EXHIBIT 150
PART 11

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 D86: Standard Test Method for Distillation of Petroleum Products at Atmospheric Pressure

CFR Section(s): 40 CFR 1065.710

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: D 86 – 07

An American National Standard

Standard Test Method for Distillation of Petroleum Products at Atmospheric Pressure[1]

This standard is issued under the fixed designation D 86; 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.

1. Scope*

1.1 This test method covers the atmospheric distillation of petroleum products using a laboratory batch distillation unit to determine quantitatively the boiling range characteristics of such products as light and middle distillates, automotive spark-ignition engine fuels, aviation gasolines, aviation turbine fuels, 1-D and 2-D regular and low sulfur diesel fuels, special petroleum spirits, naphthas, white spirits, kerosines, and Grades 1 and 2 burner fuels.

1.2 The test method is designed for the analysis of distillate fuels; it is not applicable to products containing appreciable quantities of residual material.

1.3 This test method covers both manual and automated instruments.

1.4 Unless otherwise noted, the values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for information only.

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 All standards are subject to revision, and parties to agreement on this test method are to apply the most recent edition of the standards indicated below, unless otherwise specified, such as in contractual agreements or regulatory rules where earlier versions of the method(s) identified may be required.

2.2 *ASTM Standards:* [2]

D 97 Test Method for Pour Point of Petroleum Products
D 323 Test Method for Vapor Pressure of Petroleum Products (Reid Method)
D 2892 Test Method for Distillation of Crude Petroleum (15-Theoretical Plate Column)
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D 4953 Test Method for Vapor Pressure of Gasoline and Gasoline-Oxygenate Blends (Dry Method)
D 5190 Test Method for Vapor Pressure of Petroleum Products (Automatic Method)
D 5191 Test Method for Vapor Pressure of Petroleum Products (Mini Method)
D 5842 Practice for Sampling and Handling of Fuels for Volatility Measurement
D 5949 Test Method for Pour Point of Petroleum Products (Automatic Pressure Pulsing Method)
D 5950 Test Method for Pour Point of Petroleum Products (Automatic Tilt Method)
D 5985 Test Method for Pour Point of Petroleum Products (Rotational Method)
E 1 Specification for ASTM Liquid-in-Glass Thermometers
E 77 Test Method for Inspection and Verification of Thermometers
E 1272 Specification for Laboratory Glass Graduated Cylinders
E 1405 Specification for Laboratory Glass Distillation Flasks

2.3 *Energy Institute Standards:*[3]

IP 69 Determination of Vapour Pressure—Reid Method
IP 123 Petroleum Products—Determination of Distillation Characteristics
IP 394 Determination of Air Saturated Vapour Pressure
IP Standard Methods for Analysis and Testing of Petroleum and Related Products 1996—Appendix A

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.08.0A on Distillation.

In the IP, the equivalent test method is published under the designation IP 123. It is under the jurisdiction of the Standardization Committee.

Current edition approved Jan. 15, 2007. Published February 2007. Originally approved in 1921. Last previous edition approved in 2005 as D 86–05.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., http://www.energyinst.org.uk.

*A Summary of Changes section appears at the end of this standard.

TABLE 1 Preparation of Apparatus

	Group 1	Group 2	Group 3	Group 4
Flask, mL	125	125	125	125
ASTM distillation thermometer	7C (7F)	7C (7F)	7C (7F)	8C (8F)
IP distillation thermometer range	low	low	low	high
Flask support board	B	B	C	C
diameter of hole, mm	38	38	50	50
Temperature at start of test				
Flask °C	13–18	13–18	13–18	not above
°F	55–65	55–65	55–65	ambient
Flask support and shield	not above ambient	not above ambient	not above ambient	
Receiving cylinder and 100 mL charge				
°C	13–18	13–18	13–18[A]	13–ambient[A]
°F	55–65	55–65	55–65[A]	55–ambient[A]

[A] See 10.3.1.1 for exceptions.

3. Terminology

3.1 *Definitions:*

3.1.1 *charge volume, n*—the volume of the specimen, 100 mL, charged to the distillation flask at the temperature specified in Table 1.

3.1.2 *decomposition, n—of a hydrocarbon,* the pyrolysis or cracking of a molecule yielding smaller molecules with lower boiling points than the original molecule.

3.1.2.1 *Discussion*—Characteristic indications of thermal decomposition are evolution of fumes and erratic temperature readings that usually decrease after any attempt is made to adjust the heat.

3.1.3 *decomposition point, n*—the corrected thermometer reading that coincides with the first indications of thermal decomposition of the liquid in the flask.

3.1.3.1 *Discussion*—The decomposition point, as determined under the conditions of this test method, does not necessarily correspond to the decomposition temperature in other applications.

3.1.4 *dry point, n*—the corrected thermometer reading that is observed at the instant the last drop of liquid (exclusive of any drops or film of liquid on the side of the flask or on the temperature sensor), evaporates from the lowest point in the distillation flask.

3.1.4.1 *Discussion*—The end point (final boiling point), rather than the dry point, is intended for general use. The dry point can be reported in connection with special purpose naphthas, such as those used in the paint industry. Also, it is substituted for the end point (final boiling point) whenever the sample is of such a nature that the precision of the end point (final boiling point) cannot consistently meet the requirements given in the precision section.

3.1.5 *dynamic holdup, n*—the amount of material present in the neck of the flask, in the sidearm of the flask, and in the condenser tube during the distillation.

3.1.6 *emergent stem effect, n*—the offset in temperature reading caused by the use of total immersion mercury-in-glass thermometers in the partial immersion mode.

3.1.6.1 *Discussion*—In the partial immersion mode, a portion of the mercury thread, that is, the emergent portion, is at a lower temperature than the immersed portion, resulting in a shrinkage of the mercury thread and a lower temperature reading.

3.1.7 *end point (EP) or final boiling point (FBP), n*—the maximum corrected thermometer reading obtained during the test.

3.1.7.1 *Discussion*—This usually occurs after the evaporation of all liquid from the bottom of the flask. The term maximum temperature is a frequently used synonym.

3.1.8 *front end loss, n*—loss due to evaporation during transfer from receiving cylinder to distillation flask, vapor loss during the distillation, and uncondensed vapor in the flask at the end of the distillation.

3.1.9 *initial boiling point (IBP), n*—the corrected thermometer reading that is observed at the instant the first drop of condensate falls from the lower end of the condenser tube.

3.1.10 *percent evaporated, n*—the sum of the percent recovered and the percent loss.

3.1.11 *percent loss (or observed loss), n*—one hundred minus the percent total recovery.

3.1.11.1 *corrected loss, n*—percent loss corrected for barometric pressure.

3.1.12 *percent recovered, n*—the volume of condensate observed in the receiving cylinder, expressed as a percentage of the charge volume, associated with a simultaneous temperature reading.

3.1.13 *percent recovery, n*—the maximum percent recovered, as observed in accordance with 10.18.

3.1.13.1 *corrected percent recovery, n*—the percent recovery, adjusted for the difference between the observed loss and the corrected loss, as described in Eq 8.

3.1.13.2 *percent total recovery, n*—the combined percent recovery and residue in the flask, as determined in accordance with 11.1.

3.1.14 *percent residue, n*—the volume of residue in the flask, measured in accordance with 10.19, and expressed as a percentage of the charge volume.

3.1.15 *rate of change (or slope), n*—the change in temperature reading per percent evaporated or recovered, as described in 13.2.

3.1.16 *temperature lag, n*—the offset between the temperature reading obtained by a temperature sensing device and the true temperature at that time.

3.1.17 *temperature measurement device, n*—a thermometer, as described in 6.3.1, or a temperature sensor, as described in 6.3.2.

3.1.18 *temperature reading, n*—the temperature obtained by a temperature measuring device or system that is equal to the thermometer reading described in 3.1.19.

3.1.18.1 *corrected temperature reading, n*—the temperature reading, as described in 3.1.18, corrected for barometric pressure.

3.1.19 *thermometer reading (or thermometer result), n*—the temperature of the saturated vapor measured in the neck of the flask below the vapor tube, as determined by the prescribed thermometer under the conditions of the test.

3.1.19.1 *corrected thermometer reading, n*—the thermometer reading, as described in 3.1.19, corrected for barometric pressure.

4. Summary of Test Method

4.1 Based on its composition, vapor pressure, expected IBP or expected EP, or combination thereof, the sample is placed in one of four groups. Apparatus arrangement, condenser temperature, and other operational variables are defined by the group in which the sample falls.

4.2 A 100-mL specimen of the sample is distilled under prescribed conditions for the group in which the sample falls. The distillation is performed in a laboratory batch distillation unit at ambient pressure under conditions that are designed to provide approximately one theoretical plate fractionation. Systematic observations of temperature readings and volumes of condensate are made, depending on the needs of the user of the data. The volume of the residue and the losses are also recorded.

4.3 At the conclusion of the distillation, the observed vapor temperatures can be corrected for barometric pressure and the data are examined for conformance to procedural requirements, such as distillation rates. The test is repeated if any specified condition has not been met.

4.4 Test results are commonly expressed as percent evaporated or percent recovered versus corresponding temperature, either in a table or graphically, as a plot of the distillation curve.

5. Significance and Use

5.1 The basic test method of determining the boiling range of a petroleum product by performing a simple batch distillation has been in use as long as the petroleum industry has existed. It is one of the oldest test methods under the jurisdiction of ASTM Committee D02, dating from the time when it was still referred to as the Engler distillation. Since the test method has been in use for such an extended period, a tremendous number of historical data bases exist for estimating end-use sensitivity on products and processes.

5.2 The distillation (volatility) characteristics of hydrocarbons have an important effect on their safety and performance, especially in the case of fuels and solvents. The boiling range gives information on the composition, the properties, and the behavior of the fuel during storage and use. Volatility is the major determinant of the tendency of a hydrocarbon mixture to produce potentially explosive vapors.

5.3 The distillation characteristics are critically important for both automotive and aviation gasolines, affecting starting, warm-up, and tendency to vapor lock at high operating temperature or at high altitude, or both. The presence of high boiling point components in these and other fuels can significantly affect the degree of formation of solid combustion deposits.

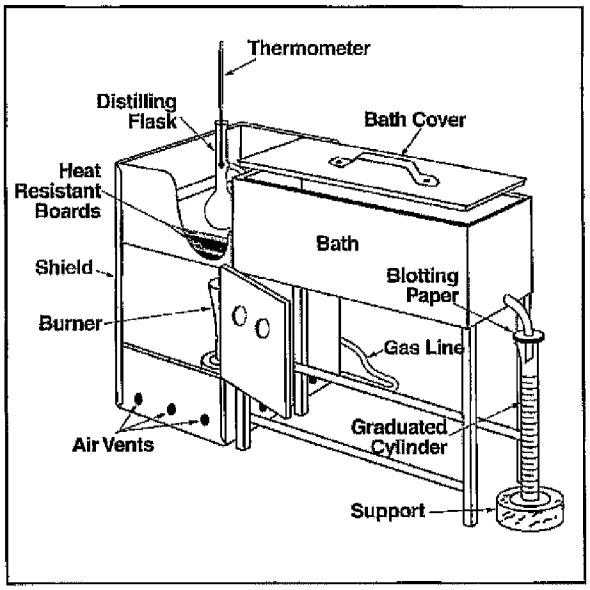


FIG. 1 Apparatus Assembly Using Gas Burner

5.4 Volatility, as it affects rate of evaporation, is an important factor in the application of many solvents, particularly those used in paints.

5.5 Distillation limits are often included in petroleum product specifications, in commercial contract agreements, process refinery/control applications, and for compliance to regulatory rules.

6. Apparatus

6.1 *Basic Components of the Apparatus*:

6.1.1 The basic components of the distillation unit are the distillation flask, the condenser and associated cooling bath, a metal shield or enclosure for the distillation flask, the heat source, the flask support, the temperature measuring device, and the receiving cylinder to collect the distillate.

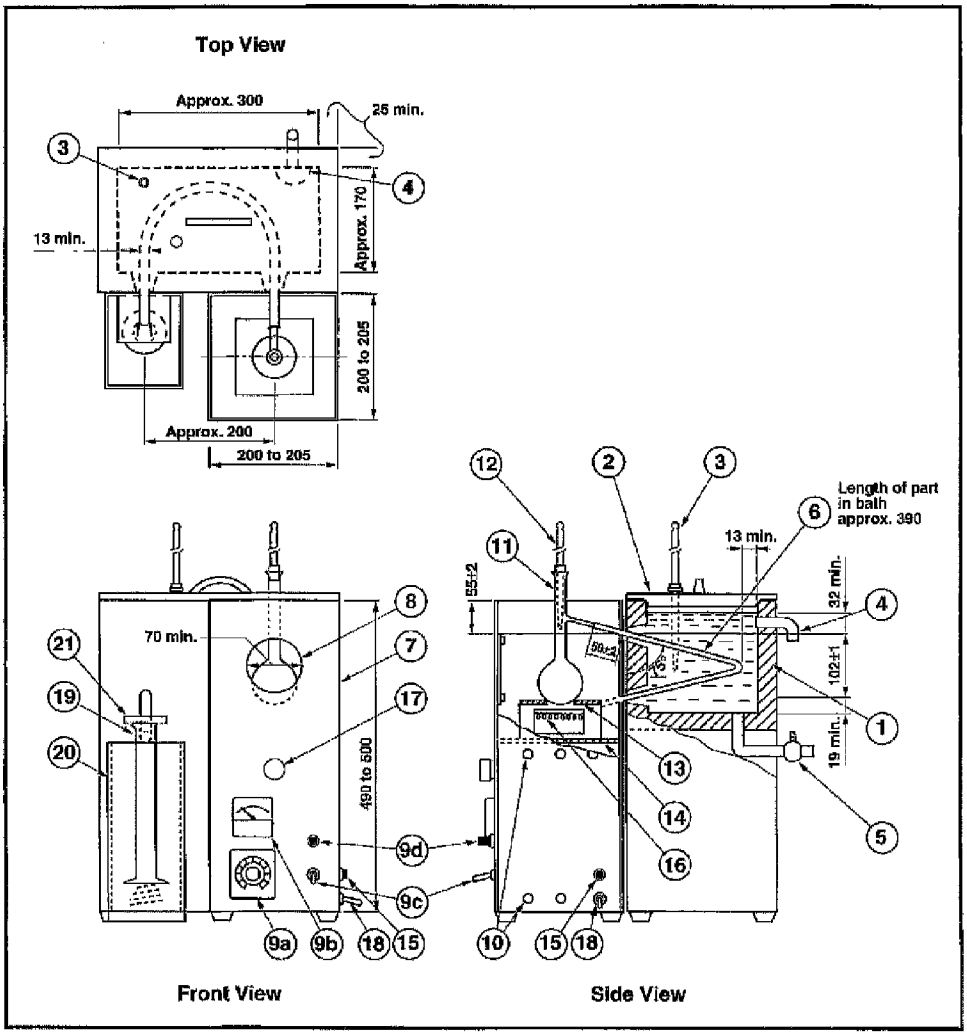
6.1.2 Figs. 1 and 2 are examples of manual distillation units.

