5 *Load Transducers*—A load transducer should be placed in series with the test specimen for the purpose of measuring magnitude and

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[10] The boldface numbers in parentheses refer to the references at the end of this standard.

sense of the axial load transmitted through the specimen. It should be designed specifically for fatigue testing and possess the following characteristics: high resistance to bending; low axial compliance; high linearity, accuracy, and sensitivity; low hysteresis. Load transducer capacity should be selected to adequately cover the range of loads to be observed, but not so large as to render large errors (that is, greater than 1 % peak measured load) in the measurement of small loads. Load transducer calibration should be verified in accordance with Practices E4 and E 467.

6.6 *Recording Systems*—The following recording systems should be considered a minimum requirement. Other data-retrieval systems, such as those incorporating digital computers, provide an acceptable substitute for the analog systems named here. Accuracy of recording systems should be kept within 1 % of full scale or close thereto in the case of oscilloscopes or oscillographic recorders.

6.6.1 *X-Y Recording*—Some means of *X-Y* recording should be used for the purpose of recording hysteresis loops of load versus deformation or stress versus strain. A potentiometric *X-Y* recorder or an oscilloscope equipped with a camera are acceptable alternatives. The potentiometric *X-Y* recorder should be used only when the rate of cycling results in a pen velocity that is less than one half of the recorder's slewing speed. At higher frequencies, the oscilloscope should be used.

6.6.2 *Strip Chart Recording*—Strip chart recorders should be available to monitor load (or strain). The frequency of strain cycling should be such that recording pen velocity never exceeds one half of the recorder's slewing speed. It is recommended that these recorders be calibrated at the testing frequency used.

6.7 *Cycle Counter*—A cycle counter shall be available for the purpose of counting and indicating total accumulated cycles of straining. A timer is a desirable adjunct to the cycle counter. When used to indicate total elapsed time to failure, it provides an excellent check against the cycle counter and frequency.

6.8 *Strain Computer*—An analog (or digital) computer is recommended for use in low-cycle fatigue tests of hourglass specimens whenever appreciable cyclic hardening and softening occur during the test. Such a computer is useful when used in the real time mode with servo-controlled testing machines and can be used for limit control of screw-driven machines. The computer should be designed to convert diametral strain and axial load signals into an axial strain signal. See Appendix X1 for conversion relations. In the case of servo-controlled machines, this axial strain signal may be used as a feedback signal for control purposes, thus simulating axial strain control. A block diagram for the analog (or digital) computer program is shown in Fig. 6.

6.9 *Calibration*—The calibration interval of all electronic recording and transducer systems should be performed in accordance with the manufacturer's recommendations; in the absence of these, the interval should be no greater than six months and even more frequent if necessary to maintain required accuracy. Calibration should be checked whenever accuracy is in doubt.

7. Specimens

7.1 *Specimen Design*—Figure 1 shows two basic specimen configurations. Figure 1(*a*) shows a recommended uniform-gage specimen and Fig. 1(*b*) shows the recommended hourglass profile. Selection of either 1(*a*) or 1(*b*) is subject to anisotropy and buckling considerations (see Note 5). Both of these recommended specimens possess a solid circular cross section and a minimum diameter of 6.35 mm (0.25 in.) in the test section. Specific cross-sectional dimensions are listed here only because they have been predominant in the generation of the low-cycle fatigue data base that exists in the open literature. Specimens possessing other diameters or tubular cross sections may be tested successfully within the scope of this recommended practice; however, crack growth rate, specimen grain size, and other considerations might preclude direct comparison with test results from the recommended specimens. While design of specimen end connections is primarily dependent upon user preference (see Note 7), a number of commonly used configurations are shown in Figs. 1(*c*), 1(*d*), and 1(*e*). Care must be exercised in the machining of uniform-gage specimens to blend the shoulder radius at the specimen ends with minimum diameter so as to avoid undercutting. So that stress concentrations are minimized, shoulder radius should be as large as possible, consistent with limitations on specimen length.

NOTE 6—Selection of either the uniform-gage section or hourglass profile is commonly based upon the magnitude of compressive strain to be imposed. The recommended uniform gage specimen is frequently suitable for strain ranges up to about 2 %. Above 2 %, hourglass specimens are recommended. Soft materials or elevated temperatures may dictate lower strain ranges. The recommended hourglass specimen possesses a profile ratio of 12:1 for radius of curvature to minimum radius of specimen. If the user wishes, different ratios between the limits of 8:1 and 16:1 may be employed. Lower limits will increase stress concentration and may affect fatigue life; higher ratios limit the specimen's buckling resistance. For some materials tested in the low life range, hourglass specimens might give different results from similarly stressed uniform-gage specimens. When test material is anisotropic, uniform specimens are recommended. It is very difficult to determine axial strains from measurements of diametral strain in hourglass specimens for many anisotropic materials.

NOTE 7—Design of specimen end connections is dependent upon user preference, fixturing, or availability of material or a combination of all three; it is constrained principally by proper consideration of axial alignment and backlash. Button-head end connections, such as those shown in Figs. 1(*d*) and 1(*e*), permit precise alignment with a specimen end clamping preload (to avoid backlash in the grip). The threaded connection, shown in Fig. 1(*c*), is useful where the available material is not thick enough to provide for the larger diameter button-head ends. The efficiency button-head connection, shown in Fig. 1(*e*), provides the button-head preloading feature, without requiring larger diameter ends. The button-head design is useful at elevated temperatures, as it does not suffer the "oxidation-sticking" experienced with threaded ends, but it may produce some specimen failures in the fixture when used at room temperature.

7.1.1 *Alternative Specimen Design for Sheet Specimens*—Often, it is desirable to obtain test specimens from sheet material that is less than 6.35 mm (0.25 in.) thick. In general, the considerations discussed in other sections apply equally to sheet testing. However, special specimen geometries and gripping arrangements, as well as more sensitive load and strain transducers, are necessary. Typical specimen designs that have been used successfully are shown in Fig. 2. The specimen in Fig. 2(*a*) has a rectangular cross section and is suitable up to strain amplitudes of at least 1 % applied to sheets as thin as 2.54 mm (0.100 in.). For higher strain amplitudes, the cylindrical cross section hourglass specimen in Fig. 2(*b*) is recommended. See Ref. **(3)** for other designs.

7.2 *Specimen Preparation*—Specimens should be prepared by a specific set of procedures that is known to provide consistent test results. Agreement between the testing organization and the user of the test results concerning preparation procedures should be obtained. The following provides recommended guidelines.

7.2.1 *Specimen Coupons and Materials*—Coupons from which specimens are machined should either be nominally homogeneous or sampled from the source material, or both, so as to be statistically representative of the properties sought in the application of the material to its end use. Thus, when material requirements allow, specimens should be removed from the fabricated component of interest. Any material orientations, such as rolling direction or casting direction, should be identified with respect to the orientation of the specimen axes. Orientation notation used in accordance with Method E 399 is acceptable such that L, T, S, LT, LS, or TS refer to the longitudinal axis of the specimen.

7.2.2 *Specimen Surface Preparation*—Specimens prepared from coupons will possess a "surface preparation history" as a consequence of machining operations, heat treatments, and the effects of environment during the storage period prior to testing. Unless the purpose of testing is to determine the influence of specific surface conditions on fatigue life, it is recommended that specimen surface preparation be performed in a manner that will have a minimum influence upon the variability in fatigue lives exhibited by the specimen group tested. Ordinarily, this would be accomplished by:

7.2.2.1 Consistently machining specimens to be as smooth and uniform in surface finish (in the gage region) as feasible for the subject material and the machining techniques available, and by employing as a final operation a machining or other "finishing" procedure that would introduce minimal surface metal distortion (see Note 8), and by

7.2.2.2 Ensuring, through the use of protective atmospheres, that surface attack, such as oxidation and corrosion, does not occur, either during heat treatments or during specimen storage, for all specimens within a program.

NOTE 8—Appendix X3 presents an example of a machining procedure that has been employed on some metals to minimize variability of machining and heat treatment influences upon fatigue life.

The exact procedure of specimen preparation and handling should be carefully documented.

7.3 *Specimen Storage*—Test specimens that may be susceptible to corrosion in moist room temperature air should be protected immediately after preparation and stored until they are tested. Specimens may be stored in a suitable protective environment, such as dry inert gas (as might be conveniently employed in a laboratory desiccator) or a vacuum.

7.4 *Materials Description*—A complete material description is desirable. It is recommended that the following metallurgical and mechanical properties be obtained:

7.4.1 *Metallurgical Characteristics*—Chemical composition, grain size (see Methods E 112), crystallographic structure, preferred orientation if present, general shape of grains (that is, equiaxed or elongated), second phase particles (see Method E 157), heat treatment (whether at the mill, during fabrication, in the laboratory, or a combination of all three), and specification designation (ASTM, ASME, AISI, Military, etc.).

7.4.2 *Mechanical Properties*—For purposes of performing the test and calculating results, it is desirable to have available the following representative mechanical properties, obtained at the appropriate temperature and measured in accordance with the applicable standards such as Methods E 8, E 9, E 111, E 132, and Practice E 209: tensile or compressive yield strength or yield point, or both, ultimate tensile strength, percent elongation, percent reduction of area, Poisson's ratio, and Young's modulus. The following true stress-strain properties may also be desirable: true fracture strength, true fracture ductility, strain hardening exponent, and strength coefficient. Hardness may also be determined in accordance with Methods A 370 or Test Method E 384, or both.

8. Procedure

8.1 *Test Environment:*

8.1.1 *Temperature*—Temperatures should be low enough that the magnitudes of time-dependent inelastic strains are negligible relative to the magnitudes of time-independent inelastic strains. For the duration of testing, specimen temperature should not vary by more than ±2°C (±3.6°F). Temperature gradients should be within ±2°C over the gage length of uniform-gage specimens. Initially, monitor the specimen temperature to determine the extent of heating due to strain cycling. If necessary, provide cooling to maintain the above limits.

NOTE 9—If the temperature cannot be maintained to within ±2°C, then temperature deviations should be reported. If possible, the effect of temperature should be demonstrated throughout the range of test temperatures. Preliminary testing is desirable if the time-dependent effects are not already known.

8.1.2 Elevated temperatures may be imposed by any of several methods: (*1*) high-frequency induction, (*2*) radiant furnace, or (*3*) immersion in an inert heated gas or liquid. In (*1*) and (*2*) above, an enclosure is recommended to prevent air currents in the vicinity of the specimen from causing undesirable temperature gradients. Specimens tested at room temperature should also be in draft-free surroundings. Temperatures below room temperature may be imposed by placing the specimen and gripping apparatus in a refrigerated chamber that may be either of the liquid or gaseous type, depending on temperature requirements and other possible environmental considerations. Liquefied gases, such as liquid nitrogen, or solidified gases, such as dry ice placed in a liquid medium, provide possible means for low-temperature testing.

NOTE 10—Use of glass insulation may avoid difficulty with wires submerged in a cooling solvent.

8.1.3 If testing is performed in air, relative humidity may be measured in accordance with Method E 337, unless it has already been determined that moisture has little or no effect on fatigue life for the material under test. If an effect is present, relative humidity should be controlled; when uncontrolled, it should be carefully monitored.

8.2 *Measurement of Test Specimen Dimensions*—For the purpose of making an accurate determination of specimen cross-sectional area, measure the reduced section as follows:

8.2.1 Measure the diameter at the center of the gage section by means of an optical comparator or other optical means read or estimated to an accuracy of 0.0125 mm (0.0005 in.) or better. A precision micrometer may be used in place of the optical comparator if its use does not damage the gage section surface in a way as to affect specimen performance. For uniform-gage specimens, check diameters for at least two other positions within the specimen gage length.

8.3 *Test Machine Control*—It is necessary to control one (or more) variable(s) (for example, stress, strain, load, displacement, or other appropriate parameter) in a manner that is in keeping with the test objectives.

8.3.1 *Control Mode*—Total axial strain amplitude is the most commonly utilized control variable in a low-cycle fatigue test. Total axial strain is often controlled continuously throughout each fatigue cycle in a manner prescribed in 8.4. It is also acceptable to control only the limits of either total axial strain or plastic axial strain. In such cases vary another variable, such as diametral strain, displacement, or load, between these limits in some cyclically consistent manner under either closed loop or other control means. For long-life fatigue tests that exhibit low but measurable levels of plastic strain, it is acceptable to control load while monitoring plastic and total strain and making periodic adjustments of mean load and load range in order to maintain the desired strain limits.

8.3.2 *Closed Loop Method*—Fatigue testing machines of the closed loop servo-controlled type often are capable of continuously controlling specific test variables such as load or displacement through appropriate selection of feedback signals. Application of scale factors to these signals thereby permits continuous control of stress or strain. Axial stress may be scaled directly from the load cell signal. Axial strain may be scaled directly from an axial extensometer signal when uniform-gage specimens are tested. When hourglass specimens are tested, an axial strain signal must be determined from a diametral extensometer signal and the force signal by means of a computer (see 6.9) if closed loop control of axial strain is desired. Closed loop control of diametral strain is generally not equivalent to closed loop control of axial strain since the relationship between axial strain and induced diametral strain changes during cycling for materials that undergo cyclic hardening and softening.

8.3.3 *Other Control Methods*—Fatigue testing machines that do not provide continuous closed loop control of either specimen load or specimen displacement generally have the capability to impose limits on the chosen test variable. However, they do not control that variable throughout the fatigue cycle. Limit control is a special case of closed loop control. Thus, load and displacement signals may be handled in a manner similar to that of 8.3.2 to determine strain limits. It is not necessary to use a computer with these machines for limit control of hourglass specimens if periodic adjustments are made to the diametral strain amplitude in such a manner as to maintain constant axial strain limits. These adjustments are necessary for materials that undergo significant cyclic hardening and softening because of attendant changes in the relationship between axial strain and induced diametral strain.

8.4 *Waveform*—The strain (or stress) versus time waveform should be identical throughout a test program unless test objectives are to determine waveform effects. In the absence of specific waveform requirements or equipment limitations, a triangular waveform is preferred.

8.5 *Strain Rate and Frequency of Cycling*—Either strain rate or frequency of cycling should be held constant for the duration of each test as well as for the duration of a test program, unless the test objective is specifically to determine either strain rate or frequency effects, respectively.

NOTE 11—While constant-strain-rate testing is often preferred, constant-frequency testing may be of greater practical significance to the fatigue analysis of certain machine components. On the other hand, constant-strain-rate testing may be experimentally more tractable than constant-frequency testing since long-life, small-strain tests in the former mode may be completed in shorter periods of time than tests conducted in the latter mode.

8.5.1 If nontriangular waveforms or equipment limitations preclude constant-strain-rate testing and time limitations preclude constant-frequency testing, other means of rate control are available. One accepted procedure is to maintain constant average strain rate (twice the product of strain range and frequency) throughout each test and for the duration of the test program. Another acceptable procedure, one that is most convenient when testing under plastic strain limit control, is to maintain constant average plastic strain rate.

8.5.2 The selected range of strain rates or frequencies should be sufficiently low as to preclude specimen heating in excess of 2°C (3.6°F). In a servo-controlled testing machine, make a comparison of the program and feedback signals to ensure that the selected rates or frequencies are and remain within system capabilities and accuracy requirements. Frequency response of extensometers (depending upon their design) is often a limiting factor in the system.

NOTE 12—Notwithstanding the need for constancy of rate, the testing rate may be reduced briefly in order to permit periodic recording operations if doing so does not change specimen behavior.

8.6 Begin all tests on the same half-cycle, tensile or compressive, unless the purpose of testing is to study initial loading effects. For some materials it is acceptable to increase strain amplitude gradually and continuously over a period not greater than 2 % of anticipated life.

8.7 *Number of Specimens*—It is suggested that a minimum of ten specimens be used to generate a fatigue strain-life curve. It is also suggested that the replication guidelines given in STP 588 **(4)** be followed, especially if subsequent statistical analysis is planned.

8.8 *Recording*—Unless computerized data retrieval systems are employed continuously, record the initial series of hysteresis loops of axial stress (or load) versus total or plastic axial strain (versus total or plastic diametral strain if an axial strain signal is not available). Record single hysteresis loops thereafter at successively larger increments of cycle count. For tests of 100 cycles or more, a minimum of ten additional hysteresis loops is desirable. When practical, continuously record the dependent variables (for example, axial stress and plastic axial strain in a total axial strain control test) as a function of time.

NOTE 13—When continuous recording is not practical either due to lengthy test durations or the limited availability of recorders, intermittent records or alternative sampling of the recorded variables is acceptable.

8.9 *Determination of Failure*—Failure can be determined in a number of ways dependent on the ultimate use of fatigue life information and the nature of the material being tested. (See Note 13.) Several commonly used criteria include the following:

8.9.1 Total separation or fracture of the specimen into two parts at (*a*) some location within the gage section of a uniform-gage specimen, or (*b*) the vicinity of the minimum diameter in an hourglass specimen. Fracture at any other location than specified above should be considered as an invalid result.

NOTE 14—A post-mortem failure analysis should be performed to uncover any unusual causes of failure. Inclusions, voids, machining defects, etc., that are not representative of the bulk material or its application may render an invalid fatigue life determination. (See 8.11.3.)

8.9.2 For fully reversed straining (or loading), a drop in the peak tensile stress (or increase in tensile peak strain) that is some preselected percentage greater than a similar drop in the peak compressive stress (or increase in compressive peak strain).

8.9.3 For strain control, cusp formation in the compressive portion of the hysteresis loop, such that the size of the cusp (Note 14) has grown to some preselected percentage of the peak compressive stress.

NOTE 15—Cusp size is taken as peak compressive stress minus stress at the inflection point of the compressive loading curve (see Fig. 7). Frequent *X-Y* recordings should be made in order to observe cusp formation.

8.9.4 A change in the rate-of-change of cyclic load range (or deformation range) that exceeds some preselected percentage change.

NOTE 16—For failure criteria 8.9.2 through 8.9.4, care should be exercised in the interpretation of cyclic stress-strain behavior when hysteresis loops exhibit serrations and cyclic hardening mechanisms compete with cyclic softening mechanisms for dominance at different times throughout life.

8.9.5 The existence of surface microcracks (for example, as observed optically or by replicas) that are larger than some preselected size consistent with test objectives.

8.10 Conduct testing at least until failure and preferably until fracture when testing needs dictate and economics allow. Record total accumulated cycles to failure (and fracture) by means of a cycle counter and check against an elapsed time meter.

8.11 *Analysis of Data*—While it is not the purpose of this recommended practice to specify data analysis techniques, the following example represents a common procedure:

8.11.1 *Determination of the Cyclic Stress - Strain Curve*—Generate a cyclic stress - strain curve from paired values of stress range and plastic strain range **(5)**. When practical, assume a simplifying mathematical expression for the cyclic stress - strain relationship.

NOTE 17—For many metals, one accepted and convenient expression of the cyclic stress - strain curve is that of a power relationship presented by Eq 1 of Appendix X1.

8.11.2 *Determination of the Strain-Life Relationship*—Generate a strain-life curve from paired values of total strain versus life or plastic strain versus life. When practical, assume a simplifying mathematical relationship.

NOTE 18—For many metals, one accepted and convenient means for representing the strain-life relationship is presented by Eq 4 of Appendix X1.

8.11.3 *Post-Mortem Examinations*—Metallographic examination of the failed specimens is desirable for a variety of purposes depending in part on user interests. Of foremost importance is a fractographic examination of the two crack surfaces to determine any unusual causes of failure that might invalidate the test results. Scanning electron microscopy and transmission electron microscopy of fracture replicas are two common methods used in such an investigation. Ref. **(6)** provides a useful basis for fractographic analysis. The techniques of light metallography and transmission electron microscopy are frequently used when studying structural changes that occur during fatigue or the effects of metallurgical structure on fatigue behavior.

9. Report

9.1 The list of items of information that follows is suggested for inclusion in any report. When publishing results in the open literature, include as much information as possible, independent of the author's purpose. Routine laboratory reports need include only information pertinent to the end use of the test data. Minimum recommended requirements are indicated by an asterisk.

9.1.1 Specific objective of testing.

9.1.2 *Specimen Materials Description* (including processing):

9.1.2.1 All available mechanical properties including: yield strength or yield point, or both, ultimate tensile strength, percent elongation, percent reduction of area, Poisson's ratio, elastic modulus, true fracture strength, true fracture ductility, static strain hardening exponent, static strength coefficient, hardness number, and degree of cold work.

9.1.2.2 All available metallurgical characteristics: certified chemical composition, grain size, crystallographic structure, preferred orientation with respect to specimen axes, general shape of grains (that is, equiaxed or elongated), second phase particles, and heat treatment. Include photomicrographs when possible to document the above properties.

9.1.3 *Specimen Description:*

9.1.3.1 Drawing of specimen design*.

9.1.3.2 Specimen machining and surface preparation procedures*.

9.1.3.3 Deviations from recommended specimens and specimen preparation procedures if any.

9.1.4 *Description of Equipment:*

9.1.4.1 Specimen fixtures.

9.1.4.2 Testing machine*.

9.1.4.3 Transducer systems (that is, load transducers, deformation transducers).

9.1.4.4 Recorders and recording equipment.

9.1.4.5 Deviations from recommended equipment, if any.

9.1.5 *Description of Testing Environment*:*

9.1.5.1 Gas, liquid, or vacuum; chemical composition of medium*.

9.1.5.2 Humidity of gaseous environment*.

9.1.5.3 Test temperature and temperature control method*.

9.1.5.4 Temperature variations or deviations, or both, from those specified.

9.1.6 *Testing Conditions and Procedure:*

9.1.6.1 Deviations from recommended procedures, if any.

9.1.6.2 Frequency of cycling (or cyclic strain rate) and description of waveform*.

9.1.6.3 Mode of control, that is, load or stress, continuous strain control, strain limit control, axial strain feedback, diametral strain feedback, etc*.

9.1.6.4 Ratio of axial strain limits (minimum and maximum) and total axial strain range*.

9.1.6.5 Procedure for maintaining constant axial strain limits.

9.1.6.6 Sign of strain at first quarter cycle, tensile or compressive.

9.1.7 *Test Results*—Tabulate the results for all test specimens. When used for purposes of structural analysis, the following two items are most important:

9.1.7.1 Initial, stabilized, or half-life values, or a combination of all three, of the dependent variables from the list of stress range, strain range, and plastic strain range. Complete curves of these values throughout the specimen life. When complete curves are impractical, curves through intermittent values are acceptable*.

9.1.7.2 Total number of cycles to failure, N_f, and failure definition used in the determination of N_f*.

9.1.7.3 Order in the sequence in which each specimen was tested.

9.1.8 Results of analysis for cyclic stress - strain properties. If data analysis is performed by means of the relationships in Appendix X1, a tabulation of results should include the cyclic strain hardening exponent and the cyclic strength coefficient.

9.1.9 Results of analysis for strain-life properties. If data analysis is performed by means of the relationships in Appendix X1, a tabulation of results should include the fatigue strength exponent, the fatigue ductility exponent, the fatigue strength coefficient, and the fatigue ductility coefficient.

9.1.10 Brief description of the fracture characteristics; results of post-test metallography and scanning electron microscopy; identification of fracture mechanism and the relative degree of transgranular and intergranular cracking.