6.1.3 In addition to the basic components described in 6.1.1, automated units also are equipped with a system to measure and automatically record the temperature and the associated recovered volume in the receiving cylinder.

6.2 A detailed description of the apparatus is given in Annex A2.

6.3 *Temperature Measuring Device*:

6.3.1 Mercury-in-glass thermometers, if used, shall be filled with an inert gas, graduated on the stem and enamel backed. They shall conform to Specification E 1 or IP Standard Methods for Analysis and Testing of Petroleum and Related Products 1996—Appendix A, or both, for thermometers ASTM



1–Condenser bath
2–Bath cover
3–Bath temperature sensor
4–Bath overflow
5–Bath drain
6–Condenser tube
7–Shield
8–Viewing window
9a–Voltage regulator
9b–Voltmeter or ammeter
9c–Power switch
9d–Power light indicator
10–Vent
11–Distillation flask
12–Temperature sensor
13–Flask support board
14–Flask support platform
15–Ground connection
16–Electric heater
17–Knob for adjusting level of support platform
18–Power source cord
19–Receiver cylinder
20–Receiver cooling bath
21–Receiver cover

FIG. 2 Apparatus Assembly Using Electric Heater

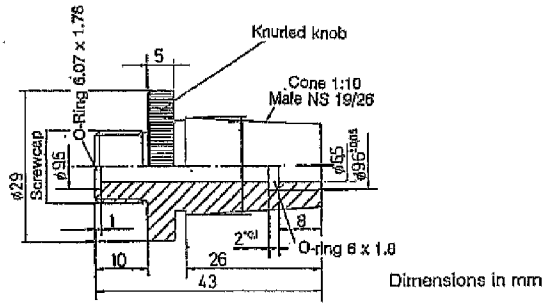


FIG. 3 PTFE Centering Device for Ground Glass Joint

7C/IP 5C and ASTM 7F for the low range thermometers, and ASTM 8C/IP 6C and ASTM 8F for the high range thermometers.

6.3.1.1 Thermometers that have been exposed for an extended period above an observed temperature of 370°C shall not be reused without a verification of the ice point or checked as prescribed in Specification E 1 and Test Method E 77.

NOTE 1—At an observed thermometer reading of 370°C, the temperature of the bulb is approaching a critical range in the glass and the thermometer may lose its calibration.

6.3.2 Temperature measurement systems other than those described in 6.3.1 are satisfactory for this test method, provided that they exhibit the same temperature lag, emergent stem effect, and accuracy as the equivalent mercury-in-glass thermometer.

6.3.2.1 The electronic circuitry or the algorithms, or both, used shall include the capability to simulate the temperature lag of a mercury-in-glass thermometer.

6.3.2.2 Alternatively, the sensor can also be placed in a casing with the tip of the sensor covered so that the assembly, because of its adjusted thermal mass and conductivity, has a temperature lag time similar to that of a mercury-in-glass thermometer.

NOTE 2—In a region where the temperature is changing rapidly during the distillation, the temperature lag of a thermometer can be as much as 3 seconds.

6.3.3 In case of dispute, the referee test method shall be carried out with the specified mercury-in-glass thermometer.

6.4 *Temperature Sensor Centering Device*:

6.4.1 The temperature sensor shall be mounted through a snug-fitting device designed for mechanically centering the sensor in the neck of the flask without vapor leakage. Examples of acceptable centering devices are shown in Figs. 3 and 4. (**Warning**—The use of a plain stopper with a hole drilled through the center is not acceptable for the purpose described in 6.4.1.)

NOTE 3—Other centering devices are also acceptable, as long as they position and hold the temperature sensing device in the proper position in the neck of the distillation column, as shown in Fig. 5 and described in 10.5.

NOTE 4—When running the test by the manual method, products with a low IBP may have one or more readings obscured by the centering device. See also 10.14.3.1.

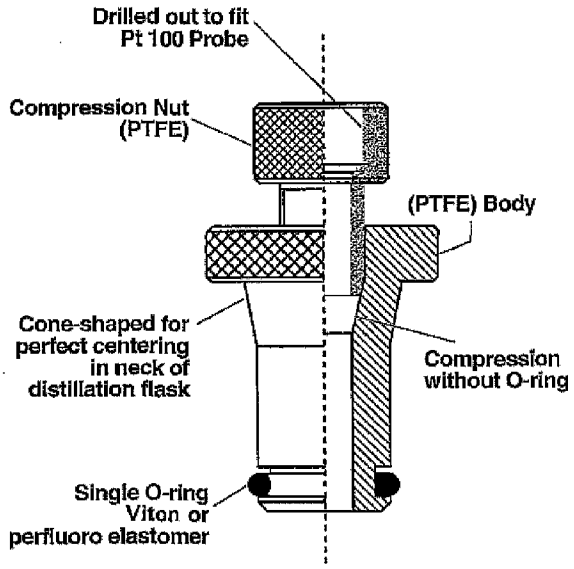


FIG. 4 Example of Centering Device Designs for Straight-Bore Neck Flasks

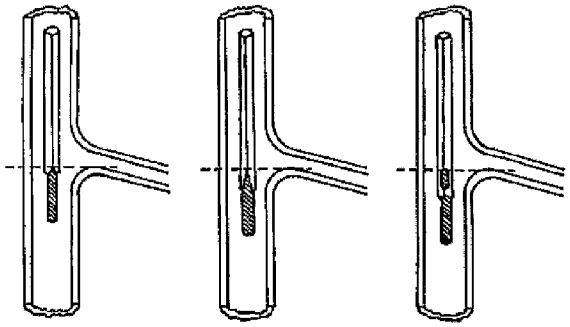


FIG. 5 Position of Thermometer in Distillation Flask

6.5 Automated equipment manufactured in 1999 and later shall be equipped with a device to automatically shut down power to the unit and to spray an inert gas or vapor in the chamber where the distillation flask is mounted in the event of fire.

NOTE 5—Some causes of fires are breakage of the distillation flask, electrical shorts, and foaming and spilling of liquid sample through the top opening of the flask.

6.6 *Barometer*—A pressure measuring device capable of measuring local station pressure with an accuracy of 0.1 kPa (1 mm Hg) or better, at the same elevation relative to sea level as the apparatus in the laboratory. (**Warning**—Do not take readings from ordinary aneroid barometers, such as those used

TABLE 2 Group Characteristics

	Group 1	Group 2	Group 3	Group 4
Sample characteristics				
Distillate type				
Vapor pressure at				
37.8°C, kPa	≥65.5	<65.5	<65.5	<65.5
100°F, psi	≥9.5	<9.5	<9.5	<9.5
(Test Methods D 323, D 4953, D 5190, D 5191, D 5482, IP 69 or IP 394)				
Distillation, IBP °C			≤100	>100
°F			≤212	>212
EP °C	≤250	≤250	>250	>250
°F	≤482	≤482	>482	>482

at weather stations and airports, since these are precorrected to give sea level readings.)

7. Sampling, Storage, and Sample Conditioning

7.1 Determine the Group characteristics that correspond to the sample to be tested (see Table 2). Where the procedure is dependent upon the group, the section headings will be so marked.

7.2 *Sampling*:

7.2.1 Sampling shall be done in accordance with Practice D 4057 or D 4177 and as described in Table 3.

7.2.1.1 *Group 1*—Condition the sample container to below 10°C, preferably by filling the bottle with the cold liquid sample and discarding the first sample. If this is not possible because, for instance, the product to be sampled is at ambient temperature, the sample shall be drawn into a bottle prechilled to below 10°C, in such a manner that agitation is kept at a minimum. Close the bottle immediately with a tight-fitting closure. (**Warning**—Do not completely fill and tightly seal a cold bottle of sample because of the likelihood of breakage on warming.)

7.2.1.2 *Groups 2, 3, and 4*—Collect the sample at ambient temperature. After sampling, close the sample bottle immediately with a tight-fitting closure.

7.2.1.3 If the sample received by the testing laboratory has been sampled by others and it is not known whether sampling has been performed as described in 7.2, the sample shall be assumed to have been so sampled.

7.3 *Sample Storage*:

7.3.1 If testing is not to start immediately after collection, store the samples as indicated in 7.3.2, 7.3.3, and Table 3. All samples shall be stored away from direct sunlight or sources of direct heat.

7.3.2 *Group 1*—Store the sample at a temperature below 10°C.

NOTE 6—If there are no, or inadequate, facilities for storage below 10°C, the sample may also be stored at a temperature below 20°C, provided the operator ensures that the sample container is tightly closed and leak-free.

7.3.3 *Group 2*—Store the sample at a temperature below 10°C.

NOTE 7—If there are no, or inadequate, facilities for storage below 10°C, the sample may also be stored at a temperature below 20°C, provided the operator ensures that the sample container is tightly closed and leak-free.

7.3.4 *Groups 3 and 4*—Store the sample at ambient or lower temperature.

7.4 *Sample Conditioning Prior to Analysis*:

7.4.1 Samples shall be conditioned to the temperature shown in Table 3 before opening the sample container.

7.4.1.1 *Groups 1 and 2*—Samples shall be conditioned to a temperature of less than 10°C (50°F) before opening the sample container.

7.4.1.2 *Groups 3 and 4*—If the sample is not fluid at ambient temperature, it is to be heated to a temperature of 9 to 21°C above its pour point (Test Method D 97, D 5949, or D 5985) prior to analysis. If the sample has partially or completely solidified during storage, it shall be vigorously shaken after melting prior to opening the sample container to ensure homogeneity.

7.4.1.3 If the sample is not fluid at room temperature, the temperature ranges shown in Table 3 for the flask and for the sample do not apply.

7.5 *Wet Samples*:

7.5.1 Samples of materials that visibly contain water are not suitable for testing. If the sample is not dry, obtain another sample that is free from suspended water.

7.5.2 *Groups 1 and 2*—If such a sample cannot be obtained, the suspended water can be removed by maintaining the sample at 0 to 10°C, adding approximately 10 g of anhydrous sodium sulfate per 100 mL of sample, shaking the mixture for approximately 2 min, and then allowing the mixture to settle for approximately 15 min. Once the sample shows no visible signs of water, use a decanted portion of the sample, maintained between 1 and 10°C, for the analysis. Note in the report that the sample has been dried by the addition of a desiccant.

NOTE 8—Suspended water in hazy samples in Groups 1 and 2 can be removed by the addition of anhydrous sodium sulfate and separating the liquid sample from the drying agent by decanting without statistically affecting the results of the test.[4]

7.5.3 *Groups 3 and 4*—In cases in which a water-free sample is not practical, the suspended water can be removed by shaking the sample with anhydrous sodium sulfate or other suitable drying agent and separating it from the drying agent by decanting. Note in the report that the sample has been dried by the addition of a desiccant.

8. Preparation of Apparatus

8.1 Refer to Table 1 and prepare the apparatus by choosing the appropriate distillation flask, temperature measuring device, and flask support board, as directed for the indicated group. Bring the temperature of the receiving cylinder, the flask, and the condenser bath to the indicated temperature.

8.2 Make any necessary provisions so that the temperature of the condenser bath and the receiving cylinder will be maintained at the required temperatures. The receiving cylinder shall be in a bath such that either the liquid level is at least

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1455.

TABLE 3 Sampling, Storage, and Sample Conditioning

		Group 1	Group 2	Group 3	Group 4
Temperature of sample bottle	°C	<10			
	°F	<50			
Temperature of stored sample	°C	<10[A]	<10	ambient	ambient
	°F	<50[A]	<50	ambient	ambient
Temperature of sample after conditioning prior to analysis	°C	<10	<10	Ambient or 9 to 21°C above pour point[B]	Ambient or
	°F	<50	<50	Ambient or 48 to 70°F above pour point[B]	Ambient or
If sample is wet		resample	resample	dry in accordance with 7.5.3	
If resample is still wet[C]		dry in accordance with 7.5.2			

[A] Under certain circumstances, samples can also be stored at temperatures below 20°C (68°F). See also 7.3.2 and 7.3.3.
[B] If sample is (semi)-solid at ambient temperature, see also 10.3.1.1.
[C] If sample is known to be wet, resampling may be omitted. Dry sample in accordance with 7.5.2 and 7.5.3.

as high as the 100-mL mark or the entire receiving cylinder is surrounded by an air circulation chamber.

8.2.1 *Groups 1, 2, and 3*—Suitable media for low temperature baths include, but are not limited to, chopped ice and water, refrigerated brine, and refrigerated ethylene glycol.