REFERENCES

(1) Raske, D. T., and Morrow, JoDean, "Mechanics of Materials in Low Cycle Fatigue Testing," *Manual on Low Cycle Fatigue Testing, ASTM STP 465,* Am. Soc. Testing Mats., 1969, pp. 1–25.

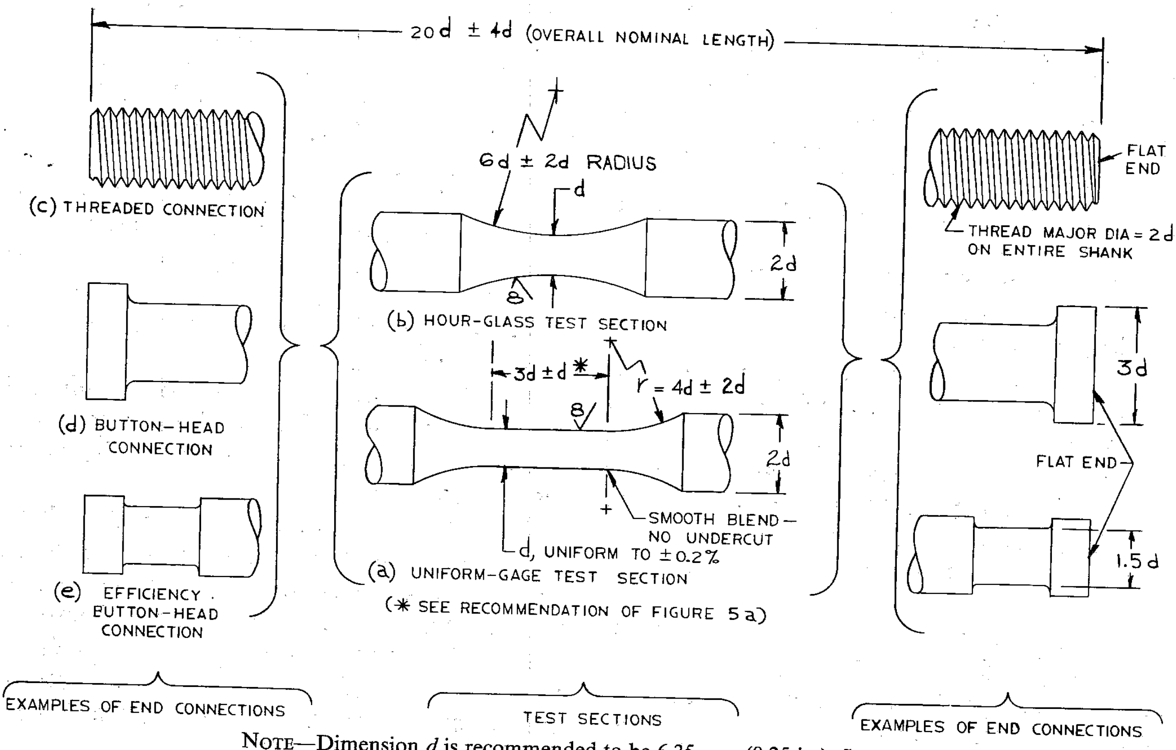
(2) Feltner, C. E., and Mitchell, M. R., "Basic Research on the Cyclic Deformation and Fracture Behavior of Materials," *idem,* pp. 27–66.

(3) Marsh, G. M., Robb, A. D., and Topper, T. H., "Techniques and Equipment for Axial Fatigue Testing of Sheet Steel," SAE Paper No. 730578, May 1973.

(4) Little, R. E., and Jebe, E. H., *Manual on Statistical Planning and Analysis for Fatigue Experiments, ASTM STP 588,* Am. Soc. Testing Mats., 1975. See also Little, R. E. and Jebe, E. H., *Statistical Design of Fatigue Experiments,* Applied Science Publishers, Ltd., London, 1975.

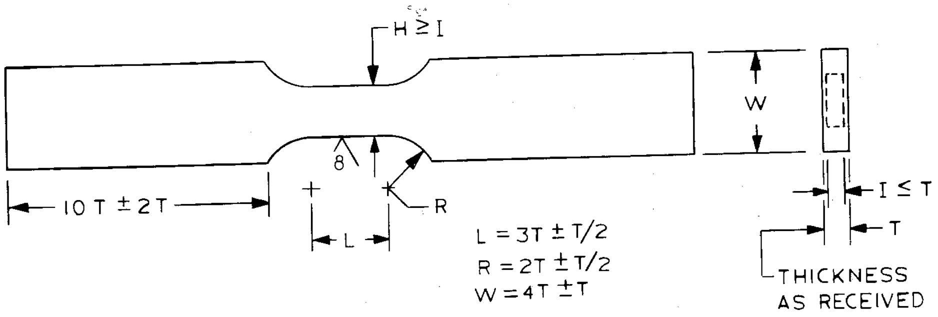
(5) Landgraf, R. W., Morrow, JoDean, and Endo, T., "Determination of the Cyclic Stress - Strain Curve," *Journal of Materials,* JMLSA, Vol 4, No. 1, March 1969, pp. 176–188.

(6) Metals Handbook, *Fractography and Atlas of Fractographs,* Am. Soc. Metals, Vol 9, Eighth Edition, 1974.



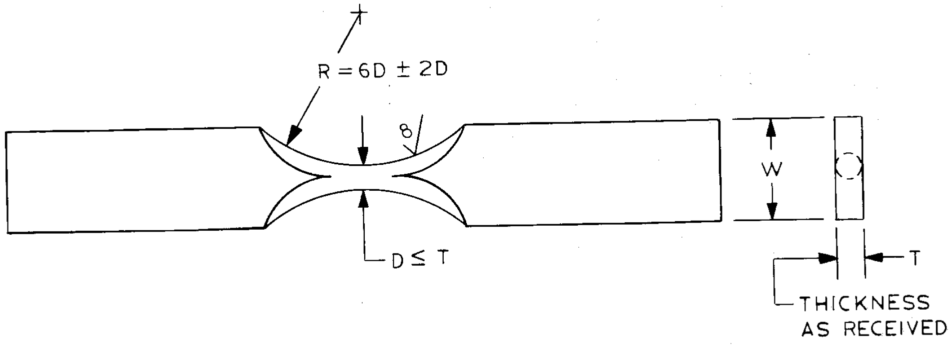
NOTE—Dimension *d* is recommended to be 6.35 mm (0.25 in.). See 7.1.

FIG. 1 Recommended Low-Cycle Fatigue Specimens.



NOTE—2.54 mm (0.1 in.) ≤ I ≤ T.

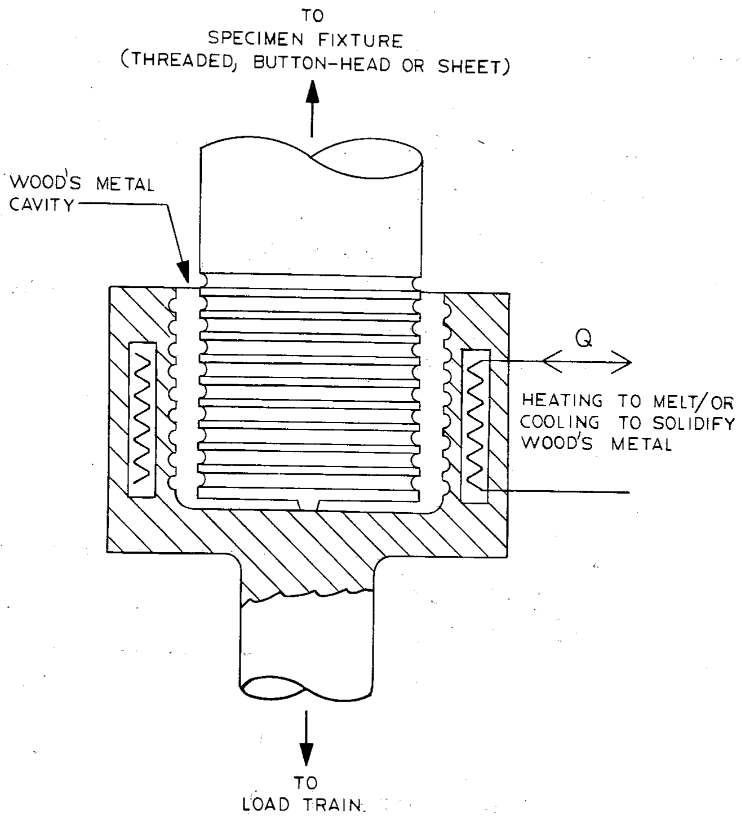
(*a*) Flat-Sheet Fatigue Specimen with Rectangular Cross Section.



NOTE—2.54 mm (0.1 in.) ≤ D.

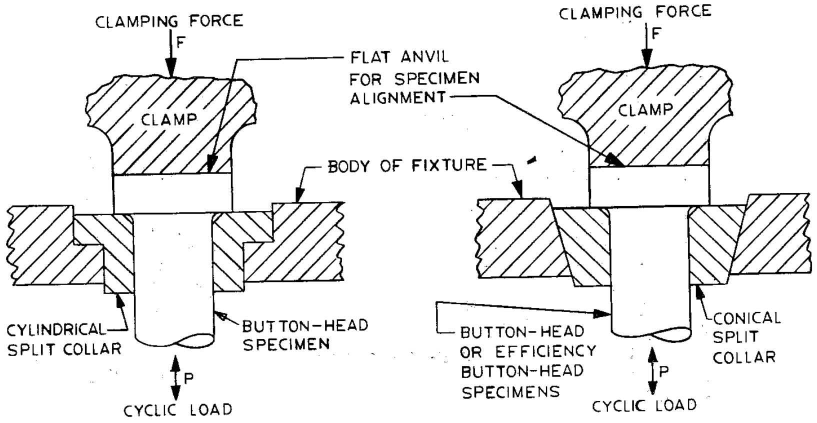
(*b*) Flat-Sheet Fatigue Specimen with Circular Cross Section.

FIG. 2 Sheet Fatigue Specimens—Alternative to Fig. 1 Specimens.

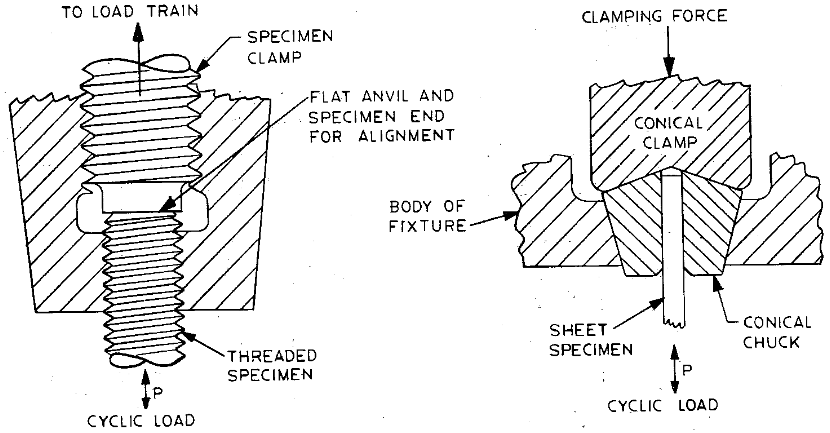


NOTE—Wood's metal pot is used to provide initially zero stress in the specimen during fixturing. This pot may be placed within a die-set to combine zero fixturing stress with rigid alignment.

FIG. 3 **Schematic of Wood's Metal Pot Showing Principle of Operation.**



(*a*) Button-Heat Fixture. (*b*) Button-Head or Efficiency Button-Head Fixture.



(*c*) Threaded Specimen Fixture. (*d*) Sheet Specimen Fixture.

NOTE—The clamping force should be greater than the cyclic load to avoid backlash within the specimen fixture.

FIG. 4 Schematic Examples of Fixturing Techniques for Various Specimen Designs.

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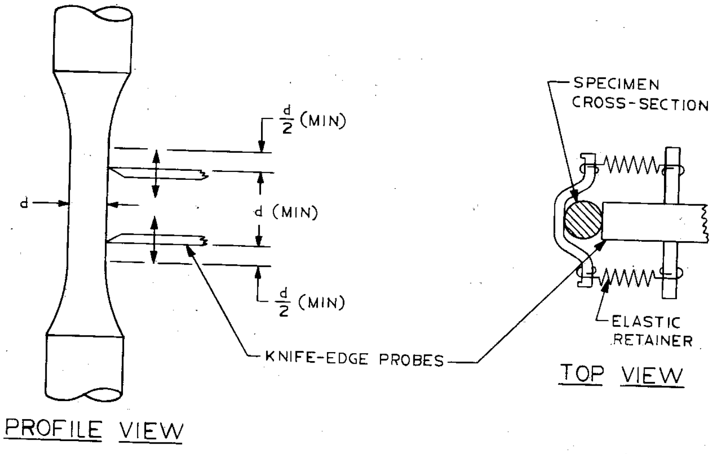


FIG. 5(*a*) **Longitudinal Displacement Measurement for Uniform Gage Specimen of Fig. 1(*a*). (Probes may Be Attached to Either Transducer 5(*c*) or 5(*d*).)**

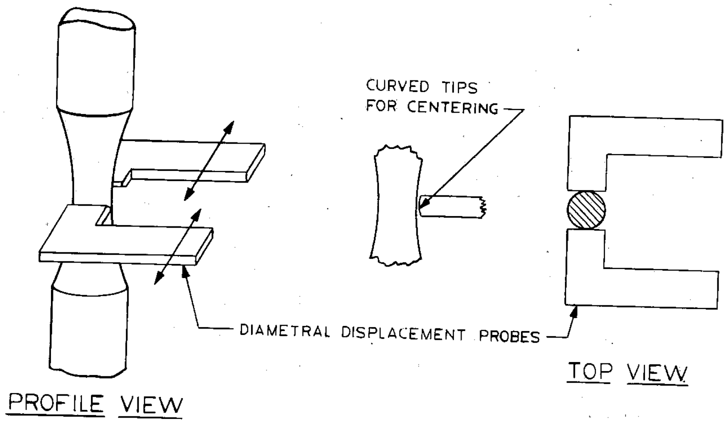


FIG. 5(*b*) **Diametral Displacement Measurement for Hourglass Specimen of Fig. 1(*b*). (Probes may Be Attached to Either Transducer 5(*c*) or 5(*d*).)**

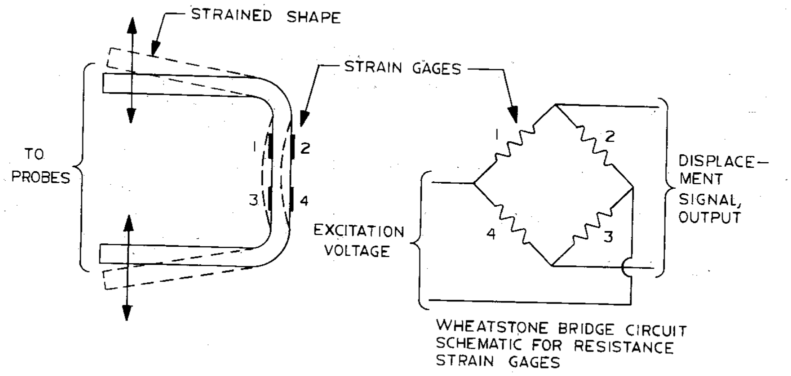


FIG. 5(*c*) **Strain-Gage Displacement Transducer.**

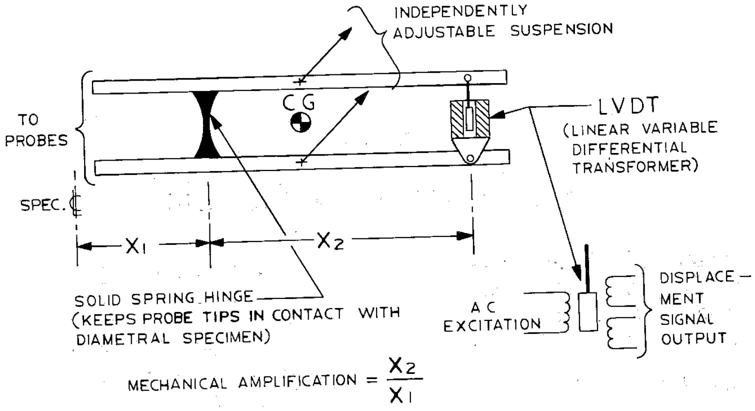


FIG. 5(*d*) LVDT Displacement Transducer.

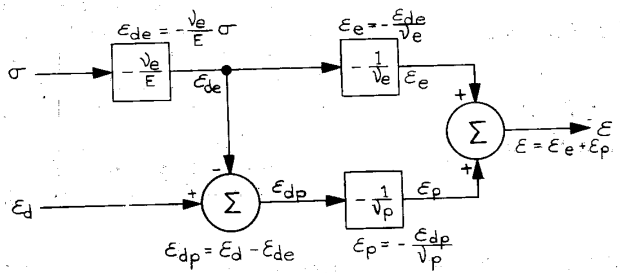
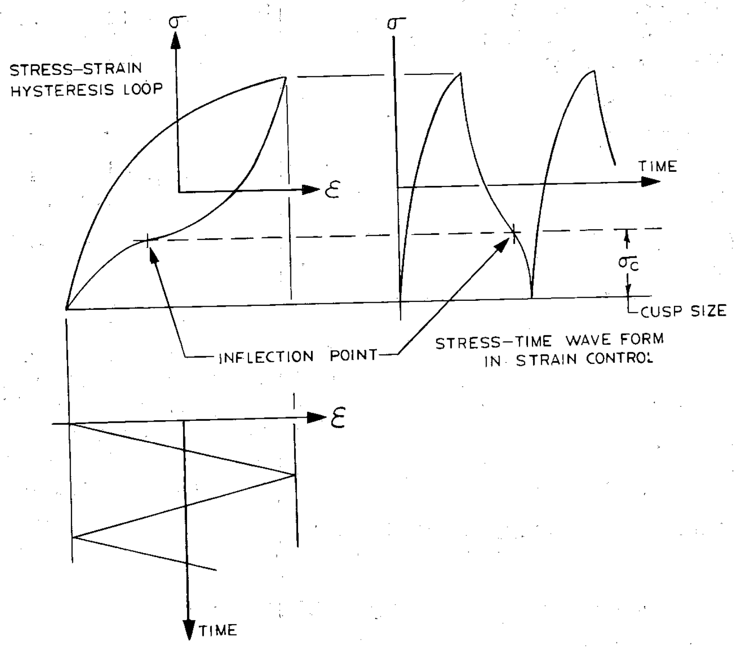


FIG. 6 Block Diagram of Strain Computer (See Appendix X2 for Discussion of Mathematical Relationships).



NOTE—The inflection point for a *stress-control* test may be similarly identified on the hysteresis loop and the *strain-time* diagram.

FIG. 7 Definition of "Cusp Size" for a Determination of Failure (See 8.9.3).

APPENDIXES

X1. FUNCTIONAL RELATIONSHIPS

X1.1 For many metals, the following empirical relationships (**1**) have been used for a convenient description of low-cycle fatigue data:

X1.1.1 *Cyclic Stress - Strain Behavior:*

$$\Delta\sigma/2 = K'(\Delta\epsilon_p/2)^{n'} \qquad (1)$$

Note that Eq 1 may be expressed in terms of total strain range, $\Delta\epsilon$, by recognizing that $\Delta\epsilon = (\Delta\sigma/E) + \Delta\epsilon_p$.

X1.1.2 *Fatigue-Life Relationships:*

$$\Delta\sigma/2 = \sigma_f'(2N_f)^b \qquad (2)$$

$$\Delta\epsilon_p/2 = \epsilon_f'(2N_f)^c \qquad (3)$$

$$\Delta\epsilon/2 = [\sigma_f'/E](2N_f)^b + \epsilon_f'(2N_f)^c \qquad (4)$$

where the variables are:

$\Delta\sigma$ = true stress range,
$\Delta\epsilon$ = true strain range,
$\Delta\epsilon_p$ = true plastic strain range,
N_f = cycles to failure, and
$2N_f$ = reversals to failure;

and the constants are:

n' = cyclic strain hardening exponent,
b = fatigue strength exponent,
c = fatigue ductility exponent,
K' = cyclic strength coefficient,
σ_f' = fatigue strength coefficient,
ϵ_f' = fatigue ductility coefficient, and
E = modulus of elasticity.

NOTE—The user is cautioned that Eqs 2 to 4 are not readily amenable to the use of conventional statistical analysis procedures.

X2. CONVERSION FROM DIAMETRAL STRAIN TO AXIAL STRAIN FOR ISOTROPIC MATERIALS

X2.1 Conversion of diametral strain to axial strain requires first the separation of the elastic and plastic components from the total strain by:

$$\begin{aligned} \epsilon &= \epsilon_e + \epsilon_p \\ \epsilon_d &= \epsilon_{de} + \epsilon_{dp} \end{aligned} \qquad (1)$$

where:

e = elastic components,
p = plastic components,
d = diametral components, and
ϵ = total axial strain.

Axial and diametral components of strain are related through Poisson's ratio, ν, as:

$$\epsilon_e = -\,\epsilon_{de}/\nu_e \text{ and } \epsilon_p = -\,\epsilon_{dp}/\nu_p \qquad (2)$$

The above expressions may be rearranged to yield:

$$\epsilon_{dp} = \epsilon_d - \epsilon_{de}$$

$$\epsilon = -\,\epsilon_{de}/\nu_e - (\epsilon_d - \epsilon_{de})/\nu_p \qquad (3)$$

The diametral elastic strain, ϵ_{de}, is related to axial stress by means of Poisson's ratio and modulus,

$$\epsilon_{de} = -\,(\nu_e\sigma)/E$$

Thus:

$$\epsilon = \sigma/E - \epsilon_d/\nu_p - (\nu_e\sigma)/(\nu_p)E \qquad (4)$$

Assuming that plastic deformation occurs under a constant volume condition:

$$\nu_p = 1/2$$

such that:

$$\epsilon = (\sigma/E)(1 - 2\nu_e) - 2\epsilon_d$$

In an experiment using a diametral strain gage and an axial load transducer, analogs of σ and ϵ_d are continuously available. Provided that elastic constants do not change with cyclic loading, the ratio ν_e/E can be found initially from the slope of the elastic portion of the σ versus ϵ_d curve and the modulus, E, may be determined in accordance with Method E 111.

X3. EXAMPLE OF MACHINING PROCEDURE

X3.1 While the following procedure was developed for machining high-strength materials with minimal attendant surface damage and alteration, it can be successfully applied to materials of lower strength. As a conservative general measure, this procedure is recommended unless: (*1*) the experimental objective is to evaluate another given surface condition or, (*2*) it is known that the material under evaluation is relatively insensitive to surface condition.

X3.2 **Procedure**

X3.2.1 In the final stages of machining, remove material in small amounts until 0.125 mm (0.005 in.) of excess material remains.

X3.2.2 Remove the next 0.1 mm (0.004 in.) of gage diameter by cylindrical grinding at a rate of no more than 0.005 mm (0.0002 in.)/pass.

NOTE X1—Some cast materials will not benefit from successive removal of material in small amounts, although this procedure is probably not to their detriment.

X3.2.3 Remove the final 0.025 mm (0.001 in.) by polishing (Note X2) longitudinally to impart a maximum of 0.2-μm (8-μin.) surface roughness.

NOTE X2—Extreme caution should be exercised in polishing to ensure that material is being properly removed rather than merely smeared to produce a smooth surface. This is a particular danger in soft materials wherein material can be smeared over tool marks, thereby creating a potentially undesirable influence on crack initiation during testing.

X3.2.4 After polishing (Note X2), all remaining grinding and polishing marks should be longitudinal. No circumferential machining should be evident when visualized at approximately 20× magnification under a light microscope.

X3.2.5 If specimen material is soft (for example, copper, aluminum, lead, etc.) at room temperature, final material removal can be performed by means of turning (rather than grinding) and subsequent polishing.

X3.2.6 Degrease the finished specimen.

X3.2.7 If heat treatment is necessary, conduct it before final machining or in such a manner as to avoid any surface damage; employ an inert protective atmosphere to eliminate surface oxidation.

X3.2.8 If surface observations are to be made, the test specimen may be electropolished in accordance with Method E 3.

X3.2.9 Imprint specimen numbers on both ends of the test section in regions of low stress, away from grip contact surfaces.

X4. ELEVATED-TEMPERATURE TESTING

X4.1 This Appendix is meant to provide guidance for conducting low-cycle fatigue tests at elevated temperatures where time-dependent strains can be present. Large segments of this recommended practice are already applicable to testing at these temperatures. The following paragraphs provide the necessary modifications to extend the practice for use in this regime. The numbers in parentheses refer to sections to which the paragraphs apply.

X4.1.1 Testing is permitted at temperatures and strain-rates where the magnitudes of time-dependent inelastic strains may be significant, relative to the magnitude of time-independent inelastic strains (1.3).

X4.1.2 No specific limitations are placed on the type of stress or strain cycling to which the test specimens are subjected. In particular, hold times on stress or strain are permitted (1.4).

X4.1.3 The presence of time-dependent inelastic strains during elevated temperature testing provides the opportunity to study the effects of these strains on fatigue life and on the cyclic stress-strain response of the material. Information about strain rate effects, relaxation behavior, and cyclic creep may also be available from these kinds of tests (3.3).

X4.1.4 Additional definitions will be found at the end of this appendix to cover elevated-temperature testing (4.1).

X4.1.5 The empirical relationships given in Appendix XI may not be valid when large time-dependent, inelastic strains are present. For this reason, all raw data should be reported to the extent possible. Data-reduction methods should be elaborated. If the data are also analyzed, details of the approach should be elaborated and information should be presented to permit analysis by other currently popular techniques (5.1).

X4.1.6 Satisfactory stress control cycling in the elevated temperature regime may require a greater degree of control on stress limits than specified in 6.2. Time-dependent effects can permit very large strains to be accumulated as a result of relatively slight errors in stress control. Consequently, special care should be exercised in this regime to avoid undesirable creep strain excursions. When, because of high cycle and long life considerations strain control testing is not practical, a pseudo strain control experiment under stress control is sometimes performed. In this case, it is desirable to monitor the strain during the test and to make modifications to the stress as necessary to remain within the required strain limits (6.2).

X4.1.7 For elevated-temperature testing it is usually necessary to provide some means for cooling the fixtures to prevent damage to other load-train components such as load cells. One method commonly used employs water-cooling coils attached to the fixtures or to other appropriate locations in the load train. Care must be taken to avoid affecting the load cell calibration or the load train alignment by the addition of cooling coils (6.3.2).

X4.1.8 Temperatures are permitted that may be high enough to produce significant time-dependent inelastic strains. Data should be presented in sufficient detail to facilitate their use to evaluate the utility of currently popular analysis techniques (8.1.1).

X4.1.9 When time-dependent effects are present, it may no longer be acceptable to control only the limits of the required stress or strain. Continuous control of the parameter of interest may be necessary to obtain the desired intra-cycle response. For example, if the load is controlled between total axial strain limits in this regime, a quite different material response will be produced than if the total axial strain is continuously controlled (8.3.1).

X4.1.10 Additional precautions should be observed when hold times are employed and time-dependent inelastic strains are present. For example, a hold on diametral strain will permit the total axial strain to change during each cycle and will not produce correct relaxation information (8.3.2).

X4.1.11 The additional complications of complex waveforms and time-dependent inelastic strains severely curtail the acceptability of limit control techniques. If the technique of limit control is used, the intra-cycle and inter-cycle variation of the parameter of interest should be monitored, and if necessary, periodic adjustments should be made to the testing machine to produce the desired response. Such changes should be reported (8.3.3).

X4.1.12 In addition to the preference for triangular waveforms for continuous cycling tests, trapezoidal waveforms with adjustable hold periods are needed for hold-time testing (8.4).

X4.1.13 It should be noted that if time-dependent effects are present, a reduced testing rate for recording purposes may produce significantly altered stress-strain response from that which would be produced at the normal testing rate. In addition, a possible effect of this periodic reduction of rate on specimen life should be considered (8.5.2, Note 11).

X4.1.14 No specific data analysis techniques are recommended for use when significant time-dependent inelastic strains are present (8.11).

X4.1.15 Dimensional instability of the specimen (unintentional changes in specimen geometry) may occur during the test as a result of time-dependent inelastic deformations. The post-mortem examination should include an evaluation of this occurence (8.11.3).

X4.1.16 Relaxation information should also be included for hold-time tests. This should include the values of the relaxed stress, the total amount of relaxation, and the change in the amount of inelastic strain during the hold period. Periodic stress-time recordings obtained with an expanded time scale may be necessary to obtain the desired resolution and to fully characterize the relaxation behavior (9.1.7.1).

X4.2 **Definitions**—In addition to the modification of certain sections as noted above, the following definitions will be used:

X4.2.1 *inelastic strain,* ϵ_{in}—that strain which is not elastic. For isothermal conditions ϵ_{in} is calculated by subtracting the elastic strain from the total strain.

X4.2.2 *hold period,* τ_h—a time interval within a cycle during which the stress or strain is held constant.

X4.2.3 *total cycle period,* τ_t—the time for the completion of one cycle.

Note—τ_t can be separated into hold and non-hold components, or as follows:

$$\tau_t = \Sigma\tau_h + \Sigma\tau_{nh} \tag{1}$$

where:

$\Sigma\tau_h$ = sum of all the hold portions of the cycle.
$\Sigma\tau_{nh}$ = sum of all the non-hold portions of the cycle.

τ_t is also equal to the reciprocal of the overall frequency when the frequency is held constant.

X4.2.4 *creep strain increment,* $\Delta\epsilon_c$—the change in the magnitude of the inelastic strain during a stress hold period.

X4.2.5 The following equations (refer to Fig. X1) are often used to define the instantaneous stress and strain relationships for many metals and alloys:

$$\epsilon = \epsilon_{in} + \epsilon_e, \text{ and} \qquad \epsilon_e = \frac{\sigma}{E^*}, \text{ (Note X1)} \tag{2}$$

and the change in strain from any point 1 to any other point 3 can be calculated as follows:

$$\epsilon_3 - \epsilon_1 = \left(\epsilon_{3in} + \frac{\sigma_3}{E^*}\right) - \left(\epsilon_{1in} + \frac{\sigma_1}{E^*}\right) \tag{3}$$

All strain points to the right of and all stress points above the origin are positive. The equation would then show an increase in inelastic strain from 1 to 3, or

$$\epsilon_{3in} - \epsilon_{1in} = \epsilon_3 - \epsilon_1 + \frac{\sigma_1}{E^*} - \frac{\sigma_3}{E^*} \tag{4}$$

Similarly, during the strain hold period, the change in the inelastic strain will be equal to the change in the stress divided by E^*, or

$$\epsilon_{3in} - \epsilon_{2in} = \frac{\sigma_2 - \sigma_3}{E^*} \tag{5}$$

Note X1—E^* represents a material variable that may be a function of environment and test conditions. It may also vary during a test as a result of metallurgical or physical changes in the specimen. In many instances, however, it is practically a constant quantity and is used rather extensively, in isothermal, constant-rate testing, in the analysis of hysteresis loops. A value for E^* can best be determined by cycling the specimen prior to the test at stress levels below the elastic limit.

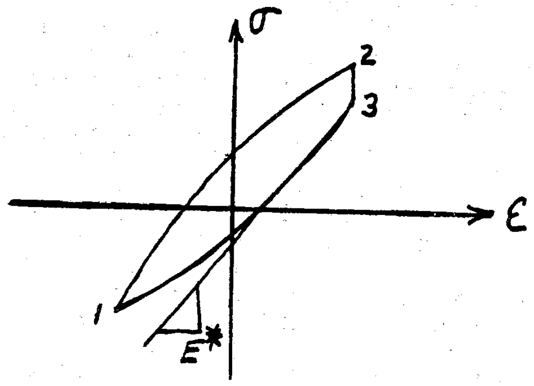


FIG. X1 Analysis of a Total Strain Versus Stress Hysteresis Loop Containing a Hold Period

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Designation: E 681 – 85

Standard Test Method for Concentration Limits of Flammability of Chemicals[1]

This standard is issued under the fixed designation E 681; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the lower and upper concentration limits of flammability of chemicals having sufficient vapor pressure to form flammable mixtures in air at one atmosphere pressure at the test temperature. This method may be used to determine these limits in the presence of inert dilution gases. No oxidant stronger than air should be used.

NOTE 1—The lower flammability limit and upper flammability limit are also referred to as the lower explosive limit (LEL) and upper explosive limit (UEL) respectively.

1.2 This test method is limited to an initial pressure of 101 kPa (1 atm) or less with a practical lower pressure limit of approximately 13.3 kPa (100 mm Hg). The maximum practical operating temperature of this equipment is approximately 150°C (302°F).

1.3 This test method is one of several being developed by Committee E-27 for determining the flammability of chemicals.

1.4 *This standard should be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and should not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.*

1.5 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precaution statements are given in Section 7.

2. Summary of Method

2.1 A uniform mixture of a gas or vapor with air is ignited in a closed vessel and the upward and outward propagation of the flame away from the ignition source is noted by visual observation. The concentration of the flammable component is varied between trials until the composition which will just sustain propagation of the flame is determined.

3. Definitions

3.1 *lower limit of flammability or lower flammable limit* (LFL)—the minimum concentration of a combustible substance that is capable of propagating a flame through a homogeneous mixture of the combustible and a gaseous oxidizer under the specified conditions of test.

3.2 *upper limit of flammability or upper flammable limit* (UFL)—the maximum concentration of a combustible substance that is capable of propagating a flame through a homogeneous mixture of the combustible and a gaseous oxidizer under the specified conditions of test.

3.3 *propagation of flame*—as used in *this method*, the upward and outward movement of the flame front from the ignition source to the vessel walls, which is determined by visual observation.

4. Significance and Use

4.1 The lower and upper limits of flammability of gases and vapors define the range of flammable concentrations in air.

4.2 Limits of flammability may be used to determine guidelines for the safe handling of volatile chemicals. They are used particularly in assessing ventilation requirements for the handling of gases and vapors.

NOTE 2—The break point between nonflammability and flammability occurs over a narrow concentration range at the lower flammable limit but is less distinct at the upper limit.

5. Interferences

5.1 This method is not applicable to certain readily oxidized chemicals. If significant oxidation takes place when the vapors are mixed with air, unreliable results are obtained. Flow systems designed to minimize hold-up time may be required for such materials.

5.2 Measured flammable limits are influenced by flame-quenching effects of the test vessel walls. The test vessel employed in this method is of sufficient size to eliminate the effects of flame quenching for most materials. However, there may be quenching effects, particularly on tests run at subambient pressures. For certain amines, halogenated materials, etc., which have large ignition-quenching distances, tests should be conducted in larger diameter vessels.

6. Apparatus

6.1 Figure 1 is a schematic diagram of the apparatus; details and dimensions are presented in Annex A1. The apparatus consists of a glass test vessel (*1*), an insulated chamber (*2*) equipped with a source of controlled-temperature air (*3*), an ignition device with an appropriate power supply (*4*), a magnetic stirrer (*5*), and a cover (*6*)

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[1] This test method is under the jurisdiction of ASTM Committee E-27 on Hazard Potential of Chemicals and is the direct responsibility of Subcommittee E27.04 on Flammability and Ignitability of Liquid Chemicals.

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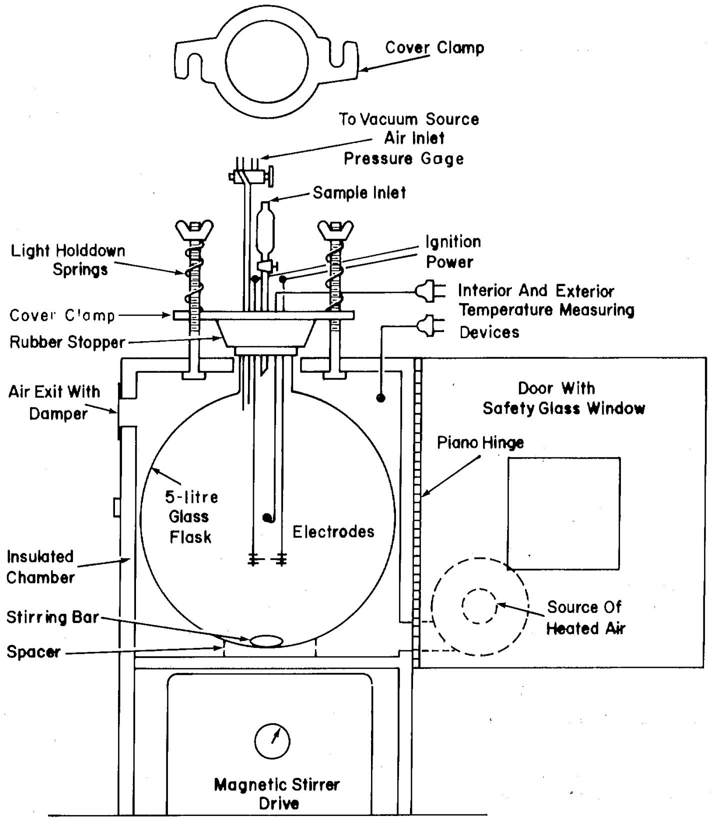

FIG. 1 Schematic Diagram of Test Apparatus

equipped with the necessary operating connections and components.

7. Safety Precautions

7.1 Tests should not be conducted in this apparatus with oxidizers stronger than air since explosive violence increases as oxidizer strength increases. Do not use oxygen, nitrous oxide, nitrogen dioxide, chlorine, etc., in this glass apparatus.

7.2 Adequate shielding must be provided to prevent injury in the event of equipment rupture, due to both implosions and explosions. A metal enclosure such as that recommended in A1.2 is one method suitable for this purpose.

7.2.1 Implosion of the test vessel at high vacuum levels is possible and, therefore, all evacuations must be made with the required shielding to protect against flying fragments.

7.2.2 Energetic explosions may be produced if tests are made at concentrations within the flammable range, between the LFL and UFL. The glass test vessel, equipped with a lightly held or loose cover, vents most explosions adequately. Nevertheless, shielding is required to protect against any probability of test vessel rupture. Methods for estimating initial test concentrations, discussed in Annexes A2, A3, and A4 may be employed to ensure that initial trials are conducted at concentrations less than the LFL or greater than the UFL.

7.2.3 In rare instances, particularly with upper limit tests, self-ignition may be encountered when air is rapidly introduced into the partially evacuated test vessel containing vaporized sample. Valves permitting remote operation, changes in sample and air introduction sequences, simple shields, and other techniques may be employed to ensure safe operations.

7.2.4 The test area should be equipped with electrical interlocks to prevent activation of the ignition source unless adequate shielding is in place.

7.3 Tests should not be conducted on thermally unstable materials which might undergo explosive decomposition reactions.

7.4 Tests should be conducted in a fume hood or other ventilated area to prevent personnel exposure to toxic chemicals or combustion products.

7.5 Precautions must be taken to ensure that the high-voltage spark ignition source does not contact temperature or pressure-measuring devices or other conductive paths which could create an electrical hazard to personnel or instrumentation outside the shielded area. Careful attention to electrical insulation integrity can reduce the possibility of hazard. Disconnects for all instrumentation lines will provide positive protection.

8. Equipment Calibration

8.1 Accurate determination of the flask volume is necessary for the calculation of flammable limits when sample measurement is on a weight or volume basis.

8.1.1 Determine the total volume of the flask as follows: Weigh a clean, dry flask with all components installed. Fill the flask with distilled water. Reinsert the cover, allowing the excess water to overflow, dry the outside of the flask, and reweigh. Record the difference in grams as the net volume of the flask in cubic centimetres. (Slight errors associated with water density differences are beyond the accuracy of this method.)

8.2 Calibrate pressure-, temperature-, and liquid-measuring devices against adequate standards.

9. Procedure

9.1 Assemble the equipment, as in Fig. 1, within an appropriate fume hood or other ventilated area and secure the door of the metal enclosure. The test vessel and all components should be clean and dry. Evacuate the system and flush with air to ensure removal of residual volatile materials that may be present as a result of cleaning or prior tests. As many as three evacuation/flush cycles may be required to ensure complete removal of combustion products between tests.

9.2 Adjust the flask to the desired test temperature. This temperature must be above the vapor condensation temperature of the mixture being tested.

9.2.1 It may be necessary to heat or insulate cover components and feed lines separately to prevent vapor condensation.

9.3 Record the actual barometric pressure at the test location.

9.4 Double check to make certain all safety precautions have been taken.

9.5 *Procedure for Sample Introduction as a Liquid:*

9.5.1 Evacuate the flask to a pressure of 1.33 to 6.65 kPa (10 to 50 mm Hg).

9.5.2 Place the desired liquid volume in a hypodermic syringe of appropriate size. Liquid volumes for initial trials may be estimated by methods given in Annex A2. Transfer the liquid to the inlet separatory funnel (see 9.5.4.1).

9.5.3 Turn on the stirrer at a minimum speed of 400 rpm.

9.5.4 Open the inlet stopcock. Allow the sample to be drawn into the flask. Close the stopcock when all the liquid has entered. Place a cover on the inlet separatory funnel.

9.5.4.1 A serum-bottle septum may be used in place of the separatory funnel. In this case, inject the sample directly into the flask by piercing the septum with the hypodermic needle. It will be necessary to make a volume correction if a significant volume of liquid is drawn from the needle or uncalibrated portion of the syringe.

9.5.5 When sample vaporization is complete, remove the separatory funnel cover and open the stopcock, permitting air to enter the test vessel slowly through the separatory funnel (see 7.2.3). Entering air sweeps traces of residual sample into the flask.

9.5.6 Release the cover hold-down devices and close the hood door.

9.5.7 Continue stirring for at least 5 min to obtain complete mixing and attainment of thermal equilibrium. Final trials should be made at longer mixing times to ensure optimal mixing conditions are achieved.

9.5.8 Turn off the stirrer.

9.5.9 Record the test temperature, T.

9.5.10 Disconnect instrumentation lines as required.

9.5.11 Darken the viewing area. Activate the ignition source. Observe for ignition and flame propagation away from the ignition source. Record as flammable any mixture producing a flame front that spreads from the ignition source and reaches the vessel walls.

NOTE 3—Mixtures having a composition just outside the flammable range exhibit a small cap of flame above the arc position; in some cases a vertical streak of flame may propagate to the vessel cover. (Absence of a cap flame may be an indication of insufficient ignition energy.) The onset of spherical, upward, and partial outward flame propagation signifies a limit or near-limit mixture. It is suggested that detailed observations of flame behavior be recorded on all trials. Include such notes as flame cap, no flame cap, upward and outward propagation, downward propagation, etc. These observations can serve as a guide to narrowing the region of uncertainty between go and no-go trials.

9.5.12 Vary sample size as required to find the minimum sample size, L_1, that gives flame propagation and the maximum sample size, L_2, below L_1, that does not give flame propagation. (The difference between L_1 and L_2 is a measure of the variability of the procedure for the material being studied.)

9.5.13 If numerous trials are required for a given series of tests, it may be necessary to remove the vessel for cleaning periodically, particularly for upper limit studies.

9.5.14 Final trials shall be made in a clean vessel.

NOTE 4—Ignition failures and inconsistent performance are occasionally encountered when highly conductive or very high ignition energy materials are tested using a spark ignition source. Limits for these materials should be determined using a fuse wire ignition source. Fuse wire ignition should also be used to confirm reduced pressure limit values arrived at on the basis of spark ignition source trials.

9.5.15 Record the values of the sample volumes L_1 and L_2. If partial propagation occurs over a range of sample sizes greater than 10 % of the sample size, the range should be specified in the report, for example, LFL = 5.4 ± 0.6 %).

9.5.16 Commence upper limit tests at a concentration greater than U_2, as defined in 9.5.17. Sample sizes for initial trials may be determined by methods given in Annex A3.

9.5.17 Record the values for the greatest sample quantity, U_1, that will propagate a flame and the least quantity, U_2, above U_1 that will not propagate a flame.

9.6 *Procedure for Sample Introduction as a Vapor:*

9.6.1 Sample concentration can be measured for gases and readily vaporized liquids on the basis of vapor pressure. In this instance, equip the vessel with a pressure transducer capable of reading to the nearest 0.0667 kPa (0.5 mm Hg). The system must also be capable of maintaining a vacuum of 0.0667 kPa or less.

9.6.2 Evacuate vessel and sample lines to a pressure of 0.0667 kPa (0.5 mm Hg or less).

9.6.3 Introduce the sample as a vapor through an appropriate inlet valve until the desired pressure is achieved. Introduce air as in 9.5.5, raising the pressure to atmospheric.

9.6.4 Carry out steps 9.5.6 through 9.5.17 as needed.

9.7 *Procedure for Sample Introduction as a Solid:*

9.7.1 Chemicals having melting points above room temperature but which totally melt and vaporize or totally sublime at the test conditions may be added to the test vessel as solids.

9.7.2 Bring the test vessel to atmospheric pressure (prior evacuation must be employed, as in 9.1, to ensure cleanliness).

9.7.3 Place the desired sample weight in the flask by raising the cover and inserting the sample.

9.7.4 Carry out steps 9.5.6 through 9.5.17 as needed.

NOTE 5—A small portion of the sample may be lost from the test vessel as the sample vaporizes and warms up to the test temperature. Losses are minimized by delaying the start of stirring until vaporization is complete. Maximum theoretical sample loss, which is small, may be readily calculated.

10. Calculations

10.1 Calculate the sample quantity, L or U, as follows:

$$L = \tfrac{1}{2}(L_1 + L_2) \tag{1}$$

$$U = \tfrac{1}{2}(U_1 + U_2) \tag{2}$$

where:

L = sample quantity used to calculate the lower flammable limit by Eq 3, and

U = sample quantity used to calculate the upper flammable limit by Eq 3.

For L_1 and L_2, see 9.5.12 . For U_1 and U_2, see 9.5.17.

10.2 Calculate the lower (LFL) and upper (UFL) flammable limits from the sample quantities. Ideal vapor phase behavior is assumed. (See Appendix X5.2 for a sample calculation and X5.1 for development of Eq 3.)

10.2.1 *Liquid Samples* (ideal vapor phase behavior is assumed):

$$\text{LFL} = \frac{(L_v)(d)(T)}{(\text{MW})(P)} \times \frac{(22.4)(P_o)(100)}{(V)(T_o)} \tag{3}$$

where:

LFL = lower flammable limit in mole or volume %,

L_v = L = sample volume from Eq 1, cm^3,

d = sample density, g/cm^3,

T = test temperature, K,

MW = sample molecular weight, g, and

P = test pressure, absolute, kPa (mm Hg).