8.2.2 *Group 4*—Suitable media for ambient and higher bath temperatures include, but are not limited to, cold water, hot water, and heated ethylene glycol.

8.3 Remove any residual liquid in the condenser tube by swabbing with a piece of soft, lint-free cloth attached to a cord or wire.

9. Calibration and Standardization

9.1 *Temperature Measurement System*—Temperature measurement systems using other than the specified mercury-in-glass thermometers shall exhibit the same temperature lag, emergent stem effect, and accuracy as the equivalent mercury-in-glass thermometer. Confirmation of the calibration of these temperature measuring systems shall be made at intervals of not more than six months, and after the system has been replaced or repaired.

9.1.1 The accuracy and the calibration of the electronic circuitry or computer algorithms, or both, shall be verified by the use of a standard precision resistance bench. When performing this verification, no algorithms shall be used to correct the temperature for lag and the emergent stem effect (see manufacturer's instructions).

9.1.2 Verification of the calibration of temperature measuring devices shall be conducted by distilling toluene in accordance with Group 1 of this test method and comparing the 50 % recovered temperature with that shown in Table 4.[5]

9.1.2.1 If the temperature reading is not within the values shown in Table 4 for the respective apparatus being used (see Note 10 and Table 4), the temperature measurement system shall be considered defective and shall not be used for the test.

NOTE 9—Toluene is used as a verification fluid for calibration; it will yield almost no information on how well an electronic measurement system simulates the temperature lag of a liquid-in-glass thermometer.

9.1.2.2 Reagent grade toluene and hexadecane (cetane), conforming to the specifications of the Committee on Analytical Reagents of the American Chemical Society,[6] shall be used. However, other grades may also be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

NOTE 10—At 101.3 kPa, toluene is shown in reference manuals as boiling at 110.6°C when measured using a partial immersion thermometer. Because this test method uses thermometers calibrated for total immersion, the results typically will be lower and, depending on the thermometer and the situation, may be different for each thermometer. At 101.3 kPa, hexadecane is shown in reference manuals as boiling at 287.0°C when measured using a partial immersion thermometer. Because this test method uses thermometers calibrated for total immersion, the results typically will be lower, and, depending on the thermometer and the situation, may be different for each thermometer.

9.1.3 A procedure to determine the magnitude of the temperature lag is described in Annex A3.

9.1.4 A procedure to emulate the emergent stem effect is described in Appendix X4.

9.1.5 To verify the calibration of the temperature measurement system at elevated temperatures, use hexadecane. The temperature measurement system shall indicate, at 50% recovered, a temperature comparable to that shown in Table 4 for the respective apparatus under Group 4 distillation conditions.

NOTE 11—Because of the high melting point of hexadecane, Group 4 verification distillations will have to be carried out with condenser temperatures >20°C.

9.2 *Automated Method*:

9.2.1 *Level Follower*—For an automated distillation apparatus, the level follower/recording mechanism of the apparatus shall have a resolution of 0.1 mL or better with a maximum error of 0.3 mL between the 5 and 100 mL points. The calibration of the assembly shall be verified in accordance with manufacturer's instructions at intervals of not more than three months and after the system has been replaced or repaired.

NOTE 12—The typical calibration procedure involves verifying the output with the receiver containing 5 and 100 mL of material respectively.

9.2.2 *Barometric Pressure*—At intervals of not more than six months, and after the system has been replaced or repaired,

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1580.

[6] *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.

TABLE 4 True and Min and Max D 86 50 % Recovered Boiling Points (°C)[A]

		Manual		Automated	
		Distillation conditions min D 86 50 % boiling point	Distillation conditions max D 86 50 % boiling point	Distillation conditions min D 86 50 % boiling point	Distillation conditions max D 86 50 % boiling point
Toluene	ASTM/IP true boiling point	Group 1, 2, and 3	Group 1, 2, and 3	Group 1, 2, and 3	Group 1, 2, and 3
	110.6	105.9	111.8	108.5	108.7
Hexadecane	ASTM/IP true boiling point	Group 4	Group 4	Group 4	Group 4
	287.0	272.2	283.1	277.0	280.0

[A] The manual and automated temperatures show in this table are the values for the 95 % tolerance interval for the 99 % population coverage. The proposed tolerance is approximately 3 × sigma. Information on the values in this table can be found in RR:D02–1560.

the barometric reading of the instrument shall be verified against a barometer, as described in 6.6.

10. Procedure

10.1 Record the prevailing barometric pressure.

10.2 *Groups 1 and 2*—Fit a low range thermometer provided with a snug-fitting cork or stopper of silicone rubber, or equivalent polymeric material, tightly into the neck of the sample container and bring the temperature of the sample to the temperature indicated in Table 3.

10.3 *Groups 1, 2, 3, and 4*—Check that the temperature of the sample is as shown in Table 3. Pour the specimen precisely to the 100-mL mark of the receiving cylinder, and transfer the contents of the receiving cylinder as completely as practical into the distillation flask, ensuring that none of the liquid flows into the vapor tube.

NOTE 13—It is important that the difference between the temperature of the specimen and the temperature of the bath around the receiving cylinder is as small as practically possible. A difference of 5°C can make a difference of 0.7 mL.

10.3.1 *Groups 3 and 4*—If the sample is not fluid at ambient temperature, it is to be heated to a temperature between 9 and 21°C above its pour point (Test Methods D 97, D 5949, D 5950, or D 5985) prior to analysis. If the sample has partially or completely solidified in the intervening period, it shall be vigorously shaken after melting, and prior to sampling, to ensure homogeneity.

10.3.1.1 If the sample is not fluid at ambient temperatures, disregard the temperature range shown in Table 1 for the receiving cylinder and sample. Prior to analysis, heat the receiving cylinder to approximately the same temperature as the sample. Pour the heated specimen precisely to the 100-mL mark of the receiving cylinder, and transfer the contents of the receiving cylinder as completely as practical into the distillation flask, ensuring that none of the liquid flows into the vapor tube.

NOTE 14—Any material that evaporates during the transfer will contribute to the loss; any material that remains in the receiving cylinder will contribute to the observed recovery volume at the time of the IBP.

10.4 If the sample can be expected to demonstrate irregular boiling behavior, that is, bumping, add a few boiling chips to the specimen. The addition of a few boiling chips is acceptable for any distillation.

10.5 Fit the temperature sensor through a snug-fitting device, as described in 6.4, to mechanically center the sensor in the neck of the flask. In the case of a thermometer, the bulb is centered in the neck and the lower end of the capillary is level with the highest point on the bottom of the inner wall of the vapor tube (see Fig. 5). In the case of a thermocouple or resistance thermometer, follow the manufacturer's instructions as to placement (see Fig. 6).

NOTE 15—If vacuum grease is used on the mating surface of the centering device, use the minimum amount of grease that is practical.

10.6 Fit the flask vapor tube, provided with a snug-fitting cork or rubber stopper of silicone, or equivalent polymeric material, tightly into the condenser tube. Adjust the flask in a vertical position so that the vapor tube extends into the condenser tube for a distance from 25 to 50 mm. Raise and adjust the flask support board to fit it snugly against the bottom of the flask.

10.7 Place the receiving cylinder that was used to measure the specimen, without drying the inside of the cylinder, into its temperature-controlled bath under the lower end of the condenser tube. The end of the condenser tube shall be centered in the receiving cylinder and shall extend therein for a distance of at least 25 mm, but not below the 100-mL mark.

10.8 *Initial Boiling Point*:

10.8.1 *Manual Method*—To reduce evaporation loss of the distillate, cover the receiving cylinder with a piece of blotting paper, or similar material, that has been cut to fit the condenser tube snugly. If a receiver deflector is being used, start the distillation with the tip of the deflector just touching the wall of the receiving cylinder. If a receiver deflector is not used, keep the drip tip of the condenser away from the wall of the receiving cylinder. Note the start time. Observe and record the IBP to the nearest 0.5°C (1.0°F). If a receiver deflector is not being used, immediately move the receiving cylinder so that the tip of the condenser touches its inner wall.

10.8.2 *Automated Method*—To reduce evaporation loss of the distillate, use the device provided by the instrument manufacturer for this purpose. Apply heat to the distillation flask and contents with the tip of the receiver deflector just touching the wall of the receiving cylinder. Note the start time. Record the IBP to the nearest 0.1°C (0.2°F).

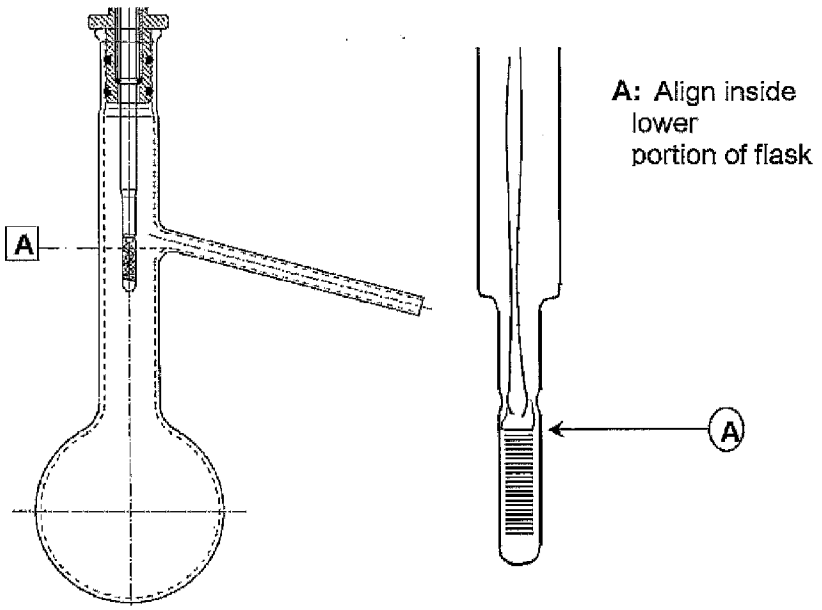


FIG. 6 Example of Recommended Placement of Pt-100 Probe Relative to Distillation Flask Sidearm for Automated D 86 Distillation Instrument

10.9 Regulate the heating so that the time interval between the first application of heat and the IBP is as specified in Table 5.

10.10 Regulate the heating so that the time from IBP to 5 or 10 % recovered is as indicated in Table 5.

10.11 Continue to regulate the heating so that the uniform average rate of condensation from 5 or 10 % recovered to 5 mL residue in the flask is 4 to 5 mL per min. (**Warning**—Due to the configuration of the boiling flask and the conditions of the test, the vapor and liquid around the temperature sensor are not in thermodynamic equilibrium. The distillation rate will consequently have an effect on the measured vapor temperature. The distillation rate shall, therefore, be kept as constant as possible throughout the test.)

NOTE 16—When testing gasoline samples, it is not uncommon to see the condensate suddenly form non-miscible liquid phases and bead up on the temperature measuring device and in the neck of the boiling flask at a vapor temperature of around 160°C. This may be accompanied by a sharp (about 3°C) dip in the vapor temperature and a drop in the recovery rate. The phenomenon, which may be due to the presence of trace water in the sample, may last for 10 to 30 s before the temperature recovers and the condensate starts flowing smoothly again. This point is sometimes colloquially referred to as the Hesitation Point.

10.12 Repeat any distillation that did not meet the requirements described in 10.9, 10.10, and 10.11.

10.13 If a decomposition point, as described in 3.1.3, is observed, discontinue the heating and proceed as directed in 10.17.

10.14 In the interval between the IBP and the end of the distillation, observe and record data necessary for the calculation and reporting of the results of the test as required by the specification involved, or as previously established for the sample under test. These observed data can include temperature readings at prescribed percentages recovered or percentages recovered at prescribed temperature readings, or both.

10.14.1 *Manual Method*—Record all volumes in the graduated cylinder to the nearest 0.5 mL, and all temperature readings to the nearest 0.5°C (1.0°F).

10.14.2 *Automated Method*—Record all volumes in the receiving cylinder to the nearest 0.1 mL, and all temperature readings to the nearest 0.1°C (0.2°F).

10.14.3 *Group 1, 2, 3, and 4*—In cases in which no specific data requirements have been indicated, record the IBP and the EP (FBP) or the dry point, or both, and temperature readings at 5, 15, 85, and 95 % recovered, and at each 10 % multiple of volume recovered from 10 to 90, inclusive.

10.14.3.1 *Group 4*—When a high range thermometer is used in testing aviation turbine fuels and similar products, pertinent thermometer readings can be obscured by the centering device. If these readings are required, perform a second distillation in accordance with Group 3. In such cases, reading from a low range thermometer can be reported in place of the obscured high range thermometer readings, and the test report shall so indicate. If, by agreement, the obscured readings are waived, the test report shall so indicate.