10.2.1.1 The second term is a constant for a given test apparatus where: P_o = standard pressure (760 mm Hg), V = vessel volume (litres), and T_o = standard temperature (273 K). (Any set of consistent units may be used for these calculations.)

10.2.1.2 Calculate upper flammable limits by replacing LFL with UFL and L_v with U_v in Eq 3.

10.2.2 *Vapor Samples* (ideal vapor phase behavior is assumed):

$$\text{LFL} = (L_p/P) \times 100 \tag{4}$$

where:

$L_p = L$ = sample partial pressure (mm Hg) from Eq 1.

10.2.2.1 Calculate upper limits by replacing LFL with UFL and L_v with U_v.

10.2.3 *Solid Samples* (ideal vapor phase behavior is assumed):

10.2.3.1 Calculate lower flammable limits by using Eq 3 with the terms (L_v) (d) replaced by L_w where: $L_w = L$ = sample weight (g) from Eq 1.

10.2.3.2 Calculate upper flammable limits by replacing LFL with UFL and L_w with U_w.

10.3 *Complex Liquids, Solids, and Mixtures*—Flammability limits of some materials cannot be calculated in terms of mole or volume percent (Eq 3) since the molecular weight of the vapors is not known. This occurs in the case of unknown materials, multicomponent mixtures and materials exhibiting nonideal vapor phase behavior. It is more meaningful to express these limits in terms of weight of combustible per unit volume of mixture (mg/litre).

NOTE 5—Such limits are often given in the literature[2] as weight of combustible per litre of air at standard conditions (0°C and 760 mm Hg). These limits may be calculated from the following expression or by a similar expression for UFL:

$$\text{LFL, mg/litre} = \frac{\text{LFL(volume \%)}}{[100 - \text{LFL(volume \%)}]\left[\dfrac{22.414(\text{litre})}{\text{MW (mg)}}\right]}$$

10.3.1 Calculate lower flammable limits of mixed vapors and materials exhibiting nonideal vapor phase behavior as follows:

$$\text{LFL}_w = \frac{L_w}{V} \quad \text{or} \quad \frac{L_v(d)}{V} \times 100 \tag{5}$$

where: LFL$_w$ = lower flammable limit, in milligrams per litre.

10.3.2 Calculate upper flammable limits using Eq 5 replacing LFL$_w$ with UFL$_w$, L_w with U_w, and L_v with U_v.

11. Report

11.1 Report flammability limits, LFL and UFL, calculated in accordance with Eqs 3, 4 or 5, along with the test temperature, test pressure, and ignition source (spark or fuse wire) used.

11.2 Report the limits initially in accordance with the units of measurement used in the determinations, that is, on a volumetric basis (mole or volume percent) for gases or vapor samples and on a gravimetric basis (milligrams per litre) for liquid or solid samples.

11.3 By substitution in Eq 3, calculated limits may then also be given for the gases or vapors on a gravimetric basis and for the liquids or solids on a volumetric basis, provided molecular weights of the combustibles are known. The report shall note if nonideal vapor phase behavior is suspected or known to occur.

11.4 Report the test variability if it exceeds 10 % of the sample size (see 9.5.1.5).

12. Precision and Bias

12.1 The precision and bias of this method have not yet been established.

12.2 The minimum precision of the method shall be ±0.5 % for limits of 10 volume %. Test data available at present are inadequate to establish any measure of repeatability or reproducibility.

ANNEXES

(Mandatory Information)

A1. DIMENSIONS AND SPECIFICATIONS OF APPARATUS (Fig. 1)

A1.1 *Test Vessel*—The test vessel shall be a borosilicate glass boiling flask, short-ring neck, 5000-cm^3 capacity, approximately 222 mm (8¾ in.) in diameter and 305 mm (12 in.) in height.

A1.2 *Insulated Chamber*—The dimensions shall be as follows:

Inside: 279 by 279 by 305 mm (11 by 11 by 12 in.) high
Height: 483 mm (19 in.). Adjust to accommodate stirrer unit.
Rear panel: ≥200 by 200-mm vent area
Top hole: 70.0-mm (2¾-in.) diameter
Air inlet hole: to fit air supply unit
Air exit hole: to accommodate a simple slide damper

A1.2.1 *Materials*—Sheet metal of at least 16 gage covered with insulation. Generally, a portion of the metal bottom must be partially removed and replaced with nonmagnetic material to permit operation of the magnetic stirrer. The rear panel should be equipped with a vent (≥ 200 by 200 mm) providing explosion relief at low over-pressures, ≤6.9 kPa (1 psi). A lightly held panel of insulating board may be used.

A1.2.2 *Door,* hinged and latched, fitted with a 102 to 127-mm (4- to 5-in.) square safe viewing window made of polycarbonate and at least 12.7-mm (½-in.) thickness or equivalent.

A1.2.3 *Bolts,* top-fitted with two ¼–20 bolts on 127-mm (5-in.) centers to secure test vessel cover.

A1.2.4 *Spacer*—A cylindrical spacer constructed of perforated metal and sized to position the top of the neck of the test vessel just above the top of the test chamber permits air circulation and facilitates insertion and removal of the test vessel.

NOTE A1.1—If heavy construction is employed for the front, top, and side walls of the chamber and in the front of the base area, and if the rear and bottom panels of the chamber are of lightweight materials, explosion venting will be to the rear, away from the operator, in the event of vessel rupture.

A1.2.5 *Alternatives*—Other thermostated chambers or ovens and heating means may be employed if they permit temperature control and proper test manipulation and observation with adequate safety.

A1.3 *Heater*—Heated air is supplied from a blower, at the rate of approximately 0.38 m^3/min (13.5 ft^3/min), feeding air through a variable electric heater of approximately 2400 W. Commercial blowers, heaters, and manual or automatic controls and combinations thereof are available.

A1.4 *Ignition Device:*

A1.4.1 *Electrode Rods,* 3.175 to 4.76-mm (⅛ to 3⁄16-in.) diameter stainless steel, 317.5 mm (12½ in.) long. The upper ends are threaded for connection to a high-voltage source and the lower ends are threaded for attachment of spark gap points, or fuse wire, or both. Electrode rods are threaded for attachment of spark gap points, or fuse wire, or both. Electrode rods are spaced at least 32 mm (1¼ in.) apart. Other materials of construction may be used as needed.

A1.4.2 *Spark Gap,* having 6.4-mm (¼-in.) electrode spacing. Gap electrode extensions may be fabricated of platinum or tungsten wire held in wire connector lugs.

A1.4.3 *Fuse Wire*—19-mm (¾-in.) loop of 40-gage copper wire attached to threaded electrode rods in place of spark gaps.

A1.4.4 *Power,* approximately 30 mA at 15 kV, supplied by the secondary of a 120-V, 60-Hz luminous tube transformer, or by an equivalent device. Power for the fuse wire is 120 V, 60 Hz.

A1.4.5 *Timer,* to limit spark duration to 0.2 to 0.4 s. Commercial interval timers are available.

A1.5 *Stirring Devices:*

A1.5.1 *Stirring Bar,* 63.5-mm (2½-in.) egg-shaped, plastic-coated, magnet bar.

A1.5.2 *Drive*—Laboratory magnetic stirrer capable of functioning through the bottom of the test chamber and vessel.

A1.6 *Test Vessel Cover*—The cover can be constructed of a No. 14 rubber stopper with necessary holes for electrodes, sample inlet device, air inlet and evacuation connection and temperature-measuring device (see Fig. 1). *It is important to note* that the stopper rests on top and not inside the neck of the flask in order to facilitate venting.

NOTE A1.2—It is possible to operate at temperatures greater than 150°C (302°F) and to obtain more positive vacuum sealing through the use of specially constructed metal covers. High temperature O-ring seals for the flask top and inlet separatory funnel, and ceramic feedthroughs for the spark ignition source may be employed.

A1.7 *Cover Retainer* (Fig. 1)—This device held in place with wing nuts, light springs, and ¼–20 bolts can improve vacuum tightness of the test vessel when used to clamp down on the vessel cover.

A1.8 *Temperature Measurement:*

A1.8.1 Thermocouple, thermistor, resistance thermometer or other device may be used. High response speed is not required but is desirable in order to sense the heating effects associated with air introduction (7.2.3).

NOTE A1.3—Certain bare wire thermocouples may cause catalytic oxidation of test vapors, as evidenced by a persistent high-temperature excursion of the thermocouple junction. If this occurs, other thermocouple materials should be employed.

A1.8.2 A temperature-measuring device outside the test vessel but in the heating chamber or inlet air stream can aid in controlling test temperature.

A1.9 *Pressure Measurement*—Any pressure-measuring system accurate to 0.067 kPa (0.5 mm Hg) in the range from 0.067 to 101.3 kPa (0.5 to 760 mm Hg) and capable of being operated at temperatures greater than the condensation temperature of the materials under test is adequate for this unit. Active volume outside the test vessel itself should be held to a minimum since all components must be above the condensation temperature of the materials being tested. Electrical heating tapes may be employed for heating components to the desired temperature.

A2. ESTIMATION OF LOWER FLAMMABLE LIMITS FOR THE PURPOSE OF SELECTING SAMPLE SIZE FOR INITIAL TESTS

A2.1 It is the responsibility of the operator to ensure that adequate safety measures are employed in selecting sample sizes and in running the flammability limit tests. These guidelines are intended to assist the operator in planning the sequence of testing to avoid mixture compositions that may cause test vessel failure.

A2.2 To avoid energetic reactions, it is important that lower limit tests commence at a concentration below the lower flammable limit. Estimated lower flammable limits may be used in conjunction with Eq 3 to calculate a starting sample size.

A2.2.1 Several methods of estimating lower flammable limits are listed below. The accuracy of these methods varies, with some being relatively precise for certain classes of chemicals. Experience with these methods will improve the ability to make a reasonable evaluation as to the precision of the estimate with various types of material.

A2.3 Lower flammable limits of most organic chemicals are in the range from 40 to 60 mg/litre,[2] (see Note 5). For most materials, a sample size equivalent to 35 mg/litre may be used for initial tests. However, reactive fuels such as hydrogen and diborane have lower limits considerably below 30 mg/litre. As has been emphasized, lower limit tests should be conducted with extreme caution and upper limit tests should not be conducted in this apparatus on such materials (see 7.2).

A2.4 Lower flammable limits may be estimated from closed-cup flash point and vapor pressure data. The following approximate relationship may be employed:

$$\frac{P_f}{P_o} \times 100 = \mathrm{LFL_e} \qquad \text{(A1.1)}$$

where:

$\mathrm{LFL_e}$ = estimated lower flammable limit, volume %,
P_f = vapor pressure of combustible at the closed-cup flash point, mm Hg,
P_o = standard atmospheric pressure = 760 mm Hg.

A2.4.1 The validity of the flash point and vapor pressure data must be established in order to ensure a reasonable estimate of the lower flammable limit.

A2.4.2 The closed-cup flash point may not represent the lowest temperature at which a material evolves flammable vapor. Therefore, a safety factor must be employed when using this method.

A2.5 Lower flammable limits may be estimated as a function of the stoichiometric composition;[2,3] that composition where complete combustion to CO_2 and H_2O and conversion of the halogens, X, to HX consumes all the oxygen in the system.

A2.5.1 The stoichiometric composition may be calculated making use of balanced chemical equations for the combustion reaction.

A2.5.2 For combustion in air, this calculation reduces to the following equation:

$$C_s = \frac{100}{1 + 4.773\left[n + q + \frac{(m - k - 2p)}{4}\right]} \qquad \text{(A1.2)}$$

where:

C_s = stoichiometric composition of the combustible in air or mol %,
n = number of carbon atoms in the molecule,
m = number of hydrogen atoms in the molecule,
p = number of oxygen atoms in the molecule,
q = number of nitrogen atoms in the molecule, and
k = number of halogen atoms in the molecule.

A2.5.3 For saturated compounds containing only carbon, hydrogen, and oxygen, the lower flammable limit equals approximately 0.54 times the stoichiometric composition.[2,3]

$$\mathrm{LFL_e} = 0.54\, C_s \qquad \text{(A1.3)}$$

A2.5.4 For saturated compounds other than those containing carbon, hydrogen, and oxygen, lower flammable limits are generally greater than 0.54 times the stoichiometric composition.[2] Since data are limited and values vary, it is recommended that the factor 0.54 be used in calculating lower flammable limits from calculated stoichiometric composition for all saturated, nonreactive hydrocarbons.

A2.6 Lower flammable limits may also be estimated on the basis of correlations of known lower flammable limits of materials which are members of the same homologous series.

A2.7 Lower flammable limits of known mixtures may be estimated from known lower flammable limits of the mixture components using Le Chatelier's law. Details of these calculations can be found in *Bulletin 627*[2] and other references.

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[2] Zabetakis, M. G., "Flammability Characteristics of Combustible Gases and Vapors," *Bulletin 627*, U.S. Bureau of Mines, XMBUA, 1965.

[3] Hilado, C. J., "A Method for Estimating Limits of Flammability," *Journal of Fire and Flammability*, Vol 6, April 1965, p. 130.

A3. ESTIMATION OF UPPER FLAMMABLE LIMITS FOR THE PURPOSE OF SELECTING SAMPLE SIZE FOR INITIAL TESTS

A3.1 Upper limits cannot be predicted as precisely as lower limits. Therefore, wide safety factors should be employed when using estimated upper limits to establish sample sizes for initial flammable limit tests (see Section 7).

A3.2 For saturated compounds containing only carbon, hydrogen, oxygen, and halogens, the upper flammable limit is generally greater than 3.5 times the stoichiometric composition.[3]

$$\mathrm{UFL_e} \geq 3.5\ \mathrm{C_s} \qquad \text{(A1.4)}$$

where: $\mathrm{UFL_e}$ = estimated upper flammable limit.

NOTE A3.1—Equation A1.4 is not valid, and upper limits exceed 3.5

times stoichiometric for certain unsaturated compounds, organic oxides, ethers, amines, and other reactive materials. It is suggested (see Section 7) that, with the exception of amines, upper limits not be conducted on such materials in this apparatus.

A3.2.1 The upper flammable limit estimated with Eq A1.4 may be used with Eq 3 to estimate a starting sample size for tests.

A4. CORRELATION OF KNOWN UPPER AND LOWER FLAMMABLE LIMITS WITH STOICHIOMETRIC COMPOSITION

A4.1 Details on a correlation of known upper and lower flammable limits with stoichiometric composition are described by Hilado.[3] These details may permit more exact estimation of lower and upper flammable limits than is proposed in A2 and A3. However, to quote from the reference, "These estimates must be very cautiously applied because there are many chemicals with unusual flammability characteristics. Estimated values are not a substitute for experimental data."

APPENDIX

(Nonmandatory Information)

XI. CALCULATION OF FLAMMABLE LIMITS FROM TEST DATA

XI.1 Basis of Eq 3

$$\text{LFL} = \frac{\text{moles of combustible in test vessel}}{\text{total moles of gas in test vessel}} \times 100 \quad \text{(X1)}$$

$$= \frac{(L_v)(d)}{\text{MW}} \times \frac{1}{\dfrac{V}{22.4} \times \dfrac{P}{(P_o)} \times \dfrac{(T_o)}{T}} \times 100 \quad \text{(X2)}$$

where:

LFL = lower flammable limit, mol or volume %,
L_v = L = liquid sample volume, cm^3, from Eq 1,
d = sample density, g/cm^3,
MW = sample molecular weight,
V = test vessel volume, litres,
22.4 = standard molar volume, litres,
P = test pressure, mm Hg,
T = test temperature, K,
P_o = standard pressure = 1 atm (760 mm Hg),
T_o = standard temperature = 273 K,

$$\text{LFL} = \frac{(L_v)(d)(T)}{(\text{MW})(P)} \times k \quad \text{(X3)}$$

$$k = \frac{(22.4)(P_o)(100)}{(V)(T_o)} \quad \text{(X4)}$$

X1.2 Sample Calculation

X1.2.1 Tests conducted in a 5100-cm^3 vessel at 24°C and 750 mm Hg with a liquid sample having a density of 0.775 g/cm^3 and a molecular weight of 84.16 produced the first flame propagation at 270 µl and no flame propagation at 260 µl.

L_v = ½ $(L_1 + L_2)$ = ½(0.270 + 0.260) = 0.265 cm^3
d = 0.775 g/cm^3
MW = 84.16
V = 5.1 litres
P = 750 mm Hg
T = 24°C = 297 K

X1.2.2 Using Eq X4:

$$k = \frac{(22.4)(760)(100)}{(5.1)(273)} = 1222.7$$

This value will remain unchanged for all tests conducted in a system of 5.1-litre total volume.

X1.2.3 Using Eq 3 (see Section 10 on Eq X3):

$$\text{LFL} = \frac{(0.265)(0.775)(297)}{(84.16)(750)} \times 1222.7 = 1.18$$

where the lower flammable limit (LFL) = 1.18 volume % at 750 mm Hg pressure and 24°C.

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Document Name: ASTM E695: Standard Method of Measuring Relative Resistance of Wall, Floor and Roof Construction to Impact Loading

CFR Section(s): 24 CFR 200.946(a)(1)(viii)

Standards Body: American Society for Testing and Materials

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: E 695 – 79 (Reapproved 1997)$^{\epsilon 1}$

An American National Standard

Standard Method of Measuring Relative Resistance of Wall, Floor, and Roof Construction to Impact Loading[1]

This standard is issued under the fixed designation E 695; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ϵ^1 Note—Keywords were added editorially in March 1997.

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1. Scope

1.1 This method covers the measurement of the relative resistance of wall, floor, and roof construction to impact loading. The test is not applicable to doors.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1517 Definitions of Terms Relating to Leather[2]

E 73 Practice for Static Load Testing of Truss Assemblies[3]

E 575 Practice for Reporting Data from Structural Tests of Building Constructions, Elements, Connections, and Assemblies[3]

E 661 Test Method for Performance of Wood and Wood-Based Floor and Roof Sheathing Under Concentrated Static and Impact Loads[3]

2.2 *Other Standards:*

ANSI Z 299.2 Voluntary Industry Performance Standards for Pressure and Velocity of Shotshell Ammunition for the Use of Commercial Manufacturers[4]

Federal Specification V-T-291E (1) Thread, Linen[5]

3. Significance and Use

3.1 The procedures outlined will provide data that can be used to evaluate the relative performance of wall, floor, and roof constructions under conditions representative of those sustained in actual service when subjected to impact by a heavy blunt object. See Test Method E 661 for evaluation of floor and roof sheathing and Practice E 73 for evaluation of roof trusses.

3.2 The method is intended to be applied to relatively light construction, including but not limited to wood floor and roof systems, partitions framed with wood or steel studs, steel floor or roof decking systems, steel siding and wall panels, or thin concrete and masonry walls or slabs and similar assemblies.

4. Summary of Method

4.1 Specimens of wall, floor, and roof construction are subjected to the impact force of a standard impact instrument. Wall sections are tested in the vertical position. Floor and roof sections are tested only in the horizontal position. Because of the inherent differences in the method of applying load, measurements obtained from tests in a horizontal mode are not comparable to measurements obtained from tests in the vertical mode.

5. Apparatus for Floor and Roof Systems, Specimen Horizontal (see Fig. 1)

5.1 *Supports*, steel rollers, two, on a rigid base.

5.2 *Impact Instrument*, made with a lead shot-filled leather bag as specified in 5.2.1-5.2.6.

5.2.1 *Leather*—The leather used in construction of the bag should be harness leather (Note 1), oak tanned (Note 1) from packer hides (Note 1) or latigo leather (Note 1), alum and vegetable tanned, or both. Leather thickness shall be expressed in ounces (Note 1) (1 oz = 0.4 mm (1/64 in.)).

Note 1—See Definitions D 1517.

5.2.2 *Thread*—Thread used in fabrication of the bag shall be linen thread of four or more plys, meeting the requirements for Type B, Class 1 or 2, of Federal Specification V-T-291E (*1*).

5.2.3 *Fabrication*—The side of the bag shall be 710 mm (28 in.) high by 735 mm (29 in.) wide of 8-oz leather 3 mm (1/8 in.) thick. The vertical edges shall be sewed together flesh side out and the seam shall be reinforced with a piece of 8-oz leather overlapping 10 mm (3/8 in.) each side. The side shall then be turned hair side out and sewed to the bottom. The base (bottom disk) shall be 230 mm (9 in.) in diameter of 12-oz leather 5 mm (3/16 in.) thick. The seam attaching the wall to the base shall be

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[1] This method is under the jurisdiction of ASTM Committee E-6 on Performance of Buildings and is the direct responsibility of Subcommittee E06.11 on Horizontal and Vertical Structures/Structural Performance of Completed Structures.

Current edition approved Oct. 29, 1979. Published February 1980.

[2] *Annual Book of ASTM Standards*, Vol 15.04.

[3] *Annual Book of ASTM Standards*, Vol 04.11.

[4] Available from American National Standards Institute, 11 West 42nd St., 13th Floor, New York, NY 10036.

[5] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

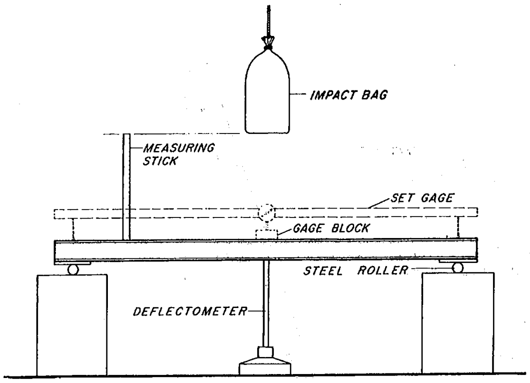

FIG. 1 Impact Load Test (Specimen Horizontal)

6 mm (¼ in.) from the edge of the base. Two rows of stitching shall be used for the vertical wall seam and the seam attaching the wall to the base.

5.2.4 *Hoisting Strap*—The strap to hoist the bag shall be made from 8-oz leather 3 mm (⅛ in.) thick by 15 mm (⅝ in.) wide by 610 mm (24 in.) long. The strap shall be passed through holes, diametrically opposite, in the side walls 40 mm (1½ in.) from the top of the wall. These holes shall be reinforced with pieces of 8-oz leather and 75 mm (3 in.) square. The leather strap shall be passed twice through a 50-mm (2-in.) diameter lifting ring and the ends fastened by sewing, riveting, or by use of a buckle. To avoid excessive stretching of the leather wall or failure of the vertical seam, a sleeve, made from 12-oz leather, of the same type as the base of the bag, shall be fitted to slip tightly around the lower portion of the bag. This sleeve should be 250 mm (9⅝ in.) high.

5.2.5 *Lead Shot*—The bag shall be loosely filled with No. 7½ (2.4 mm (0.095 in.) in diameter) chilled lead shot, in conformance with ANSI Z 299.2. Two layers of 75-mm (3-in.) thick foam rubber or similar padding shall be placed over the lead shot to prevent spillage during testing.

5.2.6 The total mass of the bag, including shot, shall be adjusted to the desired level with an accuracy of ±1 %. The mass of the bag may be adjusted to any specified mass, depending upon the information desired.

5.3 *Measuring Sticks*—A stick, laid off in 150-mm (6-in.) increments, or a series of sticks the lengths of which are multiples of 150 mm (6 in.), to measure the height of drop accurately. A graduated sliding pointer, a standard metal tape measure, or any similar device that can accurately measure the height of drop may be substituted.

5.4 *Deflectometer*, or other suitable deflectometer equipment, consisting of a metal tube having a base at the lower end and a clamp at the upper end which supports, by friction, a light metal rod. The rod shall be movable inside the tube and shall be graduated to 0.25-mm (0.01-in.) divisions.

5.5 *Set Gage*, consisting of a light, rigid frame having two legs at one end and one leg at the other end, with the distance between the legs equal to the span of the specimen. A dial micrometer graduated to 0.025-mm (0.001-in.) divisions shall be attached to the frame at midlength.

5.6 *Gage Blocks*, 300 by 300 mm (12 by 12 in.) in area, and constructed of metal or other hard surface material.

5.7 *Hold-Downs*—Clamps or other restraining devices at the specimen ends to minimize translation.

6. Apparatus for Wall Systems, Specimen Vertical (see Fig. 2)

6.1 *Steel Channels*, for support of the specimen at top and bottom.

6.2 *Rollers*, cylindrical rollers and two supporting rollers.

6.3 Impact bag, measuring sticks, deflectometer, set gage, and gage blocks conforming to the requirements specified in 5.2-5.7.

6.4 *Rigid Supporting Frame*, to which the supporting channels and deflection gage are attached.

7. Test Specimen

7.1 *Size*—The specimens shall be representative of the actual construction as to material, method of assembly, and workmanship.

7.2 *Length or Height*—The length or height of specimen for each element shall be chosen to conform approximately to the length or height of that element in actual size.

7.3 *Width*—The width of specimen shall be chosen, insofar as feasible, to include several of the principal load-carrying members to ensure that the behavior under load will simulate that anticipated under service conditions. The actual width of specimens shall be a whole number multiplied by the spacing of the principal load-carrying members, except for prefabricated panels for which the actual width shall be the width of panel used. If the structural properties of a particular construction are to be compared with another construction, there should not be a great difference in the actual widths of the specimens.

7.4 *Age*—Constructions such as concrete and masonry (brick, structural clay tile, concrete block) for which the structural properties depend upon the age of the specimen, shall be tested not less than 25 days nor more than 56 days after fabrication except in special instances such as the case of existing panels. This age requirement applies also to plastered and stuccoed constructions. Other assemblies affected by moisture shall be conditioned to constant weight or moisture content, or for at least 2 weeks at 20 ± 3°C (68 ± 6°F) and 65 ± 5 % relative humidity.

7.5 *Number*—Tests shall be made on a minimum of three like specimens. However, more tests may be necessary depending upon information and accuracy desired.

8. Procedure

8.1 For symmetrical walls apply the impact load to the outside face from at least one of the specimens, and to the inside face of the other two specimens. For asymmetrical walls, test both sides an equal number of times. This will require a minimum of four test specimens. Exception: only one side need be tested at the option of the client and laboratory depending upon information required. The report shall record which side or sides of the specimen that is tested. For floor and roof assemblies apply the impact loads only to the upper finish-floor face of the specimen.

NOTE 2—Any criteria for pass/fail or for stopping the test should be agreed upon between the sponsor and testing agency prior to testing. Some possible points include: the ability to sustain a specified static load after

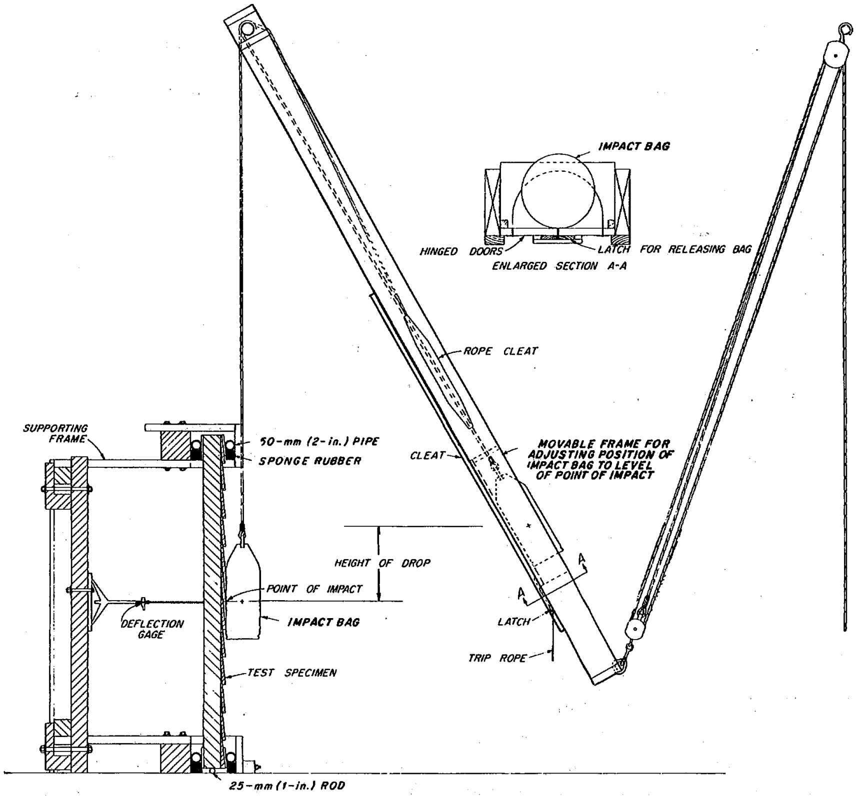

FIG. 2 Impact Load Test (Specimen Vertical)

the impact drop; limitation on the instantaneous deflection or residual set; penetration of panel; destruction of panel (to be defined); or simply a specified height of drop or number of drops per test from a specific height.

8.2 *Point of Impact*—If the construction has structural members, that is, studs or joists, test each specimen so that at least one such structural member will be struck by the impact bag. Test each specimen also so that the impact bag will strike the facing midway between two members.

NOTE 3—As written, this method may not necessarily define or test the most vulnerable part of a panel specimen. Additional tests may be required to locate such an area if this information is desired.

8.3 *Loading-Floor and Roof Assemblies-Specimen Horizontal*—Test the specimen as a simple beam on a span nominally 150 mm (6 in.) less than the specimen length. The two supports for the specimens shall prevent longitudinal restraint and shall provide bearing for the entire width of the specimen. Secure the ends of the panel by hold-downs to minimize specimen bounce. Take care to assure that the hold-downs do not affect deflection of the specimen. Apply an impact load to the upper face of the specimen by dropping the bag beginning with a height of 150 mm (6 in.) and increasing the height in 150-mm increments. Record set and instantaneous deflection measurements for each drop. For the first drop, measure the height of the bag from the upper face of the specimen at a point directly beneath the bag, and for subsequent drops, from a taut cord in contact with the upper face directly above the supports.

8.4 *Loading-Wall Assemblies Specimen Vertical*—Position the specimen on cylindrical rollers to prevent transverse restraint. The axes of the rollers shall be parallel to the faces of the specimens. The two supporting rollers shall be in contact with the vertical surface of the rigid frame and each roller shall rest horizontally on sponge rubber about 15 mm (½ in.) thick to prevent longitudinal restraint. Support the bag as a pendulum in the frame as shown in Fig. 2. Take care to assure that the hold-downs do not affect deflection of the specimen. Apply an impact load to the middle of the outer face of the specimens by releasing the bag beginning with a height of 150 mm (6 in.) and increasing the height in 150-mm increments. The maximum useful height of drop will occur when the pendulum or frame is perpendicular to the specimen. Measure the height of drop from the point of impact of the center of gravity of the bag as it strikes the specimen to this same point when the bag is in the raised position. Release the bag by smoothly and swiftly opening the hinged doors, causing it to swing as a true pendulum thus eliminating wobbling.

8.5 *Instantaneous Deflection*—Use the deflectometer (see 5.4) to measure the instantaneous deflection of the specimen. Prior to loading, hold the light metal rod in contact with the middle of the lower face of the specimen by the clamp. When the specimen deflects under the impact load, the rod is held in its lowest position by the friction clamp. Report readings to the nearest 0.25 mm (0.01 in.).

8.6 *Set*—To measure the set, place the set gage on the upper face of the specimen. Take readings by placing the set gage on the specimen with the legs at the supports and the spindle of the dial micrometer in contact with the middle of the specimen, and reading the micrometer. If the set exceeds the range of the micrometer, place gage blocks between the specimen and the spindle of the micrometer to the nearest 0.025 mm (0.001 in.).

9. Recordings

9.1 *Deflection and Set*—For each height of drop calculate the deflection between the reading of the deflectometer and the initial reading. Similarly, calculate the set as the difference between the reading of the set gage and the initial reading. Record the maximum height of drop.

10. Report

10.1 The report shall follow the format of Practice E 575. In addition, the report shall also include the following:

10.2 Description of test assembly, including:

10.2.1 Size of test specimen,

10.2.2 Details of structural design, including where applicable or required, the design stresses, design loads and safety factors of all structural members in test assembly,

10.2.3 Plan, evaluation, principal cross section, plus other sections as needed for clarity, and

10.2.4 Details of attachment of test panel in frame.

10.3 Summarize results.

10.4 It is essential that drawings required by 3.1.6 of Practice E 575 be provided.

11. Precision and Bias

11.1 Neither the within-laboratory nor the between-laboratory precision and bias of the impact load procedures recommended have been established, because any test data developed are usually of a proprietary nature and unavailable. Furthermore, the test method is expected to be used for prototype testing and not for routine quality control. This means few specimens of a kind would be tested, making any analysis unjustifiable.

12. Keywords

12.1 floor systems; impact loading; roof systems; wall systems

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Document Name: ASTM E711: Standard Test Method for Gross Calorific Value of Refuse-Derived Fuel by the Bomb Calorimeter

CFR Section(s): 40 CFR 63, Subpart DDDDD, Table 6

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: E 711 – 87 (Reapproved 1992)

Standard Test Method for Gross Calorific Value of Refuse-Derived Fuel by the Bomb Calorimeter[1]

This standard is issued under the fixed designation E 711; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the gross calorific value of a prepared analysis sample of solid forms of refuse-derived fuel (RDF) by the bomb calorimeter method.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific cautionary and precautionary statements see 6.10 and Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]
D 3177 Test Method for Total Sulfur in the Analysis Sample of Coal and Coke[3]
E 1 Specification for ASTM Thermometers[4]
E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals[5]
E 775 Test Methods for Total Sulfur in the Analysis Sample of Refuse-Derived Fuel[6]
E 790 Test Method for Residual Moisture in a Refuse-Derived Fuel Analysis Sample[6]
E 829 Practice for Preparing Refuse-Derived Fuels (RDF) Laboratory Samples for Analysis[6]

3. Terminology

3.1 *Definitions*:

3.1.1 *calorific value*—the heat of combustion of a unit quantity of a substance. It may be expressed in joules per gram (J/g), British thermal units per pound (Btu/lb), or calories per gram (cal/g) when required.

NOTE 1—The unit equivalents are as follows:
1 Btu (International Table) = 1055.06 absolute joules
1 Calorie (International Table) = 4.1868 absolute joules
1 Btu/lb = 2.326 J/g
1.8 Btu/lb = 1.0 cal/g

3.1.2 *gross calorific value*—the heat produced by combustion of a unit quantity of solid fuel, at constant volume, in an oxygen bomb calorimeter under specified conditions such that all water in the products remains in liquid form.

3.1.3 *net calorific value*—a lower value calculated from the gross calorific value. It is equivalent to the heat produced by combustion of a unit quantity of solid fuel at a constant pressure of one atmosphere, under the assumption that all water in the products remains in the form of vapor.

3.2 *Descriptions of Terms Specific to This Method*:

3.2.1 *calorimeter*—describes the bomb, the vessel with stirrer, and the water in which the bomb is immersed.

3.2.2 *energy equivalent*—the energy required to raise the temperature (Note 2) of the calorimeter system 1°C (or 1°F) per gram of sample. This is the number that is multiplied by the corrected temperature rise in degrees and divided by the sample weight in grams to give the gross calorific value after thermochemical corrections have been applied.

NOTE 2—Temperature change is measured in thermal units. Temperature changes may also be recorded in electromotive force, ohms, or other units when other types of temperature sensors are used. Consistent units must be used in both the standardization and actual calorific determination. Time is expressed in minutes. Weights are measured in grams.

3.2.3 *refuse-derived fuels*—solid forms of refuse-derived fuels from which appropriate analytical samples may be prepared are defined as follows in *ASTM STP 832*:[7]

RDF-1—Wastes used as a fuel in as-discarded form with only bulky wastes removed.
RDF-2—Wastes processed to coarse particle size with or without ferrous metal separation.
RDF-3—Combustible waste fraction processed to particle sizes, 95 % passing 2-in. square screening.
RDF-4—Combustible waste fraction processed into powder form, 95 % passing 10-mesh screening.
RDF-5—Combustible waste fraction densified (compressed) into the form of pellets, slugs, cubettes, or briquettes.

4. Summary of Test Method

4.1 Calorific value is determined in this method by burning a weighed analysis sample in an oxygen bomb calorimeter under controlled conditions. The calorific value is computed from temperature observations made before and after combustion, taking proper allowance for thermometer and thermochemical corrections. Either isothermal or adiabatic calorimeter jackets may be used.

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[1] This test method is under the jurisdiction of ASTM Committee D-34 on Waste Management and is the direct responsibility of Subcommittee D34.08 on Thermal Treatment.

Current edition approved Aug. 28, 1987. Published October 1987.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 05.05.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] *Annual Book of ASTM Standards*, Vol 15.05.

[6] *Annual Book of ASTM Standards*, Vol 11.04.

[7] *Thesaurus on Resource Recovery Terminology, ASTM STP 832*, ASTM, 1983, p. 72.

5. Significance and Use

5.1 The calorific value, or heat of combustion, is a measure of the energy available from a fuel. Knowledge of this value is essential in assessing the commercial worth of the fuel and to provide the basis of contract between producer and user.

6. Apparatus

6.1 *Test Room*—The apparatus should be operated in a room or area free of drafts that can be kept at a reasonably uniform temperature and humidity for the time required for the determination. The apparatus should be shielded from direct sunlight and radiation from other sources. Controlled room temperature and humidity are desirable.

6.2 *Oxygen Bomb*, constructed of materials that are not affected by the combustion process or products sufficiently to introduce measurable heat input or alteration of end products. If the bomb is lined with platinum or gold, all openings shall be sealed to prevent combustion products from reaching the base metal. The bomb shall be designed so that all liquid combustion products can be completely recovered by washing the inner surfaces. There shall be no gas leakage during a test. The bomb shall be capable of withstanding a hydrostatic pressure test to 21 MPa (3000 psig) at room temperature without stressing any part beyond its elastic limit.

6.3 *Calorimeter*, made of metal (preferably copper or brass) with a tarnish-resistant coating and with all outer surfaces highly polished. Its size shall be such that the bomb will be completely immersed in water when the calorimeter is assembled. It shall have a device for stirring the water thoroughly and at a uniform rate, but with minimum heat input. Continuous stirring for 10 min shall not raise the calorimeter temperature more than 0.01°C (0.02°F) starting with identical temperatures in the calorimeter, room, and jacket. The immersed portion of the stirrer shall be coupled to the outside through a material of low heat conductivity.

6.4 *Jacket*—The calorimeter shall be completely enclosed within a stirred water jacket and supported so that its sides, top, and bottom are approximately 10 mm from the jacket walls. The jacket may be arranged so as to remain at constant temperature or with provisions for rapidly adjusting the jacket temperature to equal that of the calorimeter for adiabatic operation. It shall be constructed so that any water evaporating from the jacket will not oondense on the calorimeter.

6.5 *Thermometers*—Temperatures in the calorimeter and jacket shall be measured with the following thermometers or combinations thereof:

6.5.1 *Mercury-in-Glass Thermometers*, conforming to the requirements for Thermometers 116°C or 117°C (56°F or 57°F) as prescribed in Specification E 1. Other thermometers of equal or better accuracy are satisfactory. These thermometers shall be tested for accuracy against a known standard (preferably by the National Bureau of Standards) at intervals no greater than 2.0°C (3.6°F) over the entire graduated scale. The maximum difference in correction between any two test points shall not be more than 0.02°C (0.04°F).

6.5.2 *Beckmann Differential Thermometer*, having a range of approximately 6°C in 0.01°C subdivisions reading upward and conforming to the requirements for Thermometer 115°C, as prescribed in Specification E 1. Each of these thermometers shall be tested for accuracy against a known standard at intervals no larger than 1°C over the entire graduated scale. The maximum difference between any two test points shall not be more than 0.02°C.

6.5.3 *Calorimetric-Type Platinum Resistance Thermometer, 25-*, tested for accuracy against a known standard.

6.5.4 *Other Thermometers*—A high precision electronic thermometer employing balanced thermistors or a quartz thermometer may be used, provided the temperature rise indication is accurate within ±0.003°C per 1°C rise.

6.6 *Thermometer Accessories*—A magnifier is required for reading mercury-in-glass thermometers to one tenth of the smallest scale division. This shall have a lens and holder designed so as to introduce no significant errors due to parallax. A Wheatstone bridge and galvanometer capable of measuring resistance to 0.0001 Ω are necessary for use with resistance thermometers.

6.7 *Sample Holder*—Samples shall be burned in an open crucible of platinum, quartz, or acceptable base-metal alloy. Base-metal alloy crucibles are acceptable if after a few preliminary firings the weight does not change significantly between tasks.

6.8 *Firing Wire* shall be 100 mm of No. 34 B & S nickel-chromium alloy wire or 100 mm of No. 34 B & S iron wire. Equivalent platinum or palladium wire may be used provided constant ignition energy is supplied, or measured, and appropriate corrections made.

6.9 *Firing Circuit*—A 6 to 16-V alternating or direct current is required for ignition purposes with an ammeter or pilot light in the circuit to indicate when current is flowing. A stepdown transformer connected to an alternating current lighting circuit or batteries may be used.

6.10 **CAUTION:** The ignition circuit switch shall be of momentary double-contact type, normally open, except when held closed by the operator. The switch should be depressed only long enough to fire the bomb.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[8] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water, Type III, conforming to Specification D 1193.

7.3 *Benzoic Acid, Standard (C_6H_5COOH)*—Use National Bureau of Standards SRM (Standard Reference Material) benzoic acid. The crystals shall be pelletized before use. Commercially prepared pellets may be used provided they are made from National Bureau of Standards benzoic acid.

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[8] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Analar Standards for Laboratory U.K. Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia."

The value of heat of combustion of benzoic acid, for use in the calibration calculations, shall be in accordance with the value listed in the National Bureau of Standards certificate issued with the standard.

7.4 *Methyl Orange, Methyl Red, or Methyl Purple Indicator* may be used to titrate the acid formed in the combustion. The indicator selected shall be used consistently in both calibrations and calorific determinations.

7.5 *Oxygen*, free of combustible matter. Oxygen manufactured from liquid air, guaranteed to be greater than 99.5 % pure, will meet this requirement. Oxygen made by the electrolytic process may contain a small amount of hydrogen rendering it unfit without purification.

7.6 *Sodium Carbonate, Standard Solution* (0.34 *N*)—One millilitre of this solution should be equivalent to 20.0 J in the nitric acid (HNO_3) titration. Dissolve 18.02 g of anhydrous sodium carbonate (Na_2CO_3) in water and dilute to 1 L. The Na_2CO_3 should be previously dried for 24 h at 105°C. The buret used for the HNO_3 titration shall be of such accuracy that estimations to 0.1 mL can be made. A more dilute standard solution may be used for higher sensitivity.

8. Precautions

8.1 Due to the origins of RDF in municipal waste, common sense dictates that some precautions should be observed when conducting tests on the samples. Recommended hygienic practices include use of gloves when handling RDF and washing hands before eating or smoking.

8.2 The following precautions are recommended for safe calorimeter operation:

8.2.1 The weight of solid fuel sample and the pressure of the oxygen admitted to the bomb must not exceed the bomb manufacturer's recommendations.

8.2.2 Bomb parts should be inspected carefully after each use. Threads on the main closure should be checked frequently for wear. The bomb should be returned to the manufacturer occasionally for inspection and possibly proof of firing.

8.2.3 The oxygen supply cylinder should be equipped with an approved type of safety device, such as a reducing valve, in addition to the needle valve and pressure gage used in regulating the oxygen feed to the bomb. Valves, gages, and gaskets must meet industry safety codes. Suitable reducing valves and adaptors for 2 to 3.5-MPa (300 to 500-psig) discharge pressure are obtainable from commercial sources of compressed gas equipment. The pressure gage shall be checked periodically for accuracy.

8.2.4 During ignition of a sample, the operator shall not permit any portion of his body to extend over the calorimeter.

9. Sampling[9]

9.1 RDF products are frequently nonhomogeneous. For this reason significant care should be exercised to obtain a representative laboratory sample for the RDF lot to be characterized.

9.2 The sampling method for this procedure should be based on agreement between the involved parties.

9.3 The laboratory sample must be air-dried and particle size reduced to pass a 0.5-mm screen as described in Practice E 829.

10. Standardization

10.1 Determine the energy equivalent of the calorimeter as the average of a series of ten individual runs, made over a period of not less than 3 days or more than 5 days. To be acceptable, the standard deviation of the series shall be 6.9 kJ/°C (6.5 Btu/°C) or less (see Appendix X1, Table X1). For this purpose, any individual run may be discarded only if there is evidence indicating incomplete combustion. If this limit is not met, repeat the entire series until a series is obtained with a standard deviation below the acceptable limit.

10.2 The weights of the pellets of benzoic acid in each series should be regulated to yield the same temperature rise as that obtained with the various samples tested in the individual laboratories. The usual range of weight is 0.9 to 1.3 g. Make each determination in accordance with the procedure described in Section 11, and compute the corrected temperature rise, *T*, as described in 12.1. Determine the corrections for HNO_3 and firing wire as described in 12.2 and substitute into the following equation:

$$E = [(H)(g) + e_1 + e_3 + e_4] \times t$$

where:

E = energy equivalent, J/°C,
H = heat of combustion of benzoic acid, as stated in the National Bureau of Standards certificate, J/g,
g = weight of benzoic acid, g,
t = corrected temperature rise, °C,
e_1 = titration correction, J,
e_3 = fuse wire correction, J, and
e_4 = correction for ignition energy if measured and corrected for, J.

10.3 Standardization tests should be repeated after changing any part of the calorimeter and occasionally as a check on both calorimeter and operating technique.

11. Procedure

11.1 *Weight of Sample*—Thoroughly mix the analysis sample of solid fuel in the sample bottle, taking care that the heavies and lights (fluff) are distributed in the sample (Note 3). Carefully weigh approximately 1 g of the sample directly into the crucible in which it is to be burned or into a tared weighing scoop from which the sample is transferred to the crucible. Weigh the sample to the nearest 0.1 mg. Some form of compaction may be necessary to ensure satisfactory ignition and complete combustion.

NOTE 3—In the event segregation of the heavies and lights cannot be avoided, attempt to remove sample from the bottle in such a way that a representative sample is transferred.

NOTE 4—Perform the residual moisture determination of the sample simultaneously using Test Method E 790.