10.14.4 When it is required to report the temperature reading at a prescribed percent evaporated or recovered for a sample that has a rapidly changing slope of the distillation curve in the region of the prescribed percent evaporated or recovered reading, record temperature readings at every 1 % recovered. The slope is considered rapidly changing if the

TABLE 5 Conditions During Test Procedure

		Group 1	Group 2	Group 3	Group 4
Temperature of cooling bath[A]	°C	0–1	0–5	0–5	0–60
	°F	32–34	32–40	32–40	32–140
Temperature of bath around receiving cylinder	°C	13–18	13–18	13–18	±3
	°F	55 65	55–65	55–65	±5 of charge temperature
Time from first application of heat to initial boiling point, min		5–10	5–10	5–10	5–15
Time from initial boiling point to 5 % recovered, s		60–100	60–100		
to 10 % recovered, min					
Uniform average rate of condensation from 5 % recovered to 5 mL in flask, mL/min		4–5	4–5	4–5	4–6
Time recorded from 5 mL residue to end point, min		5 max	5 max	5 max	5 max

[A] the proper condenser bath temperature will depend upon the wax content of the sample and of its distillation fractions. The test is generally performed using one single condenser temperature. Wax formation in the condenser can be deduced from (*a*) the presence of wax particles in the distillate coming off the drip tip, (*b*) a higher distillation loss than what would be expected based on the initial boiling point of the specimen, (*c*) an erratic recovery rate and (*d*) the presence of wax particles during the removal of residual liquid by swabbing with a lint-free cloth (see 8.3). The minimum temperature that permits satisfactory operation shall be used. In general, a bath temperature in the 0 to 4°C range is suitable for kerosine, Grade No. 1 fuel oil and Grade No. 1-D diesel fuel oil. In some cases involving Grade No. 2 fuel oil, Grade No. 2-D diesel fuel oil, gas oils and similar distillates, it may be necessary to hold the condenser bath temperature in the 38 to 60°C range.

change in slope (C) of the data points described in 10.14.2 in that particular area is greater than 0.6 (change of slope (F) is greater than 1.0) as calculated by Eq 1 (Eq 2).

$$\text{Change of Slope } (C) = (C_2 - C_1)/(V_2 - V_1) - (C_3 - C_2)/(V_3 - V_2) \quad (1)$$

$$\text{Change of Slope } (F) = (F_2 - F_1)/(V_2 - V_1) - (F_3 - F_2)/(V_3 - V_2) \quad (2)$$

where:

C_1 = temperature at the volume % recorded one reading prior to the volume % in question, °C,
C_2 = temperature at the volume % recorded in question, °C,
C_3 = temperature at the volume % recorded following the volume % in question, °C,
F_1 = temperature at the volume % recorded one reading prior to the volume % in question, °F,
F_2 = temperature at the volume % recorded in question, °F,
F_3 = temperature at the volume % recorded following the volume % in question, °F,
V_1 = volume % recorded one reading prior to the volume % in question,
V_2 = volume % recorded at the volume % in question, and
V_3 = volume % recorded following the volume % in question.

10.15 When the residual liquid in the flask is approximately 5 mL, make a final adjustment of the heat. The time from the 5 mL of liquid residue in the flask to the EP (FBP) shall be within the limits prescribed in Table 5. If this condition is not satisfied, repeat the test with appropriate modification of the final heat adjustment.

NOTE 17—Since it is difficult to determine when there is 5 mL of boiling liquid left in the flask, this time is determined by observing the amount of liquid recovered in the receiving cylinder. The dynamic holdup has been determined to be approximately 1.5 mL at this point. If there are no front end losses, the amount of 5 mL in the flask can be assumed to correspond with an amount of 93.5 mL in the receiving cylinder. This amount has to be adjusted for the estimated amount of front end loss.

10.15.1 If the actual front end loss differs more than 2 mL from the estimated value, the test shall be rerun.

10.16 Observe and record the EP (FBP) or the dry point, or both, as required, and discontinue the heating.

10.17 Allow the distillate to drain into the receiving cylinder, after heating has been discontinued.

10.17.1 *Manual Method*—While the condenser tube continues to drain into the graduated cylinder, observe and note the volume of condensate to the nearest 0.5 mL at 2 min intervals until two successive observations agree. Measure the volume in the receiving cylinder accurately, and record it to the nearest 0.5 mL.

10.17.2 *Automated Method*—The apparatus shall continually monitor the recovered volume until this volume changes by no more than 0.1 mL in 2 min. Record the volume in the receiving cylinder accurately to the nearest 0.1 mL.

10.18 Record the volume in the receiving cylinder as percent recovery. If the distillation was previously discontinued under the conditions of a decomposition point, deduct the percent recovered from 100, report this difference as the sum of percent residue and percent loss, and omit the procedure given in 10.19.

10.19 After the flask has cooled and no more vapor is observed, disconnect the flask from the condenser, pour its contents into a 5-mL graduated cylinder, and with the flask suspended over the cylinder, allow the flask to drain until no appreciable increase in the volume of liquid in the cylinder is observed. Measure the volume in the graduated cylinder to the nearest 0.1 mL, and record as percent residue.

10.19.1 If the 5-mL graduated cylinder does not have graduations below 1 mL and the volume of liquid is less than 1 mL, prefill the cylinder with 1 mL of a heavy oil to allow a better estimate of the volume of the material recovered.

10.19.1.1 If a residue greater than expected is obtained, and the distillation was not purposely terminated before the EP,

check whether adequate heat was applied towards the end of the distillation and whether conditions during the test conformed to those specified in Table 5. If not, repeat test.

NOTE 18—The distillation residues of this test method for gasoline, kerosine, and distillate diesel are *typically* 0.9–1.3, 0.9–1.3, and 1.0–1.4 volume %, respectively.

NOTE 19—The test method is not designed for the analysis of distillate fuels containing appreciable quantities of residual material (see 1.2).

10.19.2 *Groups 1, 2, 3, and 4*—Record the volume in the 5-mL graduated cylinder, to the nearest 0.1 mL, as percent residue.

10.20 If the intent of the distillation is to determine the percent evaporated or percent recovered at a predetermined corrected temperature reading, modify the procedure to conform to the instructions described in Annex A4.

10.21 Examine the condenser tube and the side arm of the flask for waxy or solid deposits. If found, repeat the test after making adjustments described in Footnote A of Table 5.

11. Calculations

11.1 The percent total recovery is the sum of the percent recovery (see 10.18) and the percent residue (see 10.19). Deduct the percent total recovery from 100 to obtain the percent loss.

11.2 Do not correct the barometric pressure for meniscus depression, and do not adjust the pressure to what it would be at sea level.

NOTE 20—The observed barometric reading does not have to be corrected to a standard temperature and to standard gravity. Even without performing these corrections, the corrected temperature readings for the same sample between laboratories at two different locations in the world will, in general, differ less than 0.1°C at 100°C. Almost all data obtained earlier have been reported at barometric pressures that have not been corrected to standard temperature and to standard gravity.

11.3 Correct temperature readings to 101.3 kPa (760 mm Hg) pressure. Obtain the correction to be applied to each temperature reading by means of the Sydney Young equation as given in Eq 3, Eq 4, or Eq 5, as appropriate, or by the use of Table 6. For Celsius temperatures:

$$C_c = 0.0009\,(101.3 - P_k)\,(273 + t_c) \quad (3)$$

$$C_c = 0.00012\,(760 - P)\,(273 + t_c) \quad (4)$$

For Fahrenheit temperatures:

$$C_f = 0.00012\,(760 - P)\,(460 + t_f) \quad (5)$$

where:

t_c = the observed temperature reading in °C,
t_f = the observed temperature reading in °F,
C_c and C_f = corrections to be added algebraically to the observed temperature readings,
P_k = barometric pressure, prevailing at the time and location of the test, kPa, and
P = barometric pressure, prevailing at the time and location of the test, mm Hg.

After applying the corrections and rounding each result to the nearest 0.5°C (1.0°F) or 0.1°C (0.2°F), as appropriate to the apparatus being used, use the corrected temperature readings in all further calculations and reporting.

TABLE 6 Approximate Thermometer Reading Correction

Temperature Range		Correction[A] per 1.3 kPa (10 mm Hg) Difference in Pressure	
°C	°F	°C	°F
10–30	50–86	0.35	0.63
30–50	86–122	0.38	0.68
50–70	122–158	0.40	0.72
70–90	158–194	0.42	0.76
90–110	194–230	0.45	0.81
110–130	230–266	0.47	0.85
130–150	266–302	0.50	0.89
150–170	302–338	0.52	0.94
170–190	338–374	0.54	0.98
190–210	374–410	0.57	1.02
210–230	410–446	0.59	1.07
230–250	446–482	0.62	1.11
250–270	482–518	0.64	1.15
270–290	518–554	0.66	1.20
290–310	554–590	0.69	1.24
310–330	590–626	0.71	1.28
330–350	626–662	0.74	1.33
350–370	662–698	0.76	1.37
370–390	698–734	0.78	1.41
390–410	734–770	0.81	1.46

[A] Values to be added when barometric pressure is below 101.3 kPa (760 mm Hg) and to be subtracted when barometric pressure is above 101.3 kPa.

NOTE 21—Temperature readings are not corrected to 101.3 kPa (760 mm Hg) when product definitions, specifications, or agreements between the parties involved indicate, specifically, that such correction is not required or that correction shall be made to some other base pressure.

11.4 Correct the actual loss to 101.3 kPa (760 mm Hg) pressure when temperature readings are corrected to 101.3 kPa pressure. The corrected loss, L_c, is calculated from Eq 6 or Eq 7, as appropriate, or can be read from the tables presented as Fig. X3.1 or Fig. X3.2.

$$L_c = 0.5 + (L - 0.5)/\{1 + (101.3 - P_k)/8.00\} \quad (6)$$

$$L_c = 0.5 + (L - 0.5)/\{1 + (760 - P)/60.0\} \quad (7)$$

where:

L = observed loss,
L_c = corrected loss,
P_k = pressure, kPa, and
P = pressure, mm Hg.

NOTE 22—Eq 6 and 7 above have been derived from the data in Table 7 and Eqs 5 and 6 in Test Method D 86 – 95 and earlier versions. It is probable that Eq 6 and 7 shown were the original empirical equations from which the table and equations in the Test Method D 86 – 95 and earlier versions were derived.

11.4.1 Calculate the corresponding corrected percent recovery in accordance with the following equation:

$$R_c = R + (L - L_c) \quad (8)$$

where:

L = percent loss or observed loss,
L_c = corrected loss,
R = percent recovery, and
R_c = corrected percent recovery.

TABLE 7 Data Points for Determining Slope, S_C or S_F

Slope at %	IBP	5	10	20	30	40	50	60	70	80	90	95	EP
T_L at %	0	0	0	10	20	30	40	50	60	70	80	90	95
T_U at %	5	10	20	30	40	50	60	70	80	90	90	95	V_{EP}
V_U- V_L	5	10	20	20	20	20	20	20	20	20	10	5	V_{EP}–95

11.5 To obtain the percent evaporated at a prescribed temperature reading, add the percent loss to each of the observed percent recovered at the prescribed temperature readings, and report these results as the respective percent evaporated, that is:

$$P_e = P_r + L \tag{9}$$

where:
L = observed loss,
P_e = percent evaporated, and
P_r = percent recovered.

11.6 To obtain temperature readings at prescribed percent evaporated, and if no recorded temperature data is available within 0.1 volume % of the prescribed percent evaporated, use either of the two following procedures, and indicate on the report whether the arithmetical procedure or the graphical procedure has been used.

11.6.1 *Arithmetical Procedure*—Deduct the observed loss from each prescribed percent evaporated to obtain the corresponding percent recovered. Calculate each required temperature reading as follows:

$$T = T_L + (T_H - T_L)(R - R_L)/(R_H - R_L) \tag{10}$$

where:
R = percent recovered corresponding to the prescribed percent evaporated,
R_H = percent recovered adjacent to, and higher than R,
R_L = percent recovered adjacent to, and lower than R,
T = temperature reading at the prescribed percent evaporated,
T_H = temperature reading recorded at R_H, and
T_L = temperature reading recorded at R_L.

Values obtained by the arithmetical procedure are affected by the extent to which the distillation graphs are nonlinear. Intervals between successive data points can, at any stage of the test, be no wider than the intervals indicated in 10.18. In no case shall a calculation be made that involves extrapolation.

11.6.2 *Graphical Procedure*—Using graph paper with uniform subdivisions, plot each temperature reading corrected for barometric pressure, if required (see 11.3), against its corresponding percent recovered. Plot the IBP at 0 % recovered. Draw a smooth curve connecting the points. For each prescribed percent evaporated, deduct the distillation loss to obtain the corresponding percent recovered and take from the graph the temperature reading that this percent recovered indicates. Values obtained by graphical interpolation procedures are affected by the care with which the plot is made.

NOTE 23—See Appendix X1 for numerical examples illustrating the arithmetical procedure.

11.6.3 In most automated instruments, temperature-volume data are collected at 0.1 volume % intervals or less and stored in memory. To report a temperature reading at a prescribed percent evaporated, neither of the procedures described in 11.6.1 and 11.6.2 have to be used. Obtain the desired temperature directly from the database as the temperature closest to and within 0.1 volume % of the prescribed percent evaporated.

12. Report

12.1 Report the following information (see Appendix X5 for examples of reports):

12.2 Report the barometric pressure to the nearest 0.1 kPa (1 mm Hg).

12.3 Report all volumetric readings in percentages.

12.3.1 *Manual Method*—Report volumetric readings to the nearest 0.5, and all temperature readings to the nearest 0.5°C (1.0°F).

12.3.2 *Automated Method*—Report volumetric readings to the nearest 0.1, and all temperature readings to the nearest 0.1°C (0.2°F) or less.

12.4 After barometric corrections of the temperature readings have been made, the following data require no further calculation prior to reporting: IBP, dry point, EP (FBP), decomposition point, and all pairs of corresponding values involving percent recovered and temperature readings.

12.4.1 The report shall state if the temperature readings have not been corrected for barometric pressure.

12.5 When the temperature readings have not been corrected to 101.3 kPa (760 mm Hg) pressure, report the percent residue and percent loss as *observed* in accordance with 10.19 and 11.1, respectively.

12.6 Do not use the corrected loss in the calculation of percent evaporated.

12.7 It is advisable to base the report on relationships between temperature readings and percent evaporated when the sample is a gasoline, or any other product classified under Group 1, or in which the percent loss is greater than 2.0. Otherwise, the report can be based on relationships between temperature readings and percent evaporated or percent recovered. Every report must indicate clearly which basis has been used.

12.7.1 In the manual method, if results are given in percent evaporated versus temperature readings, report if the arithmetical or the graphical procedure was used (see 11.6).