11.2 *Water in Bomb*—Add 1.0 mL of water to the bomb by a pipet. Before adding this water, rinse the bomb, and drain the excess water, and leave undried.

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[9] ASTM Subcommittee E38.01 is currently in the process of developing procedures for sampling RDF-3 and the preparation of an analysis sample. The chairman of E38.01 should be contacted for details.

11.3 *Firing Wire*—Connect a measured length of firing wire to the ignition terminals with enough slack to allow the firing wire to maintain contact with the sample.

11.4 *Oxygen*—Charge the bomb with oxygen to a consistent pressure between 20 and 30 atm (2.03 and 3.04 MPa). This pressure must remain the same for each calibration and for each calorific determination. If, by accident, the oxygen introduced into the bomb should exceed the specified pressure, do not proceed with the combustion. Detach the filling connection and exhaust the bomb in the usual manner. Discard this sample.

11.5 *Calorimeter Water*—It is recommended that calorimeter water temperature be adjusted before weighing as follows:

11.5.1 *Isothermal Jacket Method*, 1.6 to 2.0°C (3.0 to 3.5°F) below jacket temperature (Note 4).

11.5.2 *Adiabatic Jacket Method*, 1.0 to 1.4°C (2.0 to 2.5°F) below room temperature.

NOTE 5—This initial adjustment will ensure a final temperature slightly above that of the jacket for calorimeters having an energy equivalent of approximately 10 200 J/K (2450 cal/°C). Some operators prefer a lower initial temperature so that the final temperature is slightly below that of the jacket. This procedure is acceptable, provided it is used in all tests, including standardization. Use the same amount (±0.5 g) of water in the calorimeter vessel for each test and for calibration. The amount of water (2000 g is usual) can be most satisfactorily determined by weighing the calorimeter vessel and water together on a balance. The water may be measured volumetrically if it is always measured at the same temperature. Tap water may be satisfactory for use in calorimeter bucket.

11.6 *Observations, Isothermal Jacket Method*—Assemble the calorimeter in the jacket and start the stirrer. Allow 5 min for attainment of equilibrium; then record the calorimeter temperatures (Note 6) at 1-min intervals for 5 min. Fire the charge at the start of the sixth minute and record the time and temperature, T^a. Add to this temperature 60 % of the expected temperature rise, and record the time at which the 60 % point is reached (Note 5). After the rapid-rise period (about 4 to 5 min), record temperatures at 1-min intervals on the minute until the difference between successive readings has been constant for 5 min.

NOTE 6—Use a magnifier and estimate all readings (except those during the rapid rise period) to the nearest 0.002°C (0.005°F) when using ASTM Bomb Calorimeter Thermometer 56C (56F). Estimate Beckmann thermometer readings to the nearest 0.001°C. Tap mercurial thermometers with a pencil just before reading to avoid errors caused by mercury sticking to the walls of the capillary.

NOTE 7—When the approximate expected rise is unknown, the time at which the temperature reaches 60 % of the total can be determined by recording temperatures at 45, 60, 75, 90, and 105 s after firing and interpolating.

11.7 *Observations, Adiabatic Jacket Method*—Assemble the calorimeter in the jacket and start the stirrer. Adjust the jacket temperature to be equal to or slightly lower than the calorimeter, and run for 5 min to obtain equilibrium. Adjust the jacket temperature to match the calorimeter with ±0.01°C (0.02°F) and hold for 3 min. Record the initial temperature (Note 6) and fire the charge. Adjust the jacket temperature to match that of the calorimeter during the period of rise, keeping the two temperatures as nearly equal as possible during the rapid rise, and adjusting to within ±0.01°C (0.02°F) when approaching the final equilibrium temperature. Take calorimeter readings at 1-min intervals until the same temperature is observed in three successive readings. Record this as the final temperature. Do not record time intervals since they are not critical in the adiabatic method.

11.8 *Analysis of Bomb Contents*—Remove the bomb and release the pressure at a uniform rate, in such a way that the operation will require not less than 1 min. Examine the bomb interior and discard the test if unburned sample or sooty deposits are found. Carefully wash the interior of the bomb including the capsule with distilled or deionized water containing the titration indicator until the washings are free of acid. Collect the washings in a beaker and titrate the washings with standard carbonate solution. Remove and measure or weigh the combined pieces of unburned firing wire, and subtract from the original length or weight to determine the wire consumed in firing. Determine the sulfur content of the sample by any of the procedures described in Test Methods E 775.

12. Calculation

12.1 *Temperature Rise in Isothermal Jacket Calorimeter*—Using data obtained as prescribed in 11.6, compute the temperature rise, T, in an isothermal jacket calorimeter as follows:

$$T = T_c - T_a - r_1(b - a) - r_2(c - b)$$

where:

T = corrected temperature rise,
a = time of firing,
b = time (to nearest 0.1 min) when the temperature rise reaches 60 % of total,
c = time at beginning of period in which the rate of temperature change with time has become constant (after combustion),
T_a = temperature at time of firing, corrected for thermometer error (Note 7),
T_c = temperature at time c, corrected for thermometer error (Note 7),
r_1 = rate (temperature units per minute) at which temperature was rising during 5-min period before firing, and
r_2 = rate (temperature units per minute) at which temperature was rising during the 5-min period after time c. If the temperature is falling, r_2 is negative and the quantity $r_2(c - b)$ is positive.

12.2 *Temperature Rise in Adiabiatic Jacket Calorimeter*—Using data obtained as prescribed in 11.7 compute the corrected temperature rise, T, as follows:

$$T = T_f - T_a$$

where:

T = corrected temperature rise, °C or °F,
T_a = initial temperature when charge was fired, corrected for thermometer error (Note 8), and
T_f = final temperature corrected for thermometer error.

NOTE 8—With all mercury-in-glass thermometers, it is necessary to make the following corrections if the total heat value is altered by 12 J/g or more. This represents a change of 0.001°C (0.002°F) in a calorimeter using approximately 2000 g of water. The corrections include the calibration correction as stated on the calibration certificate, the setting correction for Beckman thermometers according to the directions furnished by the calibration authority, and the correction for emergent stem. Directions for these corrections are given in Appendix X2.

12.3 *Thermochemical Corrections* (Appendix X3)—Compute the following for each test:

e_1 = correction for the heat of formation of HNO_3, J. Each millilitre of standard alkali is equivalent to 20.0 J.
e_2 = correction for heat of formation of H_2SO_4, J
= 55.2 × percent of sulfur in sample × weight of sample, g.
e_3 = correction for heat of combustion of firing wire, J (Note 10)
= 9.6 J/cm or 5980 J/g for No. 34 B & S gage Chromel C
= 11.3 J/cm or 7330 J/g for No. 34 B & S iron wire.
e_4 = correction for ignition energy of platinum or palladium if measured and corrected for.

NOTE 9—There is no correction for platinum or palladium wire, provided the ignition energy is constant.

12.4 *Calorific Value*:

12.4.1 Calculate the gross calorific value (gross heat of combustion) as follows:

$$H_s = [(T)(E) - e_1 - e_2 - e_3 - e_4]/g$$

where:

H_s = gross calorific value, J/g,
T = corrected temperature rise as calculated in 12.1 or 12.2, °C or °F, consistent with the water equivalent value,
E = energy equivalent (see Section 10),
e_1, e_2, e_3, e_4 = corrections as prescribed in 12.3, and
g = weight of sample, g.

12.4.2 Calculate the net calorific value (net heat of combustion) as follows:

$$H_i = H_s - 23.96\,(H \times 9)$$

where:

H_i = net calorific value (net heat of combustion), J/g,
H_s = gross calorific value (gross heat of combustion), J/g, and
H = total hydrogen, %.

13. Precision and Bias[10]

13.1 *Precision*—The standard deviations of individual determinations, in Btu/lb, are:

Average	Within-laboratory	Between-laboratories
HHV-1:		
6400	27.1	135.5
5200	48.8	239.6
HHV-2:		
7900	32.3	118.0
7400	38.1	227.8
HHV-3:		
9700	111.3	290.4
9500	99.2	249.2
9300	40.3	67.6

13.2 These precision estimates are based on an interlaboratory study conducted in accordance with Practice E 180.

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[10] Supporting data are available on loan from ASTM Headquarters. Request RR:E38-1000.

APPENDIXES

(Nonmandatory Information)

X1. CALCULATION OF STANDARD DEVIATIONS FOR CALORIMETER STANDARDIZATION

X1.1 The example given in Table X1.1 illustrates the method of calculating standard deviations for calorimeter standardizations.

TABLE X1.1 **Standard Deviations for Calorimeter Standardization**[A]

Standardization Number	Column A Water Equivalent, (Btu/lb) × (g/°C)	Column B Code to 4400 (Column A-4400)	Column C (Column B)^2
1	4412	12	144
2	4407	7	49
3	4415	15	225
4	4408	8	64
5	4404	4	16
6	4406	6	36
7	4409	9	81
8	4410	10	100
9	4412	12	144
10	4409	9	81
Sum		92	940

Average = $\bar{x}^2$ = x/10 = (92/10) + 4400 = 4409
Variance = s^2 = Column C − (Column B)^2/n/n − 1 = 940 − (92)^2/10/9 = 10.4
Standard deviation, s = Variance = 10.4 = 3.22

[A] In this example the values of water equivalent are typical for a calorimeter calibrated such that the water equivalent multiplied by the temperature rise in °C/g of sample will give the calorific value of the sample in Btu/lb.

X2. THERMOMETER CORRECTIONS

X2.1 It is necessary to make the following corrections in the event they result in an equivalent change of 0.001°C or more.

X2.1.1 *Calibration Correction* shall be made in accordance with the calibration certificate furnished by the calibration authority.

X2.1.2 *Setting Correction* is necessary for the Beckmann thermometer. It shall be made in accordance with the directions furnished by the calibration authority.

X2.1.3 *Differential Emergent Stem Correction*—The calculation depends upon the way the thermometer was calibrated and how it is used. The following two conditions are possible:

(*a*) *Thermometers Calibrated in Total Immersion and Used in Partial Immersion*—This emergent stem correction is made as follows:

$$\text{Correction} = K(t_c - t_a)\,(t_c + t_a - L - T)$$

where:

K = 0.00016 for thermometers calibrated in °C,
0.00009 for thermometers calibrated in °F,
L = scale reading to which the thermometer was immersed,
T = mean temperature of emergent stem,
t_a = initial temperature reading, and
t_c = final temperature reading.

NOTE X2.1: *Example*—Suppose the point L, to which the thermometer was immersed was 16°C; its initial reading, t_a, was 24.127°C, its final reading, t_c, was 27.876°C; the mean temperature of the emergent stem, T, was 26°C,

then:

Differential stem correction

$$= +0.00016\ (28 - 24)\ (28 + 24 - 16 - 26)$$
$$= + 0.006°\text{C}$$

(*b*) *Thermometers Calibrated and Used in Partial Immersion but at a Different Temperature than the Calibration Temperature*—This emergent stem correction is made as follows:

$$\text{Correction} = K\,(t_c - t_a)\,(t_1 - t^{\circ})$$

where:

K = 0.00016 for thermometers calibrated in °C,
0.00009 for thermometers calibrated in °F,
t_a = initial temperature reading,
t_c = final temperature reading,
t_1 = observed stem temperature, and
t° = stem temperature at which the thermometer was calibrated.

NOTE X2.2: *Example*—Suppose the initial reading, t_a, was 80°F, the final reading, t_c, was 86°F, and that the observed stem temperature, t_1, was 82°F, and the calibration temperature, t°, was 72°F; then:

Differential stem correction

$$= 0.00009\ (86 - 90)(82 - 72)$$
$$= 0.005°\text{F}$$

X3. THERMOCHEMICAL CORRECTIONS

X3.1 *Heat of Formation of Nitric Acid*—A correction (e^1, in 12.3) of 20 J is applied for each 1 mL of standard Na_2CO_3 solution used in the acid titration. The standard solution (0.34 *N*) contains 18.02 g of Na_2CO_3/L. This correction is based on assumption that all the acid titrated is HNO_3 formed by the following reaction: ½ N_2 (g + 5⁄4 O_2 (g) + ½ H_2O (l) = HNO_3 (in 500 mol H_2O), and (*2*) the energy of formation of 1 mol of HNO_3 is approximately 500 mol of water under bomb conditions is 14.1 kcal/mol.[6] When H_2SO_4 is also present part of the correction for H_2SO_4 is contained in the e_1 correction and the remainder in the e_2 correction.

X3.2 *Heat of Formation of Sulfuric Acid*—By definition the gross calorific value is obtained when the product of the combustion of sulfur in the sample is SO_2 (g). However, in actual bomb combustion processes, the sulfur is found as H_2SO_4 in the bomb washings. A correction (e_2 in 12.4.1) of 55.2 J is applied for each percent of sulfur in the 1-g sample, that is converted to H_2SO_4. This correction is based upon the energy of formation of H_2SO_4 in solutions such as will be present in the bomb at the end of a combustion. This energy is taken as −70.5 kcal/mol.[11] A correction, of 2 × 14.1 kcal/mol of sulfur was applied in the e_1 correction, so the additional correction necessary is 70.5 − (2 × 14.1) = 42.3 kcal/mol or 5520 J of sulfur in the sample (55.2 J × weight of sample in grams × % sulfur in sample).

X3.2.1 The value of 5520 J/g of sulfur is based on a coal containing about 5 % sulfur and about 5 % hydrogen. The assumption is also made that the H_2SO_4 is dissolved entirely in water condensed during combustion of the sample.[12] If a 1-g sample of such a fuel is burned, the resulting H_2SO_4 condensed with water formed on the walls of the bomb will have a ratio of about 15 mol of water to 1 mol of H_2SO_4. For this concentration the energy of the reaction.

$$SO_2\ (g) + {}^1\!/\!{}_2O_2\ (g) + H_2O\ (l) = H_2SO_4\ (\text{in } 15 \text{ mol } H_2O)$$

under the conditions of the bomb process is −70.5 kcal/mol.

X3.2.2 Basing the calculation upon a sample of comparatively large sulfur content reduces the overall possible errors, because for smaller percentages of sulfur the correction is smaller.

X3.3 *Fuse Wire*—Calculate the heat in SI units contributed by burning the fuse wire in accordance with the directions furnished by the supplier of the wire. For example, the heat of combustion of No. 34 B & S gage Chromel C wire

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[11] Calculated from data in National Bureau of Standards Circular 500.

[12] Mott, R. A., and Parker, C., "Studies in Bomb Calorimetry IX-Formation of Sulfuric Acid," *Fuel*, Vol 37, 1958, p. 371.

is equivalent to 9.6 J/cm or 5980 J/g and that of No. 34 B & S gage iron wire is equivalent to 11.3 J/cm or 7330 J/g. There is no correction for platinum or palladium wire provided the ignition energy is constant.

X4. REPORTING RESULTS IN OTHER UNITS

X4.1 *Reporting Results in British Thermal Units (Btu) per Pound*—The gross calorific value can be expressed in British thermal units by using the thermochemical correction factors in Table X4.1 and the water equivalent expressed in (Btu/lb) × (g/°C).

TABLE X4.1 Thermochemical Correction Factors (Units in BTU)

Correction	Multiplication Factor	Multiply by
e_1 (HNO_3)	10.0	mL of 0.394 N Na_2CO_3 solution
e_2 (H_2SO_4)	23.7	% of sulfur in sample times weight of sample in grams
e_3 (fuse wire)	4.1 or	cm of No. 34 B & S gage Chromel C wire
	2570	weight (g) of Chromel C wire
e_3 (fuse wire)	4.9 or	cm of No. 34 B & S gage iron wire
	3150	weight (g) of iron wire

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An American National Standard

Standard Test Method for
Accelerated Weathering of Sealed Insulating Glass Units[1]

This standard is issued under the fixed designation E 773; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers procedures for testing the performance of preassembled permanently sealed insulating glass units against accelerated weathering.

1.2 This test method is applicable only to sealed insulating glass units, with one or two airspaces, fabricated for vision glass areas for use in buildings, such as sliding doors, windows, wall systems, and picture windows.

1.3 The unit construction used in this test method contains dimensions that are an essential component of the test. Deviations of glass or airspace sizes, or both, will affect the test results.

1.4 This test method is not applicable to sealed insulating glass units that are constructed from exterior vision materials other than glass.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* A specific hazard statement is given in Note 2.

2. Referenced Documents

2.1 *ASTM Standards:*

C 1036 Specification for Flat Glass[2]

E 546 Test Method for Frost Point of Sealed Insulating Glass Units[3]

E 774 Specification for the Classification of the Durability of Sealed Insulating Glass Units[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *chemical dew point*—the temperature (Note 1) at which organic vapor or other chemical begins to condense on the interior glass surface of a sealed insulating glass unit.

NOTE 1—The temperature as indicated by the temperature indicator of frost point apparatus when constructed and used in accordance with test procedures described in Test Method E 546.

3.1.2 *frost point*—the temperature (Note 1) below freezing point of water (0°C (32°F)) at which visible frost begins to deposit on the interior glass surface of a sealed insulating glass unit.

4. Significance and Use

4.1 This test method is intended to provide a means for evaluating the durability of the sealing system of sealed insulating glass units. A round-robin test has been conducted, and additional data are being collected. The suitability of these test methods will be further evaluated as the analysis of test results is completed.

5. Apparatus

5.1 *For High Humidity Testing*:

5.1.1 *High-Humidity Test Chamber*—A chamber of convenient dimensions capable of maintaining 60 ± 3°C (140 ± 5°F) and 95 ± 5 % relative humidity.

5.2 *For Accelerated Weather Cycle Test*:

5.2.1 *Accelerated Weather Cycle Test Apparatus*[4]—The accelerated weather cycle test apparatus shall be essentially that shown in Figs. 1-3 to provide the cycle conditions and time frame indicated in Section 8.

5.2.2 *Ultraviolet Light Source*:

NOTE 2—**Warning:** Ultraviolet light sources used in this test method are harmful, especially to the eyes. Appropriate protective measures must be observed.

5.2.2.1 The source shall consist of two fluorescent black light lamps, Type F72T12BL/HO[5,6] (Note 3), for each test specimen located as shown in Fig. 3.

5.2.2.2 Each lamp must be replaced when its ultraviolet light intensity falls below 10 kW/m^2 when measured with a long-wave ultraviolet meter[5] in direct contact with the lamp.

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[1] This test method is under the jurisdiction of ASTM Committee E-6 on Performance of Buildings and is the direct responsibility of Subcommittee E06.22 on Durability Performance of Building Constructions.

Current edition approved June 10, 1997. Published August 1997. Originally published as E 773 – 81. Last previous edition E 773 – 83 (1995).

[2] *Annual Book of ASTM Standards,* Vol 15.02.

[3] *Annual Book of ASTM Standards,* Vol 04.11.

[4] The apparatus is a modification of the device developed by the Division of Building Research of the National Research Council of Canada. The modifications include two black light lamps for each test specimen. Construction details are available from SIGMA (Sealed Insulating Glass Manufacturer's Association), 111 E. Wacker Drive, Chicago, IL 60601.

[5] The sole source of supply of the apparatus known to the committee at this time is General Electric Co., Nela Park, Cleveland, OH 44112. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

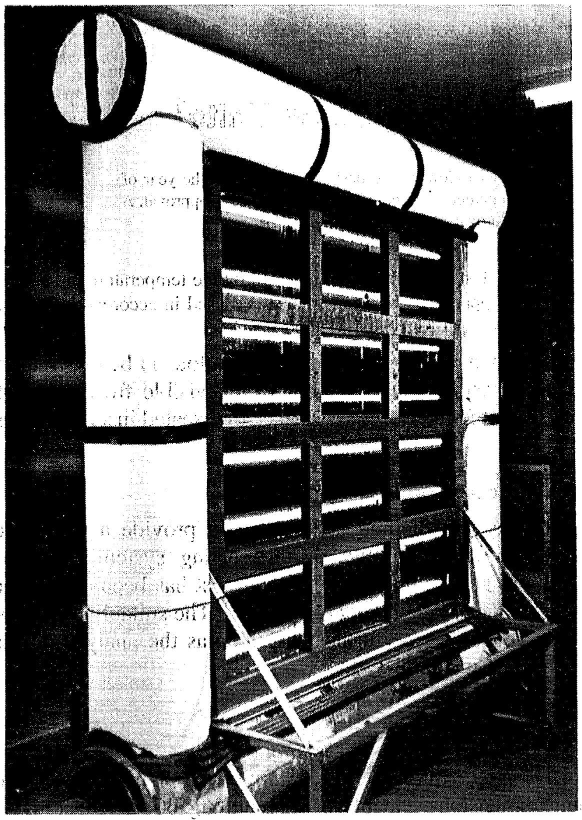

FIG. 1 Typical Accelerated Weather Cycle Test Apparatus

NOTE 3—Rated average life at 3 h per start: 12 000 h. Rated average life at 12 h per start: 18 000 h. Useful length: 1625 mm (64 in.). Wattage: 85 W. Relative black light energy output is 190 % that of F40BL lamp.

6. Test Specimens

6.1 Each test specimen shall measure 356 ± 6 mm by 508 ± 6 mm (14 ± ¼ in. by 20 ± ¼ in.) and shall be composed of two or three panes of clear, tinted or coated annealed, heat-strengthened, tempered or laminated glass.

6.1.1 The thickness of the glass pane shall be nominally 5 mm (3⁄16 in.) or 6 mm (¼ in.).

6.1.2 The airspace or spaces shall be from nominal 6 mm (¼ in.) through 13 mm (½ in.).

6.1.3 Triple pane units where the intermediate airspace divider is a plastic film are acceptable.

6.2 The thickness tolerance of the glass shall conform to Specification C 1036.

6.3 Each specimen shall be permanently and legibly marked with the designation of the manufacturer and the date of fabrication (month and year).

6.4 At least ten specimens of identical component materials and construction shall be submitted.

6.5 During all stages of exposure and storage, the units shall be stated in a vertical position with equal support to all panes and no compression loading.

6.6 Selection of units for testing shall be made at random except for units damaged in transit. Damaged units shall not be tested.

7. Preparation of Test Specimen

7.1 Uncleanable stain or scum may remain on the exterior glass surface of the specimen after the accelerated weathering test. Measures should be taken to have a clear view of the interior glass surface for detection of frost. For example, place a mask of plastic tape[7] 50 by 50 mm (2 by 2 in.) or larger, on the central region of both exterior glass surfaces. Remove the mask for frost point measurement.

7.2 The sealed insulating glass units shall be sealed a minimum of 4 weeks to allow for stabilization before the high humidity exposure in accordance with 8.1 begins. The manufacturer has the option to waive this requirement.

8. Procedure

8.1 *High Humidity Test*:

8.1.1 Expose six specimens in the high-humidity test chamber at 60 ± 3°C (140 ± 5°F) and 95 ± 5 % relative humidity. Arrange the specimens so that each specimen has at least 6-mm (¼-in.) clearance all around the four sides. Protect the high humidity chamber from overheating with a protective device.

8.1.1.1 Equip the high humidity chamber with a continuous temperature recording device placed in an area that gives the average temperature in the chamber.

8.1.2 When the specified time period has been attained, remove the test specimens and determine the frost point in accordance with Test Method E 546. Observe for chemical dew point.