12.8 Report if a drying agent, as described in 7.5.2 or 7.5.3, was used.

12.9 Fig. X1.1 is an example of a tabular report. It shows the percent recovered versus the corresponding temperature reading and versus the corrected temperature reading. It also shows the percent loss, the corrected loss, and the percent evaporated versus the corrected temperature reading.

TABLE 8 Repeatability and Reproducibility for Group 1

Evaporated Point, %	Manual Repeatability[A]		Manual Reproducibility[A]		Automated Repeatability[A]		Automated Reproducibility[A]	
	°C	°F	°C	°F	°C	°F	°C	°F
IBP	3.3	6	5.6	10	3.9	7	7.2	13
5	$1.9+0.86S_C$	$3.4+0.86S_F$	$3.1+1.74S_C$	$5.6+1.74S_F$	$2.1+0.67S_C$	$3.8+0.67S_F$	$4.4+2.0S_C$	$7.9+2.0S_F$
10	$1.2+0.86S_C$	$2.2+0.86S_F$	$2.0+1.74S_C$	$3.6+1.74S_F$	$1.7+0.67S_C$	$3.0+0.67S_F$	$3.3+2.0S_C$	$6.0+2.0S_F$
20	$1.2+0.86S_C$	$2.2+0.86S_F$	$2.0+1.74S_C$	$3.6+1.74S_F$	$1.1+0.67S_C$	$2.0+0.67S_F$	$3.3+2.0S_C$	$6.0+2.0S_F$
30–70	$1.2+0.86S_C$	$2.2+0.86S_F$	$2.0+1.74S_C$	$3.6+1.74S_F$	$1.1+0.67S_C$	$2.0+0.67S_F$	$2.6+2.0S_C$	$4.7+2.0S_F$
80	$1.2+0.86S_C$	$2.2+0.86S_F$	$2.0+1.74S_C$	$3.6+1.74S_F$	$1.1+0.67S_C$	$2.0+0.67S_F$	$1.7+2.0S_C$	$3.0+2.0S_F$
90	$1.2+0.86S_C$	$2.2+0.86S_F$	$0.8+1.74S_C$	$1.4+1.74S_F$	$1.1+0.67S_C$	$2.0+0.67S_F$	$0.7+2.0S_C$	$1.2+2.0S_F$
95	$1.2+0.86S_C$	$2.2+0.86S_F$	$1.1+1.74S_C$	$1.9+1.74S_F$	$2.5+0.67S_C$	$4.5+0.67S_F$	$2.6+2.0S_C$	$4.7+2.0S_F$
FBP	3.9	7	7.2	13	4.4	8	8.9	16

[A] S_C or S_F is the average slope (or rate of change) calculated in accordance with 13.2.

13. Precision and Bias

13.1 *Precision*:

13.1.1 The precision of this test method has been determined by the statistical examination of interlaboratory test results obtained by 26 laboratories on 14 gasolines, by 4 laboratories on 8 samples of kerosine by the manual procedure, 3 laboratories on 6 samples of kerosine by the automated procedure, and 5 laboratories on 10 samples of diesel fuel by both the manual and automated procedures. Table A1.1 lists which tables and figures are to be used for the different fuel groups, distillation methods, and temperature scales.

13.1.2 The following terms are used in this section: (*1*) r = repeatability and (*2*) R = reproducibility. The value of any of these terms will depend upon whether the calculations were carried out in °C or °F.

13.2 *Slope or Rate of Change of Temperature*:

13.2.1 To determine the precision of a result, it is generally necessary to determine the slope or rate of change of the temperature at that particular point. This variable, denoted as S_C or S_F, is equal to the change in temperature, either in °C or in °F, respectively, per percent recovered or evaporated.

13.2.2 For Group 1 in the manual method and for all groups in the automated method, the precision of the IBP and EP does not require any slope calculation.

13.2.3 With the exception stated in 13.2.2 and in 13.2.4, the slope at any point during the distillation is calculated from the following equations, using the values shown in Table 7:

$$S_C \text{ (or } S_F) = (T_U - T_L) / (V_U - V_L) \quad (11)$$

where:

S_C = is the slope, °C/volume %,
S_F = is the slope, °F/volume %,
T_U = is the upper temperature, °C (or °F),
T_L = is the lower temperature, °C (or °F),
V_U = is the volume % recovered or evaporated corresponding to T_U,
V_L = is the volume % recovered or evaporated corresponding to T_L, and
V_{EP} = is the volume % recovered or evaporated corresponding to the end point.

13.2.4 In the event that the distillation end point occurs prior to the 95 % point, the slope at the end point is calculated as follows:

$$S_C \text{ (or } S_F) = (T_{EP} - T_{HR}) / (V_{EP} - V_{HR}) \quad (12)$$

where:

T_{EP} or T_{HR} is the temperature, in °C or °F at the percent volume recovered indicated by the subscript,

V_{EP} or V_{HR} is the volume % recovered.

13.2.4.1 The subscripts in Eq 12 refer to:

EP = end point
HR = highest reading, either 80 % of 90 %, prior to the end point.

13.2.5 For points between 10 to 85 % recovered which are not shown in Table 7, the slope is calculated as follows:

$$S_C \text{ (or } S_F) = 0.05\,(T_{(V+10)} - T_{(V-10)}) \quad (13)$$

13.2.6 For samples in Group 1, the precision data reported are based on slope values calculated from percent evaporated data.

13.2.7 For samples in Group 2, 3, and 4, the precision data reported (Table 8) are based on slope values calculated from percent recovered data.

13.2.8 When results are reported as volume % recovered, slope values for the calculation of precision are to be determined from percent recovered data; when results are reported as volume % evaporated slope values are to be determined from % evaporated data.

13.3 *Manual Method*:

13.3.1 *Repeatability*:

13.3.1.1 *GROUP 1*—The difference between successive 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 values calculated from Table 9 in only one case in twenty.

13.3.1.2 *GROUPS 2, 3, and 4*—The difference between successive 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 values calculated from the values in Table 9 in only one case in twenty.

13.3.2 *Reproducibility*:

TABLE 9 Repeatability and Reproducibility for Groups 2, 3 and 4 (Manual Method)

	Repeatability[A]		Reproducibility[A]	
	°C	°F	°C	°F
IBP	$1.0+0.35S_C$	$1.9+0.35S_F$	$2.8+0.93S_C$	$5.0+0.93S_F$
5—95 %	$1.0+0.41S_C$	$1.8+0.41S_F$	$1.8+1.33S_C$	$3.3+1.33S_F$
FBP	$0.7+0.36S_C$	$1.3+0.36S_F$	$3.1+0.42S_C$	$5.7+0.42S_F$
% volume at temperature reading	$0.7+0.92/S_C$	$0.7+1.66/S_F$	$1.5+1.76/S_C$	$1.53+3.20/S_F$

[A] Calculate S_C or S_F from 13.2.

13.3.2.1 *GROUP 1*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the normal and correct operation of this method, exceed the values calculated from Table 9 in only one case in twenty.[7]

13.3.2.2 *GROUPS 2, 3, and 4*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the normal and correct operation of this test method, exceed the values calculated from the data in Table 9 in only one case in twenty.[8]

13.4 *Automated Method*:

13.4.1 *Repeatability*:

13.4.1.1 *GROUP 1*—The difference between successive 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 values calculated from Table 8 in only one case in twenty.

13.4.1.2 *GROUPS 2, 3, and 4*—The difference between successive 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 values calculated from Table 10 in only one case in twenty.

13.4.2 *Reproducibility*:

13.4.2.1 *GROUP 1*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the normal and correct operation of this test method, exceed the values calculated from Table 8 in only one case in twenty.[7]

13.4.2.2 *GROUPS 2, 3, and 4*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the normal and correct operation of this test method, exceed the values calculated from Table 10 in only one case in twenty.

13.5 *Bias*:

13.5.1 *Bias*—Due to the use of total immersion thermometers, or temperature sensing systems designed to emulate them, the distillation temperatures in this test method are somewhat lower than the true temperatures. The amount of bias depends on the product being distilled and the thermometer used.

13.5.2 *Relative Bias*—There exists a bias between the empirical results of distillation properties obtained by this test method and the true boiling point distillation curve obtained by Test Method D 2892. The magnitude of this bias, and how it relates to test precision, has not been rigorously studied.

13.5.3 *Relative Bias*—An interlaboratory study[5] conducted in 2003 using manual and automated apparatus has concluded that there is no statistical evidence to suggest that there is a bias between manual and automated results.

14. Keywords

14.1 batch distillation; distillates; distillation; laboratory distillation; petroleum products

[7] Precision data obtained from RR study on both manual and automated D 86 units by North American and IP Laboratories.

[8] Table 9 has been derived from the nomographs in Figs. 6 and 7 in ASTM D 86–97.

TABLE 10 Repeatability and Reproducibility for Groups 2, 3 and 4 (Automated)

Collected, %	Repeatability[A]		Reproducibility[A]	
	°C	°F	°C	°F
IBP	3.5	6.3	8.5	15.3
2 %	3.5	6.3	$2.6 + 1.92S_C$	$4.7 + 1.92S_F$
5 %	$1.1 + 1.08S_C$	$2.0 + 1.08S_F$	$2.0 + 2.53S_C$	$3.6 + 2.53S_F$
10 %	$1.2 + 1.42S_C$	$2.2 + 1.42S_F$	$3.0 + 2.64S_C$	$5.4 + 2.64S_F$
20–70 %	$1.2 + 1.42S_C$	$2.2 + 1.42S_F$	$2.9 + 3.97S_C$	$5.2 + 3.97S_F$
80 %	$1.2 + 1.42S_C$	$2.2 + 1.42S_F$	$3.0 + 2.64S_C$	$5.4 + 2.64S_F$
90–95 %	$1.1 + 1.08S_C$	$2.0 + 1.08S_F$	$2.0 + 2.53S_C$	$3.6 + 2.53S_F$
FBP	3.5	6.3	10.5	18.9

[A] S_C or S_F is the average slope (or rate of change) calculated in accordance with 13.5.

ANNEXES

(Mandatory Information)

A1. REPEATABILITY AND REPRODUCIBILITY DEFINITION AIDS

A1.1 Table A1.1 is an aid for determining which repeatability and reproducibility table or section, is to be used.

TABLE A1.1 Summary of Aids for Definition of Repeatability and Reproducibility

Group	Method	Temperature Scale	Table or Section to Use	
			Repeatability	Reproducibility
1	Manual	°C	Table 8	Table 8
		°F	Table 8	Table 8
1	Automated	°C	Table 8	Table 8
		°F	Table 8	Table 8
2,3,4	Manual	°C	Table 9	Table 9
		°F	Table 9	Table 9
2,3,4	Automated	°C	Table 10	Table 10
		°F	Table 10	Table 10

A2. DETAILED DESCRIPTION OF APPARATUS

A2.1 *Distillation Flasks*—Flasks shall be of heat resistant glass, constructed to the dimensions and tolerances shown in Fig. A2.1 and shall otherwise comply with the requirements of Specification E 1405. Flask A (100 mL) may also be constructed with a ground glass joint, in which case the diameter of the neck shall be the same as the 125-mL flask.

NOTE A2.1—For tests specifying dry point, specially selected flasks with bottoms and walls of uniform thickness are desirable.

A2.2 *Condenser and Condenser Bath*—Typical types of condenser and condenser baths are illustrated in Figs. 1 and 2.

A2.2.1 The condenser shall be made of seamless noncorrosive metal tubing, 560 ± 5 mm in length, with an outside diameter of 14 mm and a wall thickness of 0.8 to 0.9 mm.

NOTE A2.2—Brass or stainless steel has been found to be a suitable material for this purpose.

A2.2.2 The condenser shall be set so that 393 ± 3 mm of the tube is in contact with the cooling medium, with 50 ± 3 mm outside the cooling bath at the upper end, and with 114 ± 3 mm outside at the lower end. The portion of the tube projecting at the upper end shall be set at an angle of 75 ± 3° with the vertical. The portion of the tube inside the condenser bath shall be either straight or bent in any suitable continuous smooth curve. The average gradient shall be 15 ± 1° with respect to the horizontal, with no 10-cm section having a gradient outside of the 15 ± 3° range. The projecting lower portion of the condenser tube shall be curved downward for a length of 76 mm and the lower end shall be cut off at an acute angle. Provisions shall be made to enable the flow of the distillate to run down the side of the receiving cylinder. This can be accomplished by using a drip-deflector, which is attached to the outlet of the tube. Alternatively, the lower portion of the condenser tube can be curved slightly backward to ensure

contact with the wall of the receiving cylinder at a point 25 to 32 mm below the top of the receiving cylinder. Fig. A2.3 is a drawing of an acceptable configuration of the lower end of the condenser tube.

A2.2.3 The volume and the design of the bath will depend on the cooling medium employed. The cooling capacity of the bath shall be adequate to maintain the required temperature for the desired condenser performance. A single condenser bath may be used for several condenser tubes.

A2.3 *Metal Shield or Enclosure for Flask.* (Manual units only).

A2.3.1 *Shield for Gas Burner (see* Fig. 1*)*—The purpose of this shield is to provide protection for the operator and yet allow easy access to the burner and to the distillation flask during operation. A typical shield would be 480-mm high, 280-mm long and 200-mm wide, made of sheet metal of 0.8-mm thickness (22 gauge). The shield shall be provided with at least one window to observe the dry point at the end of the distillation.

A2.3.2 *Shield for Electric Heater (see* Fig. 2*)*—A typical shield would be 440-mm high, 200-mm long, and 200-mm wide, made of sheet metal of approximately 0.8-mm thickness (22 gauge) and with a window in the front side. The shield shall be provided with at least one window to observe the dry point at the end of the distillation.

A2.4 *Heat Source:*

A2.4.1 *Gas Burner* (see Fig. 1), capable of bringing over the first drop from a cold start within the time specified and of continuing the distillation at the specified rate. A sensitive manual control valve and gas pressure regulator to give complete control of heating shall be provided.

A2.4.2 *Electric Heater* (see Fig. 2), of low heat retention.

NOTE A2.3—Heaters, adjustable from 0 to 1000 W, have been found to be suitable for this purpose.

A2.5 *Flask Support:*

A2.5.1 *Type 1*—Use a Type 1 flask support with a gas burner (see Fig. 1). This support consists of either a ring support of the ordinary laboratory type, 100 mm or larger in diameter, supported on a stand inside the shield, or a platform adjustable from the outside of the shield. On this ring or platform is mounted a hard board made of ceramic or other heat-resistant material, 3 to 6 mm in thickness, with a central opening 76 to 100 mm in diameter, and outside line dimensions slightly smaller than the inside boundaries of the shield.

A2.5.2 *Type 2*—Use a Type 2 flask support assembly with electric heating (see Fig. 2 as one example). The assembly consists of an adjustable system onto which the electric heater is mounted with provision for placement of a flask support board (see A2.6) above the electric heater. The whole assembly is adjustable from the outside of the shield.

A2.6 *Flask Support Board*—The flask support board shall be constructed of ceramic or other heat-resistant material, 3 to 6 mm in thickness. Flask support boards are classified as A, B, or C, based on the size of the centrally located opening, the dimension of which is shown in Table 1. The flask support board shall be of sufficient dimension to ensure that thermal heat to the flask only comes from the central opening and that extraneous heat to the flask other than through the central opening is minimized. (**Warning**—Asbestos-containing materials shall not be used in the construction of the flask support board.)

A2.7 The flask support board can be moved slightly in different directions on the horizontal plane to position the distillation flask so that direct heat is applied to the flask only through the opening in this board. Usually, the position of the flask is set by adjusting the length of the side-arm inserted into the condenser.

A2.8 Provision shall be made for moving the flask support assembly vertically so that the flask support board is in direct contact with the bottom of the distillation flask during the distillation. The assembly is moved down to allow for easy mounting and removal of the distillation flask from the unit.

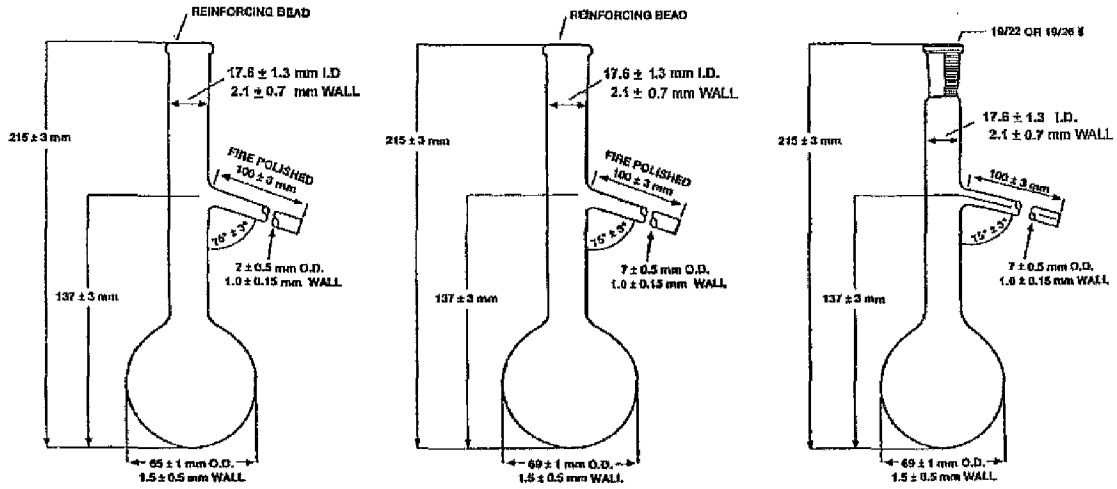
A2.9 *Receiving Cylinders* -The receiving cylinder shall have a capacity to measure and collect 100 mL. The shape of the base shall be such that the receiver does not topple when placed empty on a surface inclined at an angle of 13° from the horizontal.

A2.9.1 *Manual Method*—The cylinder shall be graduated at intervals of 1 mL and have a graduation at the 100-mL mark. Construction details and tolerances for the graduated cylinder are shown in Fig. A2.4.

A2.9.2 *Automated Method*—The cylinder shall conform to the physical specifications described in Fig. A2.4, except that graduations below the 100-mL mark are permitted, as long as they do not interfere with the operation of the level follower. Receiving cylinders for use in automated units may also have a metal base.

A2.9.3 If required, the receiving cylinder shall be immersed during the distillation to above the 100-mL graduation line in a cooling liquid contained in a cooling bath, such as a tall-form beaker of clear glass or transparent plastic. Alternatively, the receiving cylinder may be placed in a thermostated bath air circulation chamber.

A2.10 *Residue Cylinder*—The graduated cylinder shall have a capacity of 5 or 10 mL, with graduations into 0.1 mL subdivisions, beginning at 0.1 mL. The top of the cylinder may be flared, the other properties shall conform to Specification E 1272.



Flask A, 100 mL Flask B, 125 mL Flask B, 125 mL

FIG. A2.1 Flask A, 100 mL, Flask B, 125 mL, and Flask B with Ground Glass Joint, 125 mL

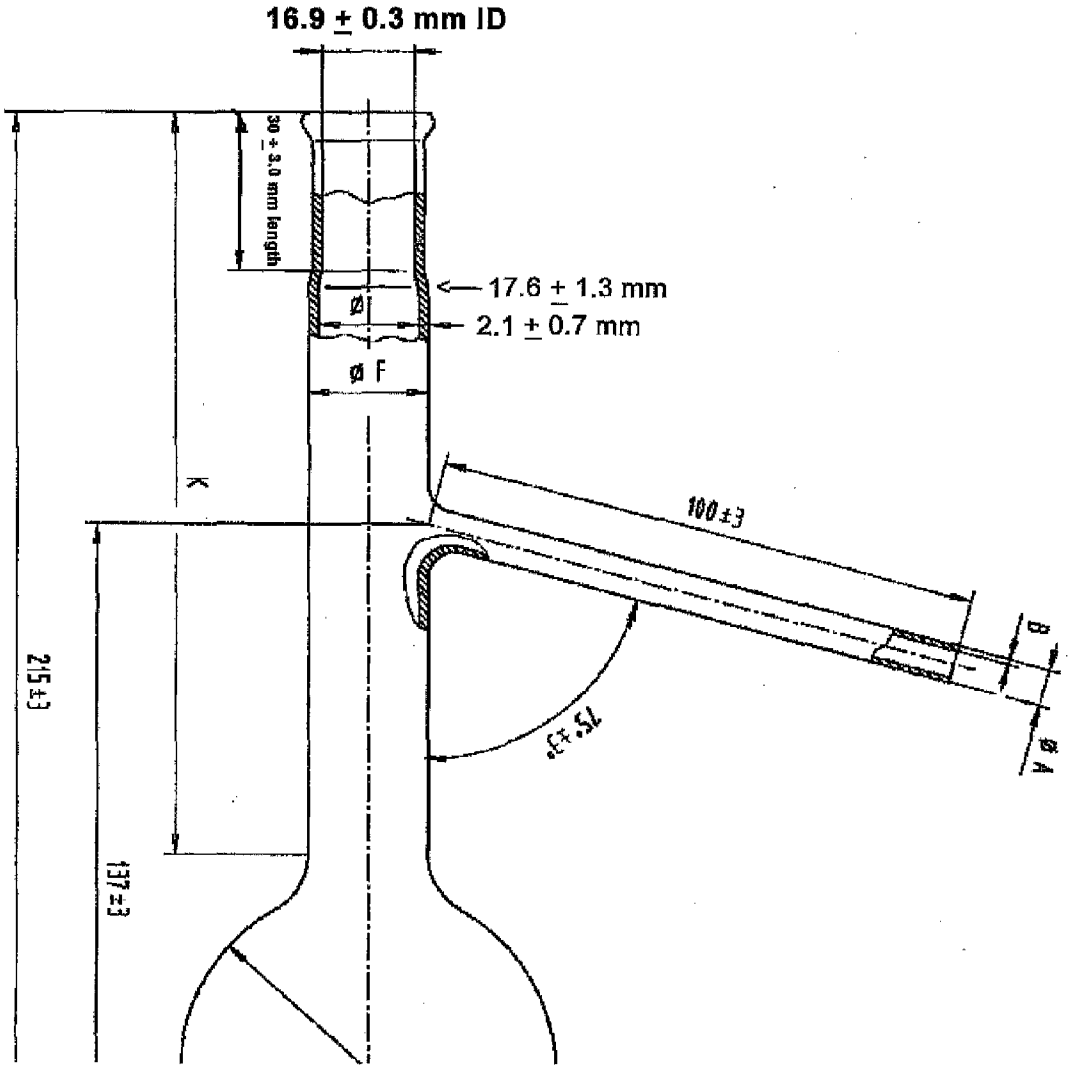
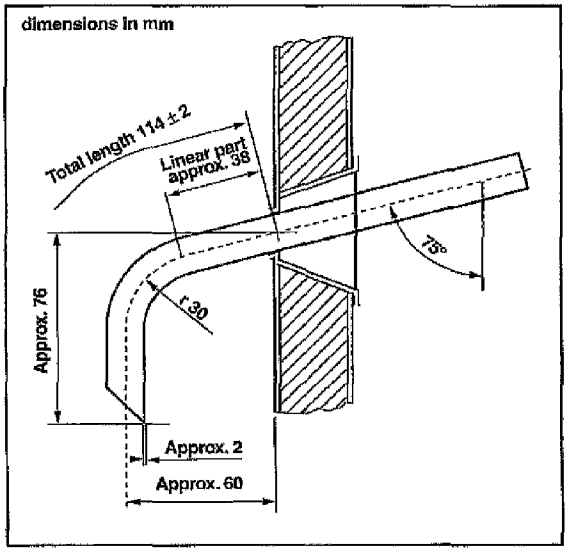
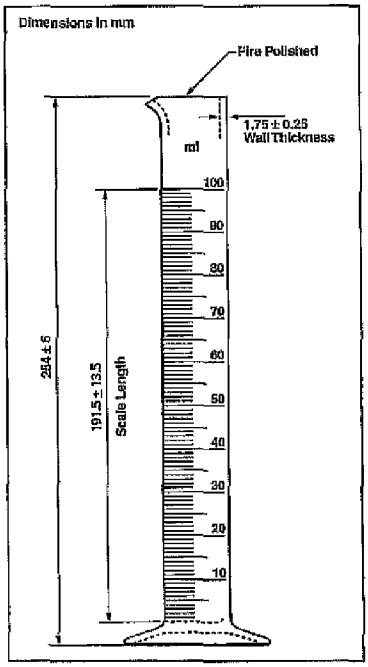


FIG. A2.2 Detail of Upper Neck Section



Lower End of Condenser Tube

FIG. A2.3 Lower End of Condenser Tube



NOTE—1 to 100 mL in 1 mL graduations; tolerance ± 1.0 mL.

FIG. A2.4 100 mL Graduated Cylinder

A3. DETERMINATION OF THE DIFFERENCE IN LAG TIME BETWEEN AN ELECTRONIC TEMPERATURE MEASUREMENT SYSTEM AND A MERCURY-IN-GLASS THERMOMETER

A3.1 The response time of an electronic temperature measuring device is inherently more rapid than that of a mercury-in-glass thermometer. The temperature measuring device assembly in general use, consisting of the sensor and its casing, or an electronic system and its associated software, or both, is so designed that the temperature measuring system will simulate the temperature lag of the mercury-in-glass thermometer.

A3.2 To determine the difference in lag time between such a temperature measuring system and a mercury-in-glass thermometer, analyze a sample such as gasoline, kerosine, jet fuel, or light diesel fuel with the electronic temperature measurement system in place and in accordance with the procedures described in this test method. In most cases this is the standard distillation step performed with an automated unit.

A3.2.1 Do not use a single pure compound, a very narrow boiling range product, or a synthetic blend of less than six compounds for this test.

A3.2.2 Best results are obtained with a sample that is typical of the sample load of the laboratory. Alternatively, use a full-range mixture with a 5 to 95 % boiling range of at least 100°C.

A3.3 Replace the electronic temperature measuring device with a low range or a high range mercury-in-glass thermometer, depending on the boiling range of the sample.

A3.4 Repeat the distillation with this thermometer, and manually record the temperature at the various percent recovered as described in 10.14.

A3.5 Calculate the values for the repeatability for the observed slope ($\Delta T/\Delta V$) for the different readings in the test.

A3.6 Compare the test data obtained using these two temperature measuring devices. The difference at any point shall be equal to, or less than, the repeatability of the method at that point. If this difference is larger, replace the electronic temperature measuring device or adjust the electronics involved, or both.

A4. PROCEDURE TO DETERMINE THE PERCENT EVAPORATED OR PERCENT RECOVERED AT A PRESCRIBED TEMPERATURE READING

A4.1 Many specifications require specific percentages evaporated or recovered at prescribed temperature readings, either as maxima, minima, or ranges. The procedures to determine these values are frequently designated by the terms Exxx or Rxxx, where xxx is the desired temperature.

NOTE A4.1—Regulatory standards on the certification of reformulated gasoline under the complex model procedure require the determination of E 200 and E 300, defined as the percent evaporated fuel at 93.3°C (200°F) and 148.9°C (300°F), respectively. E 158, the percent evaporated at a distillation temperature of 70°C (158°F), is also used in describing fuel volatility characteristics. Other typical temperatures are R 200 for kerosines and R 250 and R 350 for gas oils, where R 200, R 250, and R 350 are the percent recovered fuel at 200°C, 250°C, and 350°C, respectively.