8.2 *Accelerated Weather Cycle Test*:

8.2.1 Place the test specimens that were tested in accordance with 8.1 in the accelerated weather cycle test apparatus. Mount the specimens so that one exterior surface of the specimen is exposed to weathering cycles and the other to room temperature (Note 4). Install all specimens as shown in Fig. 2, taking care that no stress is induced in the test specimens by the method of fastening. The test specimens shall be oriented in the accelerated weather cycle exposure with the number one surface facing the weather changes as it does in normal field exposure. Ensure that the bearing edge and the weathering or exposed side remain the same throughout all testing.

NOTE 4—A temperature in the range from 15 to 30°C (60 to 85°F).

8.2.2 *Cycling*—Each cycle shall be 6 h ± 5 min and composed of the following test condition (see Fig. 4):

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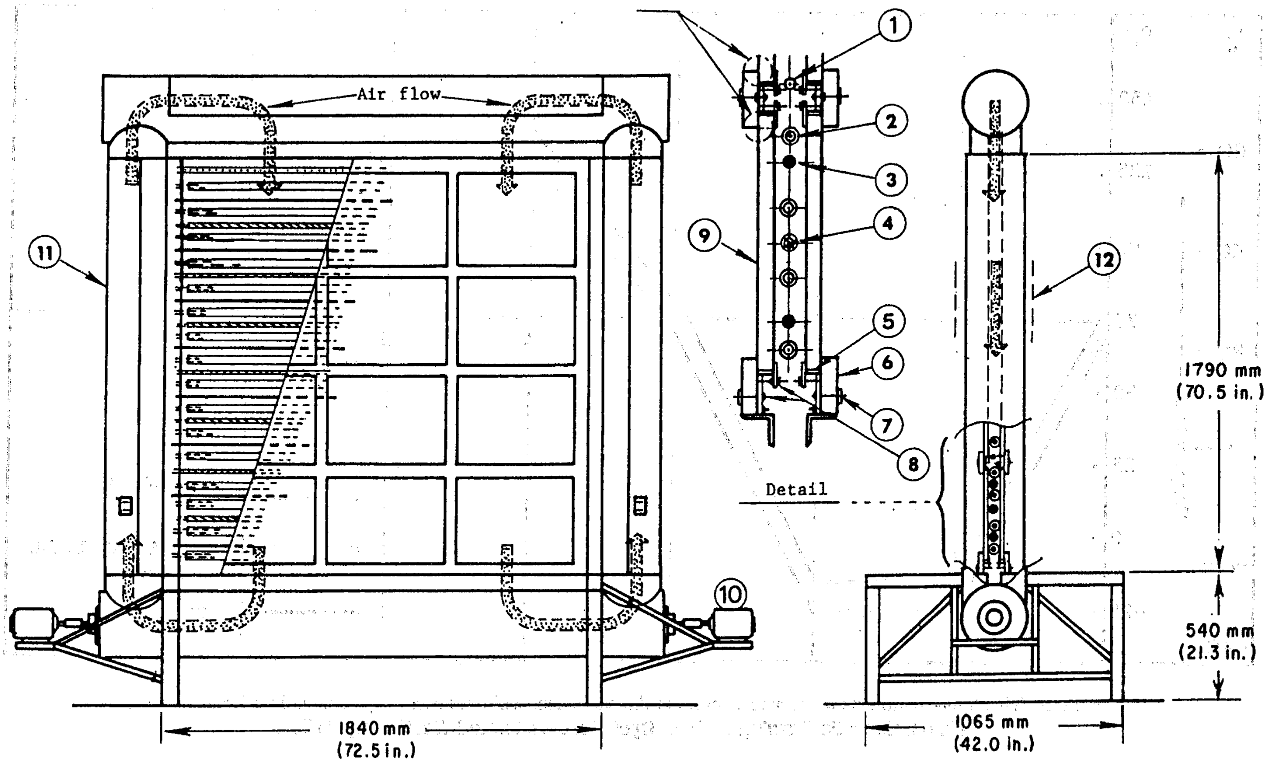
[6] The sole source of supply of the apparatus known to the committee at this time is the Black-Ray UV Meter with J221 sensor cell available from Ultra-Violet Products, Inc., 5100 Walnut Grove Ave., San Gabriel, CA 91778. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[7] The sole source of supply of the apparatus known to the committee at this time is Scotch Plastic Tape # 471 available from 3M Co., 3M Center, Commercial Office Supply Div., Bldg. 230-3 South-17, St. Paul, MN 55101. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.



Description: 1. Water spray pipe, 2. Cooling coil, 3. Fluorescent black light lamp, F72T12 BL/HO, 4. Heating coil, 5. Rubber pad, 6. Polystyrene insulation, 7. Rubber washer, 8. Clamping device, 9. Test specimen, 10. Fan motor, 11. Air duct, 12. Insulation.

FIG. 2 Schematic Drawing of Typical Accelerated Weather Cycle Test Apparatus

8.2.2.1 During the first 1 h ± 5 min, decrease the temperature from room temperature to − 30 ± 3°C (−20 ± 5°F).

8.2.2.2 Maintain temperature at − 30± 3°C (−20 ± 5°F) for 1 h ± 5 min.

8.2.2.3 Turn on heat and allow temperature to rise from − 30± 3°C (−20 ± 5°F) to room temperature over a period of 1 h ± 5 min.

8.2.2.4 Over a time period of 1 h ± 5 min, start water spray and ultraviolet lamps and control the temperature rise from room temperature to 57 ± 3°C (135 ± 5°F). Turn off water spray after 30 min to allow temperature to continue to rise to 57 ± 3°C.

8.2.2.5 Maintain temperature at 57 ± 3°C (135± 5°F) and continue ultraviolet exposure for a period of 1 h ± 5 min. Humidity remains very high during this hour; consequently, additional water spray is not necessary.

8.2.2.6 Over a period of 1 h ± 5 min, decrease temperature from 57 ± 3°C (135 ± 5°F) to room temperature, and continue ultraviolet exposure. At the end of this period, turn off ultraviolet exposure.

8.2.2.7 Protect the accelerated weathering chamber from overheating and from overcooling with protective devices.

8.2.2.8 Equip the accelerated weather cycle chamber with a continuous temperature recording device placed in an area that gives the average temperature inside the chamber.

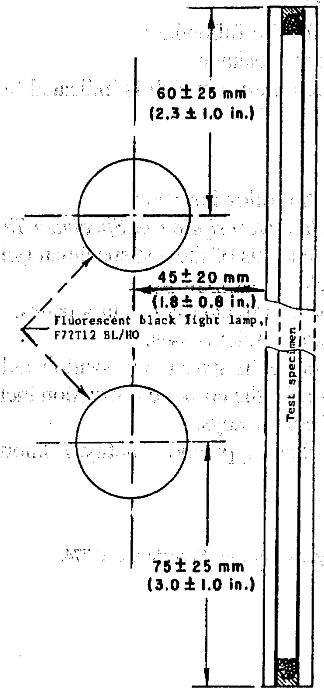
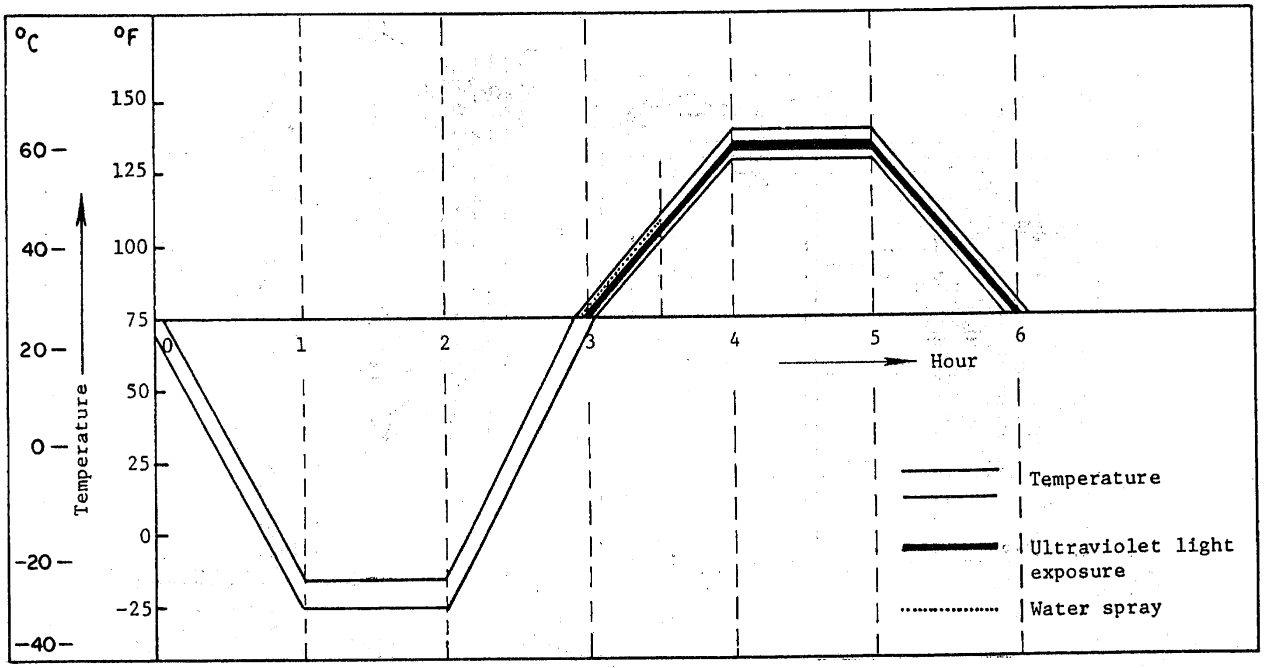


FIG. 3 Location of Fluorescent Black Light Lamp Relative to the Test Specimen



NOTE 1—This figure represents a linear response, but the equipment will not necessarily respond in a linear manner.

FIG. 4 Schematic Drawing of Each Cycle for Accelerated Weather Cycle Test

8.2.3 When the number of cycles prescribed[8] has been attained, remove the specimens and determine the frost point in accordance with Test Method E 546.

9. Observations

9.1 Observe for the following:

9.1.1 Specimen breakage.

9.1.2 Specimen failure, which is indicated by visible water in the cavity.

10. Report

10.1 Report the following data:

10.1.1 *Complete Description of Specimen Tested*:

10.1.1.1 Dimensions of the test specimen (width by height) and overall thickness.

10.1.1.2 Type and thickness of glass panes.

10.1.1.3 Airspace thicknesses.

10.1.1.4 Describe the spacer composition and configuration.

10.1.1.5 Describe the corner construction including the type and number of corner keys.

10.1.1.6 Dessicant type and quantity, if known, and sealant type.

10.1.1.7 Dimensions of sealant.

10.1.1.8 Manufacturer and manufactured date (month, if known, and year).

10.1.2 *Duration of Test*:

10.1.2.1 Duration of high humidity test described in 8.1 (number of days or weeks).

10.1.2.2 Duration of accelerated weather cycle test described in 8.2 (number of cycles, days or weeks).

10.1.3 Chemical dew point, if observed.

10.1.4 Specimen breakage, if observed.

10.1.5 Massive specimen failure, if observed.

11. Precision and Bias

11.1 *Precision*—The precision of the procedures in Test Method E 773 for measuring accelerated weatherability is being determined.

11.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedures in Test Method E 773 for accelerated weatherability, bias has not been determined.

12. Keywords

12.1 insulating glass units; sealed insulating glass units

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[8] For insulating glass units, see Specification E 774.

CERTIFICATE

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OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: E 774 – 97

An American National Standard

Standard Specification for the Classification of the Durability of Sealed Insulating Glass Units[1]

This standard is issued under the fixed designation E 774; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification is applicable to those sealed insulating glass units, with one or two airspaces.

1.2 The classification of test specimens is based on frost or chemical dew point after the specified test duration(s).

1.3 Qualification under this specification is intended to provide a basis for the classification of the durability of sealed insulating glass units. SIGMA field correlation studies started in 1980 show that units that have achieved a Level A classification have less than 1 % field failure rate in ten years provided the glazing system weeps water away from contact with the unit edge seal. Class C and CB tested units have significantly higher field failure rates in ten years. SIGMA continues to gather correlation data.[2]

1.4 This specification is not applicable to units that are constructed from vision materials other than glass.

1.5 This specification does not cover other physical requirements such as appearance, thermophysical properties, heat and light transmission, and glass displacement.

Note 1—Sealed insulating glass units classified according to this specification are not necessarily suitable for structurally glazed applications. Factors such as sealant longevity to long term direct ultraviolet light exposure and sealant tensile strength must be reviewed for these applications.

2. Referenced Documents

2.1 *ASTM Standards:*

E 546 Test Method for Frost Point of Sealed Insulating Glass Units[3]

E 773 Test Method for Accelerated Weathering of Sealed Insulating Glass Units[3]

E 1887 Test Method for Fog Determination[3]

3. Terminology

3.1 Definition:

3.1.1 *sealed insulating glass unit*—a preassembled unit, comprising sealed panes of glass separated by dehydrated space(s), intended for vision areas of buildings. The unit is normally used for windows, window walls, picture windows, sliding doors, patio doors, or other types of windows or doors.

4. Classification

4.1 Sealed insulating glass units tested in accordance with this specification shall be classified into one of three classes based on response to the tests and qualification requirements as shown in Table 1.

5. Performance Requirements

5.1 *Class C*—There shall be no frost or chemical dew point of each specimen when measured at a temperature of −34°C (−30°F) in accordance with Test Method E 546.

5.2 *Class B*—There shall be no frost or chemical dew point when measured at a temperature of −29°C (−20°F) in accordance with Test Method E 546.

5.3 *Class A*—There shall be no frost or chemical dew point when measured at a temperature of −29°C (−20°F) in accordance with Test Method E 546.

5.4 *Fog*—No fog shall be visible after testing in accordance with Test Method E 1887.

6. Test Specimens

6.1 Specimen design and construction for accelerated weathering tests shall be established by Test Method E 773.

7. Test Methods

7.1 Classify the sealed insulating glass units by following Test Method E 773 in accordance with Table 1 of this specification.

7.1.1 *Accelerated Weathering Test*:

7.1.1.1 Test six randomly selected test specimens for durations as shown in Table 1, Class C in accordance with Test Method E 773.

7.1.1.2 If the test specimens qualify for Class C as described in Section 5 of this specification, they may be tested further for durations as shown in Table 1, Class B. Place the specimens in the test racks so that the bearing edge and the weathering or exposed side remain the same as tested in previous class.

7.1.1.3 If the test specimens qualify for Class B as described in Section 5 of this specification, they may be tested further for durations as shown in Table 1, Class A.

7.1.1.4 Breakage of only two specimens is permitted

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[1] This specification is under the jurisdiction of ASTM Committee E-6 on Performance of Buildings and is the direct responsibility of Subcommittee E06.22 on Durability Performance of Building Constructions.

Current edition approved June 10, 1997. Published August 1997. Originally published as E 774 – 81. Last previous edition E 774 – 92.

[2] Available from SIGMA, 111 East Wacker Drive, Suite 600, Chicago, IL 60601.

[3] *Annual Book of ASTM Standards*, Vol 04.11.

TABLE 1 Classification and Physical Requirements for Seal Durability Test Method E 773

Classification of Specimen	Record Initial Frost Point and Chemical Dew Point	Accelerated Weathering Test			
			Duration of Test		
		Number of Specimens	High Humidity Test,^{A} (days)	Accelerated Weather Cycle Test,^{B} cycles	Final Qualification of Frost Point and Chemical Dew Point
Class C		6 each	14	140	lower than – 34°C (–30°F)
Class B		6 each	14	56	lower than – 29°C (–20°F)
Class A		6 each	14	56	lower than – 29°C (–20°F)

Fogging Test		
Number of Specimens	Duration of Test, days	Qualification
2 each	7	no visible fog

[A] See Procedure section of Test Method E 773.

[B] See Procedure section of Test Method E 773.

throughout the test. Replace any broken specimen with one from the same lot, and test from the beginning.

NOTE 2—Breakage due to laboratory handling, that is dropped units, is not considered to be test breakage. Laboratory broken units shall be replaced and retested from the beginning.

7.1.1.5 If third specimen breakage occurs during the test for any reason, do not qualify this set of test specimens for that tested class.

7.1.1.6 If any specimen has filled with water or shows visible condensation in the air space, do not qualify this set of test specimens for that tested class except as permitted in 7.1.1.4 (that is, breakage).

7.1.2 *Fogging Test*:

7.1.2.1 Select two specimens as shown in Table 1.

7.1.2.2 Test the two test specimens for durations as shown in Table 1, Class C in accordance with Test Method E 1887.

7.2 The test durations as shown in Table 1 are net testing periods. If the test is interrupted for any reason, the remaining portions shall be completed to qualify for the testing class.

NOTE 3—It is suggested that extra units be submitted. Twelve units have been shown to be adequate in many cases.

8. Report

8.1 Report the following information:

8.1.1 Detailed description of test specimen, test durations, and all reportable test data of Test Methods E 773 and E 1887.

8.1.2 Classes for which the test specimens qualified.

9. Acceptance or Rejection

9.1 When all test specimens have met the requirements as described in Section 5 for any particular class, this set of test specimens shall be accepted for that tested class.

9.2 If any specimen fails to meet the requirements as described in Section 5 for any particular class, this set of specimens shall be rejected for that tested class.

10. Keywords

10.1 insulating glass units; sealed insulating glass units

APPENDIX

(Nonmandatory Information)

X1. TEST DATA GATHERING

FIG. X1.1 Test Report for Specification E 774

Date ____________

Manufacturer ______________________ Ref. No. __________ Testing Lab. ______________________ Ref. No. __________

Address ______________________ Address ______________________

Attention ______________________ Tel. __________ Attention ______________________ Tel. __________

Description of Test Specimen:

Size (width by height) ______________________ Type and amount of desiccator ______________________

Thickness of glass ______________________ Type of sealant(s) ______________________

Thickness of air space ______________________ Other features (band, barrier coat, etc.) ______________________

Type of spacer ______________________ Manufactured date (month and year) ______________________

Test Results

Specimen No.	Class C					Class B			Class A		
	Initial Frost Point	Fogging Test, days	High-Humidity Test,[A] days	Accelerated Weather Cycle Test,[A] cycles	Remark	High-Humidity Test,[A] days	Accelerated Weather Cycle Test,[A] cycles	Remark	High-Humidity Test,[A] days	Accelerated Weather Cycle Test,[A] cycles	Remark
		7	14	140		14	56		14	56	
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											

[A] Actual frost/chemical dew point, or pass/fail.

CERTIFICATE

By Authority Of
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By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

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CFR Section(s): 40 CFR 63, Subpart DDDDD, Table 6

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: E 776 – 87 (Reapproved 1992)

Standard Test Method for Forms of Chlorine in Refuse-Derived Fuel[1]

This standard is issued under the fixed designation E 776; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the forms of chlorine in refuse-derived fuel-three (RDF): total chlorine, water-soluble chloride, and water-insoluble chlorine.

1.2 This test method may be applicable to any waste material from which a laboratory analysis sample can be prepared.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements see Section 6 and 11.2.1.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

E 144 Practice for Safe Use of Oxygen Combustion Bombs[3]

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals[4]

E 287 Specification for Burets[3]

E 711 Test Method for Gross Calorific Value of Refuse-Derived Fuel by the Bomb Calorimeter[5]

E 829 Practice for Preparing Refuse-Derived Fuel (RDF) Laboratory Samples for Analysis[5]

3. Descriptions of Terms Specific to This Standard

3.1 *refuse-derived fuels*—solid forms of refuse-derived fuels from which appropriate analytical samples may be prepared are defined as follows in *ASTM STP 832*:[6]

RDF-1—Wastes used as a fuel in as-discarded form with only bulky wastes removed.

RDF-2—Wastes processed to coarse particle size with or without ferrous metal separation.

RDF-3—Combustible waste fraction processed to particle sizes, 95 % passing 2-in. square screening.

RDF-4—Combustible waste fraction processed into powder form, 95 % passing 10-mesh screening.

RDF-5—Combustible waste fraction densified (compressed) into the form of pellets, slugs, cubettes, or briquettes.

3.2 *total chlorine*—all chlorine as determined in the refuse-derived fuel.

3.3 *water-insoluble chlorine*—water-insoluble chlorides and chlorine in the refuse-derived fuel.

3.4 *water-soluble chloride*—those chlorides which are water-solubilized by water extraction as determined in the refuse-derived fuel.

4. Summary of Test Method

4.1 The forms of chloride and chlorine are determined. The various procedures in the method convert the forms of chlorine into a water-soluble chloride form that can be quantitated by titration.

4.1.1 *Total Chlorine*—The sample is combusted in an oxygen atmosphere. The chlorine is converted to chloride and absorbed in an alkaline solution.

4.1.2 *Water-Soluble Chlorides*—A portion of the analysis sample is successively extracted with hot chloride-free water.

4.1.3 *Water-Insoluble Chlorine*—Water-insoluble chlorine is calculated from the results of the total chlorine and the water-soluble chloride determination where:

water-insoluble chlorine = total chlorine − water-soluble chlorides

4.2 The chlorides contained in the alkaline solution (4.1.1) and the extraction solution (4.1.2) are determined by potentiometric (see Section 13) or modified Volhard titration (see Section 14).

5. Significance and Use

5.1 The standard is available to producers and users of RDF for determining the content and forms of chlorine present in the fuel.

6. Precautions

6.1 Due to the origins of RDF in municipal waste, common sense dictates that some precautions should be observed when conducting tests on the samples. Recommended hygienic practices include use of gloves when handling RDF; wearing dust masks (NIOSH-approved type), especially while milling RDF samples; conducting tests under negative pressure hood when possible; and washing hands before eating or smoking.

7. Interferences

7.1 *Potentiometric Titration Method A:*

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[1] This test method is under the jurisdiction of ASTM Committee D-34 on Waste Management and is the direct responsibility of Subcommittee D34.08 on Thermal Treatment.

Current edition approved Aug. 28, 1987. Published October 1987. Originally published as E 776 – 81. Last previous edition E 776 – 81.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] *Annual Book of ASTM Standards*, Vol 15.05.

[5] *Annual Book of ASTM Standards*, Vol 11.04.

[6] *Thesaurus on Resource Recovery Terminology, ASTM STP 832*, ASTM, 1983, p. 72.