A4.2 Determine the barometric pressure, and calculate the correction to the desired temperature reading using Eq 3, Eq 4, or Eq 5 for t = xxx°C (or t_f = xxx°F).

A4.2.1 *Manual Method*—Determine this correction to 0.5°C (1°F).

A4.2.2 *Automated Method*—Determine this correction to 0.1°C (0.2°F).

A4.3 Determine the expected temperature reading to yield xxx°C (or xxx°F) after the barometric correction. To obtain the expected value, add the absolute value of the calculated correction to the desired temperature if the barometric pressure is above 101.3 kPa. If the barometric pressure is below 101.3 kPa, subtract the absolute value of the calculated correction from the desired temperature.

A4.4 Perform the distillation, as described in Section 10, while taking into account A4.5 and A4.6.

A4.5 *Manual Distillation:*

A4.5.1 In the region between about 10°C below and 10°C above the desired expected temperature reading determined in A4.3 record the temperature reading in intervals of 1 volume %.

A4.5.2 If the intent of the distillation is to solely determine the value of Exxx or Rxxx, discontinue the distillation after at least another 2 mL of distillate have been collected. Otherwise, continue the distillation, as described in Section 10, and determine the observed loss, as described in 11.1.

A4.5.2.1 If the intent of the distillation is to determine the value of Exxx and the distillation was terminated after about 2 mL of distillate was collected beyond the desired temperature, allow the distillate to drain into the receiving graduate. Allow the contents of the flask to cool to below approximately 40°C and then drain its contents into the receiving graduate. Note the volume of product in the receiving graduate to the nearest 0.5 mL at 2 min intervals until two successive observations agree.

A4.5.2.2 The amount recovered in the receiving graduate is the percent recovery. Determine the amount of observed loss by subtracting the percent recovery from 100.0.

A4.6 *Automated Distillation:*

A4.6.1 In the region between about 10°C below and 10°C above the desired expected temperature reading determined in A4.3, collect temperature-volume data at 0.1 volume % intervals or less.

A4.6.2 Continue the distillation, as described in Section 10, and determine the percent loss, as described in 11.1.

A4.7 *Calculations:*

A4.7.1 *Manual Method*—If a volume % recovered reading is not available at the exact temperature calculated in A4.3, determine the percent recovered by interpolation between the two adjacent readings. Either the linear, as described in 11.6.1, or the graphical procedure, as described in 11.6.2, is permitted. The percent recovered is equal to Rxxx.

A4.7.2 *Automated Method*—Report the observed volume to 0.1 volume % corresponding to the temperature closest to the expected temperature reading. This is the percent recovered, or Rxxx.

A4.7.3 *Manual and Automated Methods*—To determine the value of Exxx, add the observed loss to the percent recovered, Rxxx, as determined in A4.7.1 or A4.7.2 and as described in Eq 9.

A4.7.3.1 As prescribed in 12.6, do not use the corrected loss.

A4.8 *Precision:*

A4.8.1 The statistical determination of the precision of the volume % evaporated or recovered at a prescribed temperature has not been directly measured in an interlaboratory program. It can be shown that the precision of the volume % evaporated or recovered at a prescribed temperature is equivalent to the precision of the temperature measurement at that point divided by the rate of change of temperature versus volume % evaporated or recovered. The estimation of precision becomes less precise at high slope values.

A4.8.2 Calculate the slope or rate of change in temperature reading, S_C(or S_F), as described in 13.2 and Eq 11 and using temperature values bracketing the desired temperature.

A4.8.3 Calculate the repeatability, r, or the reproducibility, R, from the slope, S_C (or S_F), and the data in Table 8, Table 9, or Table 10.

A4.8.4 Determine the repeatability or reproducibility, or both, of the volume % evaporated or recovered at a prescribed temperature from the following formulas:

$$^{r}\text{volume \%} = r/S_C\,(S_F) \quad \text{(A4.1)}$$

$$^{R}\text{volume \%} = R/S_C\,(S_F) \quad \text{(A4.2)}$$

where:

^{r}volume % = repeatability of the volume % evaporated or recovered,

^{R}volume % = reproducibility of the volume % evaporated or recovered,

r = repeatability of the temperature at the prescribed temperature at the observed percent distilled,

R = reproducibility of the temperature at the prescribed temperature at the observed percent distilled, and

$S_C(S_F)$ = rate of change in temperature reading in °C (°F) per the volume % evaporated or recovered.

A4.8.5 Examples on how to calculate the repeatability and the reproducibility are shown in Appendix X2.

APPENDIXES

(Nonmandatory Information)

X1. EXAMPLES ILLUSTRATING CALCULATIONS FOR REPORTING OF DATA

X1.1 The observed distillation data used for the calculation of the examples below are shown in the first three columns of Fig. X1.1.

X1.1.1 Temperature readings corrected to 101.3 kPa (760 mm Hg) pressure (see 11.3) are as follows:

$$\text{correction } (°C) = 0.0009\,(101.3 - 98.6)\,(273 + t_c) \quad \text{(X1.1)}$$

$$\text{correction } (°F) = 0.00012\,(760 - 740)\,(460 + t_f) \quad \text{(X1.2)}$$

X1.1.2 Loss correction to 101.3 kPa (see 11.4) are as follows. The data for the examples are taken from Fig. X1.1.

$$\text{corrected loss} = (0.5 + (4.7 - 0.5)/ \quad \text{(X1.3)}$$

$$\{1 + (101.3 - 98.6)/8.0\} = 3.6$$

X1.1.3 Recovery correction to 101.3 kPa (see 11.4.1) are as follows:

$$\text{corrected recovery} = 94.2 + (4.7 - 3.6) = 95.3 \quad \text{(X1.4)}$$

X1.2 *Temperature Readings at Prescribed Percent Evaporated:*

X1.2.1 Temperature reading at 10 % evaporated (4.7 % observed loss = 5.3 % recovered) (see 11.6.1) are as follows:

$$T_{10E}\,(°C) = 33.7 + [(40.3 - 33.7) \quad \text{(X1.5)}$$

$$(5.3 - 5)/(10 - 5)] = 34.1°C$$

$$T_{10E}\,(°F) = 92.7 + [(104.5 - 92.7) \quad \text{(X1.6)}$$

$$(5.3 - 5)/(10 - 5)] = 93.1°F$$

X1.2.2 Temperature reading at 50 % evaporated (45.3 % recovered) (see 11.6.1) are as follows:

$$T_{50E}\,(°C) = 93.9 + [(108.9 - 93.9) \quad \text{(X1.7)}$$

$$(45.3 - 40)/(50 - 40)] = 101.9°C$$

$$T_{50E}\,(°F) = 201 + [(228 - 201) \quad \text{(X1.8)}$$

$$(45.3 - 40)/(50 - 40)] = 215.3°F$$

X1.2.3 Temperature reading at 90 % evaporated (85.3 % recovered) (see 11.6.1) are as follows:

$$T_{90E}\,(°C) = 181.6 + [(201.6 - 181.6) \quad \text{(X1.9)}$$

$$(85.3 - 85)/(90 - 85)\,] = 182.8°C$$

$$T_{90F}(°F) = 358.9 + [(394.8 - 358.9)$$ (X1.10)
$$(85.3 - 85)/(90 - 85)] = 361.0°F$$

X1.2.4 Temperature reading at 90 % evaporated (85.3 % recovered) not corrected to 101.3 kPa pressure (see 11.6.1) are as follows:

$$T_{90S}(°C) = 180.5 + [(200.4 - 180.5)$$ (X1.11)
$$(85.3 - 85)/(90 - 85)] = 181.7°C$$
$$T_{90E}(°F) = 357 + [(392 - 357)$$ (X1.12)
$$(85.3 - 85)/(90 - 85)] = 359.1°F$$

NOTE X1.1—Results calculated from °C data may not correspond exactly to results calculated from °F data because of errors in rounding.

Sample ID:
Date analyzed:
Equipment No:
Remarks:

Barometric pressure: 98.6 kPa
Analyst:

	Barometric pressure observed 98.6 kPa 740 mm Hg		corrected 101.3 kPa 760 mm Hg		procedure arithmetical/graphical		
% recovered	°C	°F	°C	°F	% evaporated	T_{evap} °C	°F
IBP	25.5	78	26.2	79.2	5	26.7	80.0
5	33.0	91	33.7	92.7	10	34.1	93.4
10	39.5	103	40.3	104.5	15	40.7	105.2
15	44.0	115	46.8	116.2	20	47.3	117.1
20	54.5	130	55.3	131.5	30	65.7	150.2
30	74.0	165	74.8	166.7	40	84.9	184.9
40	93.0	199	93.9	201.0	50	101.9	215.3
50	108.0	226	108.9	228.0	60	116.9	242.4
60	123.0	253	124.0	255.1	70	134.1	273.3
70	142.0	288	143.0	289.4	80	156.0	312.8
80	166.5	332	167.6	333.6	85	168.4	335.1
85	180.5	357	181.6	358.9	90	182.8	361.0
90	200.4	393	201.6	394.8	95	202.4	396.3
EP	215.0	419	216.2	421.1			
recovered, %	94.2		95.3				
residue, %	1.1		1.1				
loss, %	4.7		3.6				

FIG. X1.1 Example of Test Report

X2. EXAMPLES OF CALCULATION OF REPEATABILITY AND REPRODUCIBILITY OF VOLUME % (RECOVERED OR EVAPORATED) AT A PRESCRIBED TEMPERATURE READING

X2.1 Some specifications require the reporting of the volume % evaporated or recovered at a prescribed temperature. Table X2.1 shows the distillation data of a Group 1 sample as obtained by an automated unit.

X2.2 *Example Calculation:*

X2.2.1 For a Group 1 sample exhibiting distillation characteristics as per Table X2.1, as determined by an automated unit, the reproducibility of the volume evaporated,^{R}volume %, at 93.3°C (200°F) is determined as follows:

X2.2.1.1 Determine first the slope at the desired temperature:

$$S_C\,\% = 0.1\,(T_{(20)} - T_{(10)}) \quad \text{(X2.1)}$$
$$= 0.1\,(94 - 83)$$
$$= 1.1$$
$$S_F\,\% = 0.1\,(T_{(20)} - T_{(10)})$$
$$= 0.1\,(201 - 182)$$
$$= 1.9$$

X2.2.2 From Table 9, determine the value of R, the reproducibility at the observed percentage distilled. In this case, the observed percentage distilled is 18 % and

$$R = 3.3 + 2.0\,(S_C) \quad \text{(X2.2)}$$
$$= 3.3 + 2.0 \times 1.1$$
$$= 5.5$$
$$R = 6.0 + 2.0\,(S_F)$$
$$= 6.0 + 2.0 \times 1.9$$
$$= 9.8$$

X2.2.3 From the calculated value of R, determine the value of volume, as described in A4.8.4.

$$R \text{ volume } \% = R/(S_C) \quad \text{(X2.3)}$$
$$= 5.5/1.1$$
$$= 5.0$$
$$R \text{ volume } \% = R/(S_F)$$
$$= 9.8/1.9$$
$$= 5.1$$

TABLE X2.1 Distillation Data from a Group 1 Sample Automated Distillation

Distillation Point Recovered, mL	Temperature° C	Temperature °F	Volume (mL) Recovered at 93.3°C (200°F)
			18.0
10	84	183	
20	94	202	
30	103	217	
40	112	233	
Distillation Point Evaporated, mL	**Temperature° C**	**Temperature° F**	**Volume (mL0 Evaporated at 93.3°C (200°F)**
			18.4
10	83	182	
20	94	201	
30	103	217	
40	111	232	

X3. TABLES OF CORRECTED LOSS FROM MEASURED LOSS AND BAROMETRIC PRESSURE

X3.1 The table presented as Fig. X3.1 can be used to determine the corrected loss from the measured loss and the barometric pressure in kPa.

X3.2 The table presented as Fig. X3.2 can be used to determine the corrected loss from the measured loss and the barometric pressure in mm Hg.