TABLE 1 Typical Data Sheet and Calculation of End Point from a Potentiometric Titration of Chloride

Volume of 0.025 *N* $AgNO_3$ solution, mL	Millivolt Reading	Volume of 0.025 *N* $AgNO_3$ solution, mL	Millivolt Reading	Volume of 0.025*N* $AgNO_3$ solution, mL	Millivolt Reading
0.0	−1.38	18.4	−0.65	20.2	+0.26
1.0	−1.29	18.6	−0.63	20.3	+0.38
2.0	−1.26	18.8	−0.59	20.4	+0.47
4.0	−1.21	19.0	−0.55	20.5	+0.55
6.0	−1.18	19.1	−0.53	20.6	+0.60
8.0	−1.14	19.2	−0.51	20.7	+0.65
10.0	−1.10	19.3	−0.48	20.8	+0.69
12.0	−1.04	19.4	−0.46	20.9	+0.72
14.0	−0.97	19.5	−0.42	21.0	+0.75
15.0	−0.95	19.6	−0.38	21.5	+0.90
16.0	−0.86	19.7	−0.33	22.0	+0.98
17.0	−0.79	19.8	−0.27	22.5	+1.04
17.5	−0.75	19.9	−0.19	23.0	+1.09
18.0	−0.70	20.0	−0.06	24.0	+1.17
18.2	−0.67	20.1	+0.08	25.0	+1.24

The greatest change of voltage per unit increment addition of $AgNO_3$ solution occurs between 19.8 and 20.5 mL. Determine the end point and titer as follows:

mL	mV	1st Derivative	2nd Derivative
19.8	−0.27		
19.9	−0.19	+0.08	+0.05
20.0	−0.06	+0.13	+0.01
20.1	+0.08	+0.14	+0.04
20.2	+0.26	+0.18	−0.06
20.3	+0.38	+0.12	−0.03
20.4	+0.47	+0.09	−0.01
20.5	+0.55	+0.08	

Interpolation:

$$20.1\ \text{mL} + \left(\frac{0.04}{0.10} \times 0.1\right) = 20.14\ \text{mL}$$

7.1.1 Iodide and bromide are also titrated as chloride. Ferricyanide causes high results and must be removed. Chromate and dichromate interfere and should be reduced to the chromic state or be removed. Ferric iron interferes if present in an amount substantially higher than the amount of chloride. Chromic ions, ferrous ions, and phosphates do not interfere.

7.1.2 Grossly contaminated sample solutions usually require pretreatment. Where contamination is minor, some contaminants can be destroyed simply by the addition of nitric acid.

7.2 *Volhard Titration Method B:*

7.2.1 Compounds that have a strong oxidizing action interfere by reacting with thiocyanate. These compounds should be reduced beforehand by treatment with ferrous sulfate or a similar reducing agent.

7.2.2 Salts of mercury and palladium interfere by reacting with thiocyanate. They may be removed by precipitation with hydrogen sulfide before the addition of silver nitrate. The excess of sulfide is easily removed by gently boiling the acid solution for a few minutes. Sulfite can be eliminated in the same way.

7.2.3 Cyanide is also precipitated by silver nitrate. It is usually determined separately by the Liebig-Deniges method and a correction is applied to the results of the Volhard titration.[7]

7.2.4 The Volhard method, as with the potentiometric method, directly applied to a mixture of halides can determine only total halide content excluding fluoride. Preliminary treatment is necessary for the determination of chloride alone in a mixture.[8]

8. Apparatus

8.1 *Balance*, having a sensitivity of 0.1 mg.

8.2 *Apparatus for Bomb Combustion of the Sample.*

8.2.1 *Oxygen Bomb*, similar to that used in the determination of the calorific value of refuse-derived fuels as described in Test Method E 711.

8.2.2 *Capsule*, for holding the sample, approximately 25 mm in diameter at the top, approximately 12 mm deep, and conforming to Test Method E 711.

8.2.3 *Firing Wire*, as specified in Test Method E 711.

8.2.4 *Firing Circuit*, as specified in Test Method E 711.

8.2.5 *Metal Vessel*, cylindrical, such that the bomb will be fully immersed when approximately 2 L of water are added.

8.3 *Magnetic Stirrer and Stirring Bars.*

8.4 *Apparatus for Potentiometric Titration:*

8.4.1 *Potentiometric Titration Assembly*, using a silver indicator electrode and a calomel reference electrode containing a saturated sodium nitrate solution as a bridge.

NOTE 1—All glassware and graduated apparatus should be Class A or equivalent as described in Specification E 287.

9. Reagents

9.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the

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[7] Scott's *Standard Method of Chemical Analysis*, edited by M. H. Furman, D. Van Nostrand Co., Inc., New York, NY.

[8] Koltoff, I. M., and Stenger, V. A., *Volumetric Analysis II*, Interscience Publishers, Inc., New York, NY.

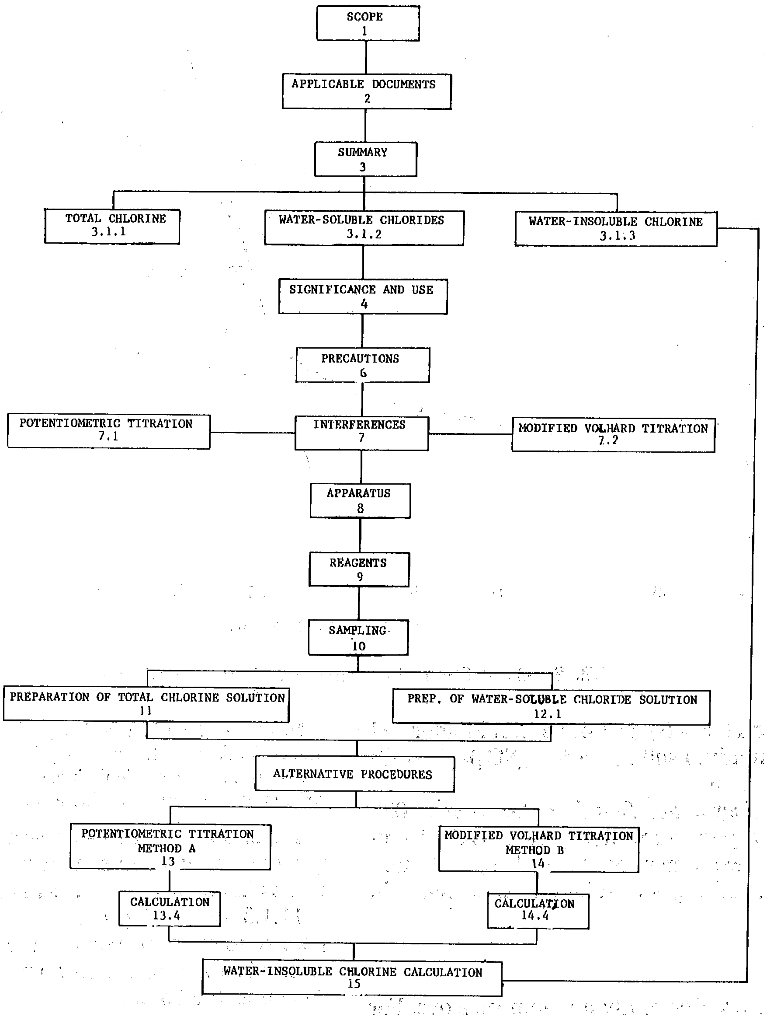

FIG. 1 Forms of Chlorine in Refused-Derived Fuel

Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[9] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

9.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean at least Type III reagent water conforming to Specification D 1193.

9.3 *Potassium Hydroxide Solution* (0.2 *N*)—Dissolve 13.2 g of potassium hydroxide (KOH) in water and dilute to 1 L with water.

9.4 *Oxygen*—The oxygen used for combustion shall be free of combustible matter. Oxygen manufactured from liquid air, guaranteed to be greater than 99.5 % pure, will meet this requirement.

9.5 *Sodium Chloride* (NaCl)—Primary standard quality (purity of 100 ± 0.02 %).

9.6 *Sodium Chloride, Primary Standard Solution* (0.025 *N*)—Crush 10 to 20 g of primary standard sodium chloride (NaCl) to 100-mesh fineness and dry in a glass container at 120°C for 2 h. Stopper and keep desiccated. Dissolve 5.844 g ± 0.1 mg of dried primary standard NaCl in water and dilute to 1 L. Dilute 25.00 mL of this solution to 100.0 mL.

9.7 *Methanol.*

9.8 *Nitric Acid* (1+1)—Mix equal volumes of concentrated nitric acid (HNO_3, sp, gr, 1.42) and water.

9.9 *Silver Nitrate, Standard Solution* (0.025 *N*)—Dissolve 4.247 g of silver nitrate ($AgNO_3$) in water and dilute to 1 L. Store in an amber glass bottle. Standardize against 0.025 *N* sodium chloride solution as directed in 13.1.1 and 14.1.1.

9.10 *Potassium Chromate - Potassium Dichromate Indicator* (K_2CrO_4 - $K_2Cr_2O_7$)—Dissolve 4.2 g of K_2CrO_4 and 0.7 g of $K_2Cr_2O_7$ in 100 mL of water.

9.11 *Nitrobenzene.*

9.12 *Ferric Ammonium Sulfate Indicator Solution*—Add

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[9] Reagent Chemicals, American Chemical Society Specifications, Am. Chemical Soc., Washington, DC, For suggestions on the testing of reagents not listed by the American Chemical Society, see "Analar Standards for Laboratory U.K. Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia."

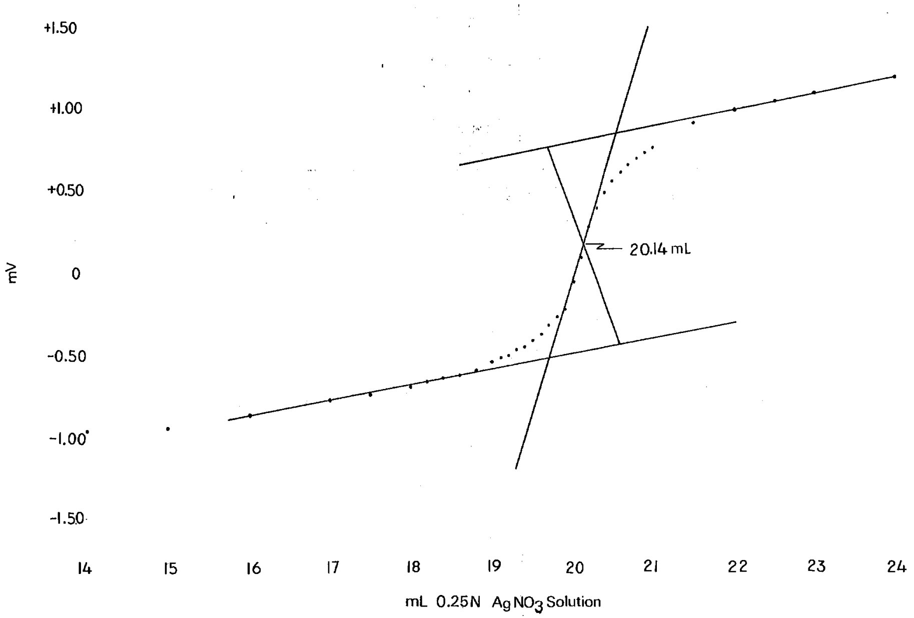

FIG. 2 Graph From a Potentiometric Titration of Chloride

sufficient concentrated HNO_3 (sp gr 1.42) to a cold saturated solution of ferric ammonium sulfate $[FeNH_4(SO_4)_2 \cdot 12H_2O]$ to remove the brown color.

9.13 *Potassium Thiocyanate, Standard Solution* (0.025 *N*)—Dissolve 3 g of potassium thiocyanate (KCNS) in freshly distilled or boiled water, dilute to 1 L, and standardize against 0.025 *N* $AgNO_3$ solution as directed in 14.1.2.

10. Sampling

10.1 RDF products are frequently nonhomogeneous. For this reason significant care should be exercised to obtain a representative laboratory sample from the RDF lot to be characterized.

10.2 The sampling method for this procedure should be based on agreement between the involved parties.

10.3 The laboratory sample must be air-dried and the particle size reduced to pass a 0.5-mm screen as described in Practice E 829. This procedure must be performed carefully to preserve the sample's representative characteristics (other than particle size) while preparing the analysis sample to be used in the procedures.

PREPARATION OF CHLORINE SOLUTIONS FOR ANALYSIS

11. Total Chlorine (Oxygen Bomb Method)

11.1 *Preparation of Sample and Bomb* (see Note 3):

11.1.1 *Sample Weight*—Weigh to the nearest 0.1 mg about 1 g of thoroughly mixed air-dried analysis RDF sample into the bomb capsule. A pellet may be made from the air-dried analysis RDF sample, accurately weighed, and placed into the bomb capsule. Place the capsule containing the sample into the capsule holder.

NOTE 2—There is a tendency for chlorine to adhere to the bomb walls, especially if the bomb is pitted or has been used previously to determine high levels of chlorine. Unless the bomb is thoroughly cleaned before use, the blanks may have values in excess of reality.

11.1.2 *Firing Wire*—Connect a length of firing wire to the ignition terminals in such a manner that the loop of firing wire is in contact with the sample.

11.1.3 *Bomb Preparation*—Add 20 to 25 mL of 0.02 *N* potassium hydroxide solution to the bomb and wet the entire internal surface of the bomb with this solution (see Note 3). Assemble the bomb.

NOTE 3—Sodium hydroxide solution at appropriate concentration may be used.

11.2 *Addition of Oxygen*—Admit oxygen to the bomb slowly to avoid blowing the sample from the capsule until a pressure of 25 atm is reached.

11.2.1 **Caution**—The following precautions are recommended for safe calorimeter operation. Additional precautions are given in Practice E 144.

11.2.1.1 The weight of RDF sample and the pressure of the oxygen admitted to the bomb must not exceed the bomb manufacturer's recommendations.

11.2.1.2 Bomb parts should be inspected carefully after each use. Threads on the main closure should be checked frequently for wear. Cracked or significantly worn parts should be replaced. The bomb should be returned to the manufacturer occasionally for inspection and possibly proof firing.

11.2.1.3 The oxygen supply cylinder should be equipped with an approved type of safety device, such as a reducing valve, in addition to the needle valve and pressure gage used in regulating the oxygen feed to the bomb. Valves, gages, and gaskets must meet industry safety codes. Suitable reducing valves and adaptors for 300 to 500 psi (2070 to 3450 kPa) discharge pressures are obtainable from commercial sources of compressed gas equipment. The pressure gage shall be checked periodically for accuracy.

11.2.1.4 During ignition of a sample, the operator must not permit any portion of his body to extend over the calorimeter.

11.3 *Preparation of the Chlorine Solution*—Immerse the bomb in a cold-water bath, connect it to the firing circuit, and close the circuit to ignite the sample. Allow the bomb to stand in the water bath for not less than 10 min after firing. Remove the bomb from the water bath, invert the bomb and shake for about 10 min (Note 4). Release the pressure at a slow, uniform rate so that the pressure is reduced to atmospheric in not less than 1 min. Open the bomb and examine the inside for traces of unburned material or sooty deposits. If any are found, discard the determination and thoroughly wash all parts of the bomb interior before using it again. If no unburned material or sooty deposits are present, rinse the interior of the bomb, the sample capsule, and the interior surface of the bomb cover with a fine jet of hot water and collect the contents and washings in a beaker or flask.

NOTE 4—Inverting and shaking a heavy, wet, and possibly slippery bomb may present a hazard. Precautions should be taken when performing this function.

12. Extraction of Water-Soluble Chlorides

12.1 *Preparation of the Chloride Solution*—Weigh to the nearest 0.1 mg about 5 g of thoroughly mixed, air-dried RDF analysis sample. Transfer to a 500-mL beaker and add 100 mL of hot water. Heat to close to boiling for ½ h with occasional stirring to ensure thorough wetting of the sample. Decant the supernatant liquid through a fast, qualitative filter paper collecting the filtrate in a beaker. Repeat the extraction two more times, each time using 100 mL of water and pooling the filtrates. After the third extraction, wash the sample thoroughly using 100 mL of hot water, adding the wash water to the pooled filtrates.

12.2 *Preparation of the Chloride Solution* (Alternative Method)—Weigh to the nearest 0.1 mg about 5 g of thoroughly mixed air-dried analysis sample (Note 5). Transfer to a 500-mL beaker and add 100 mL of hot water. Heat to close-to-boiling for ½ h with occasional stirring to ensure thorough wetting of the sample. Remove the beaker from the hot plate and allow the sample to settle. Decant the supernatant to a 100-mL centrifuge tube and centrifuge for ½ h at approximately 1200 rpm. Decant the supernatant through a fast qualitative filter paper. Repeat the extraction and centrifuging two more times, each time using 60 mL of water and pooling the filtrates. After the third extraction, wash the centrifuge tube and filter paper thoroughly using 60 mL of hot water, adding the wash water to the pooled filtrates (Notes 6 and 7).

NOTE 5—The weight of the sample used can be adjusted when higher or lower concentrations of water-soluble chlorides are anticipated.

NOTE 6—When high concentrations of chlorides are anticipated, dilute the cooled extraction solution to volume in a suitably sized volumetric flask and continue the analysis with a suitably sized aliquot.

NOTE 7—If the chloride solution is highly colored or turbid the potentiometric method (Section 13) should be used. If the Volhard method is to be used (Section 14), the color and turbidity must first be removed by aluminum hydroxide [Al(OH)$_3$] coagulation or by some other suitable method of turbidity or color removal without interfering with the chloride content of the solution.[10]

ALTERNATIVE PROCEDURES

13. Potentiometric Titration (Method A)

13.1 *Standardization of Reagents*—Prepare a standard solution of silver nitrate by adding 10.0 mL of standard 0.025 *N* NaCl solution to a 250-mL beaker. Add 40 to 50 mL of water and 50 mL of methanol (Note 9). Add 2 mL of HNO_3 (1 + 1) and titrate potentiometrically with the $AgNO_3$ solution as directed in 13.3. Calculate the normality of the $AgNO_3$ solution as follows:

$$\text{Normality of } AgNO_3 = \text{mL of NaCl solution used} \times \text{normality of NaCl solution/mL of } AgNO_3 \text{ solution required}$$

NOTE 8—Methanol increases the sensitivity of the inflection point. However, it may be omitted if it is found to interfere with or damage certain electrode systems.

13.2 *Blank Determination*—Prepare a solution for blank determination in the same manner as the preparation of chlorine solutions for the respective type of chlorine desired as described in Sections 11 or 12 using all the reagents in the same quantities and eliminating the RDF sample. Add to that blank solution 0.025 N NaCl solution as used in the standardization of $AgNO_3$ solution (13.1.1). Proceed as directed in 13.3. The difference in the volume of $AgNO_3$ solution required in this titration and that volume required for the standardization titration (13.1.1) is the true titer blank.

$$A - B = \text{true titer blank}$$

where:

A = millilitres of $AgNO_3$ required for the blank determination and

B = millilitres of $AgNO_3$ required for the standardization determination (13.1).

13.3 *Procedure:*

13.3.1 Concentrate the chlorides in the prepared solution (11.3, 12.1, or 12.2, respectively) for the type of chlorine desired by evaporation to about 50 mL. Add 50 mL of methanol and neutralize with HNO_3 (1+1) using phenolphthalein as an indicator. Add 2 mL of HNO_3 (1+1) in excess. Immerse the electrodes into the sample solution. While gently stirring on a magnetic stirrer, titrate by adding 0.10-mL increments of standard $AgNO_3$ solution and record the millivolt reading after each addition (Notes 9 and 10). Titrate well beyond the end point.

NOTE 9—Larger increments of $AgNO_3$ solution may be added before and after the end point. However, as the end point is reached, the 0.10-mL increment will give the most accurate end point.

NOTE 10—The end point is reached when the change of voltage is greatest upon the addition of the given increment of standard $AgNO_3$.

13.3.2 Plot the millivolt readings versus the millilitres of standard $AgNO_3$ solution added. Determine the end point (point of inflection) graphically from the titration curve or mathematically (second derivative) from the tabulated data. (See Table 1 and Fig. 1 for examples.)

13.4 *Calculations*—Calculate the weight percent of chloride as follows:

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[10] APHA, 14th ed., Method 408-A, Section 3a.

$$\text{Chloride, weight \%} = [C - (A - B)] \times N \times 0.03545 \times 100/W$$

where:

A = millilitres of $AgNO_3$ solution required for the titration of the blank (13.2),
B = millilitres of $AgNO_3$ solution required for the titration of the NaCl standard (13.1.1),
C = millilitres of $AgNO_3$ solution required for the titration of the sample (13.3.2),
N = normality of the $AgNO_3$ solution (13.1),
W = grams of the analysis sample used, and
0.03545 = milliequivalent weight of chloride.

14. Volhard Titration (Method B)

14.1 *Standardization of Reagents:*

14.1.1 *Silver Nitrate, Standard Solution*—Add 20.0 mL of standard 0.025 *N* NaCl solution to a 250-mL Erlenmeyer flask. Add 1 mL of $K_2CrO_4 - K_2Cr_2O_7$ indicator. Dilute to about 100 mL with water. Titrate with $AgNO_3$ solution to a faint red-brown end point. Calculate the normality of the $AgNO_3$ solution as follows:

$$\text{Normality of } AgNO_3 = \text{mL of NaCl solution used} \times \text{normality of NaCl solution/mL of } AgNO_3 \text{ solution required}$$

14.1.2 *Potassium Thiocyanate, Standard Solution*—Add 20.0 mL of 0.025 *N* $AgNO_3$ solution to a 250-mL glass-stoppered Erlenmeyer flask. Add 10 mL of HNO_3 (1+1) and make up to approximately 100 mL with water. Add 5 to 10 mL of nitrobenzene, stopper, and shake for 1 min. Add 8 to 10 drops of $FeNH_4(SO_4)_2$ indicator solution and titrate with KCNS solution. The end point is reached when the solution becomes faintly orange-pink in color. Calculate the normality of the KCNS solution as follows:

$$\text{Normality of KCNS} = \text{mL of } AgNO_3 \text{ solution used} \times \text{normality of } AgNO_3 \text{ solution/mL of KCNS solution required}$$

14.2 *Blank Determination*—Prepare a solution for the blank determination in the same manner as the preparation of chlorine solutions for the respective type of chlorine desired as described in Sections 11 and 12, using all the reagents in the same quantities and eliminating the RDF sample. Proceed as described in 14.3.

14.3 *Procedure*—Transfer quantitatively the chloride solution (11.3, 12.1, or 12.2, respectively) for the type of chlorine desired into a 250-mL glass-stoppered Erlenmeyer flask. Add 10 mL of HNO_3 (1+1), stirring gently during the acid addition. Add 20.0 mL of standard $AgNO_3$ solution. Stopper, mix, and let stand in the dark for 15 min. Cool to room temperature. Add 5 to 10 mL of nitrobenzene and shake for 1 min. Add 8 to 10 drops of $FeNH_4(SO_4)_2$ solution and titrate with standard KCNS solution against a white background. The end point of the determination is reached when the solution becomes faintly orange-pink in color.

14.4 *Calculations*—Calculate the weight percent of chloride as follows:

$$\text{Chloride (chlorine), weight \%} = \frac{(B - A) \times N \times 0.03545 \times 100}{W}$$

where:

A = millilitres of KCNS solution required for the titration of the sample (14.3),
B = millilitres of KCNS solution required for the titration of the blank (14.2),
N = normality of the KCNS solution (14.1.2),
W = grams of the analysis sample used, and
0.03545 = milliequivalent weight of chloride.

15. Water-Insoluble Chlorine

15.1 Calculate the percent of water-insoluble chlorine as follows: percent of total chlorine minus percent of water-insoluble chloride.

16. Precision and Bias[11]

16.1 *Precision:*

16.1.1 The standard deviations of individual determinations, in percent absolute, are as follows:

Typical Average Value,%	Within-Laboratory, %	Between Laboratories, %
Total Chlorine:		
0.49	0.03	0.11
Water-Soluble Chlorides:		
0.37	0.02	0.05

16.1.2 These precision estimates are based on an interlaboratory study conducted in accordance with Practice E 180.

16.2 *Bias*—The bias of this test has not been determined because of the lack of a recognized standard reference material.

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[11] Supporting data are available on loan from ASTM Headquarters. Request RR: E38-1000.