Barometric Pressure, kPa

		from	76.1	80.9	84.5	87.8	89.6	91.5	93.1	94.1	95.5	96.4	97.2	97.9	98.4	98.9	99.5	100.0	100.4	100.8	101.2	101.5	102.0	102.4	102.8	103.2
		through	80.8	84.4	87.2	89.5	91.4	93.0	94.0	95.4	96.3	97.1	97.6	98.3	98.8	99.4	99.9	100.3	100.7	101.1	101.4	101.9	102.3	102.7	103.1	103.5
Observed Loss	/— Corrected Loss ————>																									
Units																										
	0		0.37	0.35	0.33	0.31	0.29	0.27	0.25	0.23	0.20	0.18	0.16	0.14	0.13	0.11	0.09	0.08	0.04	0.02	−0.00	−0.02	−0.06	−0.09	−0.13	−0.17
	1		0.68	0.65	0.67	0.69	0.71	0.73	0.75	0.78	0.80	0.82	0.84	0.86	0.87	0.89	0.92	0.94	0.96	0.98	1.00	1.03	1.06	1.09	1.13	1.17
	2		0.99	0.95	1.01	1.08	1.14	1.20	1.26	1.33	1.40	1.46	1.52	1.57	1.62	1.68	1.75	1.81	1.87	1.94	2.00	2.08	2.17	2.27	2.38	2.51
	3		1.15	1.25	1.36	1.46	1.57	1.67	1.77	1.88	1.99	2.09	2.19	2.28	2.37	2.47	2.58	2.69	2.79	2.90	3.00	3.13	3.29	3.45	3.63	3.84
	4		1.41	1.56	1.70	1.84	1.99	2.14	2.28	2.43	2.59	2.73	2.87	3.00	3.12	3.26	3.41	3.56	3.70	3.85	4.00	4.18	4.40	4.63	4.89	5.18
	5		1.68	1.86	2.04	2.23	2.42	2.61	2.79	2.98	3.19	3.37	3.55	3.71	3.87	4.05	4.25	4.44	4.62	4.81	5.00	5.23	5.51	5.81	6.14	6.52
	6		1.94	2.16	2.39	2.61	2.84	3.06	3.30	3.53	3.78	4.01	4.23	4.42	4.62	4.84	5.08	5.31	5.53	5.77	6.00	6.28	6.63	6.99	7.40	7.86
	7		2.20	2.46	2.73	3.00	3.27	3.55	3.80	4.08	4.36	4.65	4.90	5.14	5.37	5.63	5.91	6.18	6.44	6.73	7.00	7.33	7.74	8.17	8.65	9.20
	8		2.46	2.76	3.07	3.38	3.70	4.02	4.31	4.63	4.96	5.29	5.58	5.85	6.12	6.41	6.74	7.08	7.36	7.69	8.00	8.38	8.86	9.35	9.90	10.53
	9		2.72	3.07	3.41	3.76	4.12	4.49	4.82	5.18	5.57	5.92	6.26	6.56	6.87	7.20	7.57	7.93	8.27	8.65	9.00	9.43	9.97	10.53	11.16	11.87
	10		2.98	3.37	3.76	4.15	4.55	4.95	5.33	5.73	6.17	6.56	6.94	7.28	7.62	7.99	8.41	8.81	9.19	9.60	10.00	10.48	11.06	11.71	12.41	13.21
	11		3.24	3.67	4.10	4.53	4.97	5.43	5.84	6.28	6.77	7.20	7.61	7.99	8.37	8.78	9.24	9.68	10.10	10.56	11.00	11.53	12.20	12.89	13.67	14.55
	12		3.50	3.97	4.44	4.92	5.40	5.90	6.35	6.83	7.36	7.84	8.29	8.71	9.12	9.57	10.07	10.58	11.02	11.52	12.00	12.59	13.31	14.07	14.92	15.89
	13		3.76	4.27	4.78	5.30	5.83	6.36	6.86	7.39	7.96	8.47	8.97	9.42	9.86	10.36	10.90	11.43	11.93	12.48	13.00	13.64	14.43	15.25	16.17	17.22
	14		4.03	4.58	5.13	5.69	6.25	6.83	7.38	7.94	8.56	9.11	9.64	10.13	10.61	11.15	11.74	12.31	12.85	13.44	14.00	14.69	15.54	16.43	17.43	18.56
	15		4.29	4.88	5.47	6.07	6.68	7.30	7.87	8.49	9.15	9.75	10.32	10.85	11.36	11.93	12.57	13.18	13.76	14.40	15.00	15.74	16.66	17.61	18.68	19.90
	16		4.55	5.18	5.81	6.45	7.10	7.77	8.38	9.04	9.75	10.39	11.00	11.56	12.11	12.72	13.40	14.06	14.68	15.36	16.00	16.79	17.77	18.79	19.94	21.24
	17		4.81	5.48	6.16	6.84	7.53	8.24	8.89	9.59	10.35	11.03	11.68	12.27	12.86	13.51	14.23	14.93	15.59	16.31	17.00	17.84	18.86	19.97	21.19	22.58
	18		5.07	5.79	6.50	7.22	7.96	8.71	9.40	10.14	10.94	11.66	12.35	12.99	13.61	14.30	15.07	15.80	16.50	17.27	18.00	18.89	20.00	21.15	22.44	23.91
	19		5.33	6.08	6.84	7.61	8.38	9.18	9.91	10.69	11.54	12.30	13.03	13.70	14.36	15.09	15.90	16.68	17.42	18.23	19.00	19.94	21.11	22.33	23.70	25.25
	20		5.59	6.39	7.18	7.99	8.81	9.65	10.41	11.24	12.14	12.94	13.71	14.41	15.11	15.88	16.73	17.55	18.33	19.18	20.00	20.99	22.23	23.51	24.95	26.59
Tenths																										
	0.0		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	0.1		0.03	0.03	0.03	0.03	0.04	0.04	0.05	0.05	0.06	0.06	0.06	0.07	0.07	0.07	0.08	0.08	0.09	0.09	0.10	0.10	0.11	0.11	0.12	0.13
	0.2		0.05	0.06	0.07	0.08	0.09	0.09	0.10	0.11	0.12	0.13	0.14	0.14	0.15	0.16	0.17	0.17	0.18	0.19	0.20	0.21	0.22	0.24	0.25	0.27
	0.3		0.08	0.09	0.10	0.12	0.13	0.14	0.15	0.17	0.18	0.19	0.20	0.21	0.22	0.24	0.25	0.26	0.27	0.28	0.30	0.32	0.33	0.35	0.38	0.40
	0.4		0.10	0.12	0.14	0.15	0.17	0.19	0.20	0.22	0.24	0.26	0.27	0.28	0.30	0.32	0.33	0.35	0.37	0.38	0.40	0.42	0.45	0.47	0.50	0.54
	0.5		0.13	0.15	0.17	0.19	0.21	0.23	0.25	0.28	0.30	0.32	0.34	0.35	0.37	0.39	0.42	0.44	0.46	0.48	0.50	0.53	0.56	0.59	0.63	0.67
	0.6		0.16	0.18	0.21	0.23	0.26	0.28	0.31	0.33	0.36	0.38	0.41	0.43	0.45	0.47	0.50	0.52	0.55	0.58	0.60	0.63	0.67	0.71	0.75	0.80
	0.7		0.18	0.21	0.24	0.27	0.30	0.33	0.36	0.39	0.42	0.45	0.47	0.50	0.52	0.55	0.58	0.61	0.64	0.67	0.70	0.74	0.78	0.83	0.88	0.94
	0.8		0.21	0.24	0.27	0.31	0.34	0.38	0.41	0.44	0.48	0.51	0.54	0.57	0.60	0.63	0.67	0.70	0.73	0.77	0.80	0.84	0.89	0.94	1.00	1.07
	0.9		0.24	0.27	0.31	0.35	0.38	0.42	0.46	0.50	0.54	0.57	0.61	0.64	0.67	0.71	0.75	0.79	0.82	0.86	0.90	0.95	1.00	1.06	1.13	1.20

FIG. X3.1 Corrected Loss from Observed Loss and Barometric Pressure kPa

Barometric Pressure, mm Hg.

		from	571	607	634	655	672	686	698	706	716	726	729	734	738	742	745	750	753	756	759	762	765	768	771	774
		through	606	633	654	671	685	697	705	715	722	728	733	737	741	745	749	752	755	758	761	764	767	770	773	776
Observed Loss	/— Corrected Loss ————>																									
Units																										
	0		0.37	0.35	0.33	0.31	0.29	0.27	0.25	0.23	0.20	0.18	0.16	0.14	0.13	0.11	0.09	0.07	0.05	0.02	−0.00	−0.03	−0.06	−0.09	−0.13	−0.17
	1		0.69	0.65	0.67	0.69	0.71	0.73	0.75	0.77	0.80	0.82	0.84	0.85	0.87	0.89	0.91	0.93	0.95	0.98	1.00	1.03	1.06	1.09	1.13	1.17
	2		0.99	0.95	1.01	1.07	1.14	1.20	1.26	1.32	1.39	1.45	1.51	1.57	1.62	1.68	1.74	1.80	1.86	1.93	2.00	2.08	2.17	2.27	2.38	2.50
	3		1.15	1.25	1.36	1.46	1.56	1.67	1.77	1.87	1.99	2.09	2.19	2.28	2.36	2.46	2.57	2.67	2.77	2.88	3.00	3.13	3.29	3.44	3.63	3.83
	4		1.41	1.55	1.70	1.84	1.99	2.14	2.27	2.42	2.58	2.72	2.86	2.99	3.11	3.25	3.40	3.54	3.68	3.83	4.00	4.19	4.39	4.62	4.88	5.17
	5		1.67	1.86	2.04	2.22	2.41	2.61	2.78	2.97	3.18	3.36	3.54	3.70	3.86	4.03	4.23	4.41	4.59	4.79	5.00	5.24	5.50	5.80	6.13	6.50
	6		1.93	2.16	2.38	2.61	2.84	3.07	3.29	3.52	3.77	3.99	4.21	4.41	4.60	4.82	5.05	5.28	5.50	5.74	6.00	6.29	6.61	6.97	7.36	7.84
	7		2.19	2.46	2.72	2.99	3.26	3.54	3.79	4.07	4.36	4.63	4.88	5.12	5.35	5.60	5.88	6.15	6.41	6.69	7.00	7.34	7.72	8.15	8.63	9.17
	8		2.46	2.76	3.07	3.37	3.69	4.01	4.30	4.62	4.95	5.27	5.56	5.83	6.09	6.36	6.71	7.02	7.32	7.64	8.00	8.40	8.84	9.33	9.88	10.50
	9		2.72	3.08	3.41	3.76	4.11	4.48	4.81	5.17	5.55	5.90	6.23	6.54	6.84	7.17	7.54	7.89	8.23	8.60	9.00	9.45	9.95	10.50	11.13	11.84
	10		2.98	3.38	3.75	4.14	4.54	4.94	5.31	5.71	6.15	6.54	6.91	7.25	7.58	7.95	8.37	8.76	9.14	9.55	10.00	10.50	11.06	11.68	12.38	13.17
	11		3.24	3.68	4.09	4.52	4.96	5.41	5.82	6.26	6.74	7.17	7.58	7.96	8.33	8.74	9.19	9.63	10.05	10.50	11.00	11.56	12.17	12.86	13.63	14.51
	12		3.50	3.96	4.43	4.91	5.39	5.88	6.33	6.81	7.34	7.81	8.26	8.67	9.07	9.52	10.02	10.50	10.96	11.46	12.00	12.61	13.28	14.03	14.88	15.84
	13		3.76	4.27	4.78	5.29	5.81	6.35	6.85	7.36	7.93	8.44	8.93	9.38	9.82	10.31	10.85	11.37	11.87	12.41	13.00	13.66	14.39	15.21	16.13	17.17
	14		4.02	4.57	5.12	5.67	6.24	6.82	7.34	7.91	8.53	9.06	9.61	10.09	10.57	11.09	11.69	12.24	12.78	13.36	14.00	14.71	15.51	16.39	17.38	18.51
	15		4.28	4.87	5.46	6.06	6.66	7.28	7.85	8.46	9.12	9.71	10.28	10.80	11.31	11.88	12.51	13.11	13.68	14.31	15.00	15.77	16.62	17.57	18.63	19.84
	16		4.54	5.17	5.80	6.44	7.09	7.75	8.35	9.01	9.72	10.35	10.95	11.51	12.06	12.65	13.33	13.98	14.59	15.27	16.00	16.82	17.73	18.74	19.88	21.18
	17		4.80	5.47	6.14	6.82	7.51	8.22	8.86	9.56	10.31	10.98	11.63	12.22	12.80	13.45	14.16	14.86	15.50	16.22	17.00	17.87	18.84	19.92	21.13	22.51
	18		5.06	5.77	6.49	7.21	7.94	8.69	9.37	10.11	10.91	11.62	12.30	12.93	13.55	14.23	14.99	15.72	16.41	17.17	18.00	18.93	19.95	21.10	22.38	23.84
	19		5.32	6.07	6.88	7.59	8.36	9.15	9.88	10.66	11.50	12.25	12.98	13.64	14.29	15.02	15.82	16.59	17.32	18.12	19.01	19.98	21.06	22.27	23.64	25.18
	20		5.58	6.37	7.17	7.97	8.79	9.62	10.38	11.20	12.09	12.89	13.65	14.35	15.04	15.80	16.64	17.46	18.23	19.08	20.01	21.03	22.17	23.45	24.89	26.51
Tenths																										
	0.0		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	0.1		0.03	0.03	0.03	0.04	0.04	0.05	0.05	0.05	0.06	0.07	0.07	0.07	0.07	0.08	0.08	0.09	0.09	0.10	0.10	0.11	0.11	0.12	0.13	0.13
	0.2		0.05	0.06	0.07	0.08	0.08	0.09	0.10	0.11	0.12	0.13	0.13	0.14	0.15	0.16	0.17	0.17	0.18	0.19	0.20	0.21	0.22	0.24	0.25	0.27
	0.3		0.08	0.09	0.10	0.11	0.13	0.14	0.15	0.16	0.18	0.19	0.20	0.21	0.22	0.24	0.25	0.26	0.27	0.29	0.30	0.32	0.33	0.35	0.38	0.40
	0.4		0.10	0.12	0.14	0.15	0.17	0.19	0.20	0.22	0.24	0.25	0.27	0.28	0.30	0.31	0.33	0.35	0.36	0.38	0.40	0.42	0.44	0.47	0.50	0.53
	0.5		0.13	0.15	0.17	0.19	0.21	0.23	0.25	0.27	0.30	0.32	0.34	0.35	0.37	0.39	0.41	0.43	0.45	0.48	0.50	0.53	0.56	0.59	0.63	0.67
	0.6		0.16	0.18	0.21	0.23	0.25	0.28	0.30	0.33	0.36	0.38	0.40	0.43	0.45	0.47	0.50	0.52	0.55	0.57	0.60	0.63	0.67	0.71	0.75	0.80
	0.7		0.18	0.21	0.24	0.27	0.30	0.33	0.35	0.38	0.42	0.44	0.47	0.50	0.52	0.55	0.59	0.61	0.64	0.67	0.70	0.74	0.78	0.82	0.88	0.93
	0.8		0.21	0.24	0.27	0.31	0.34	0.37	0.41	0.44	0.48	0.51	0.54	0.57	0.60	0.63	0.66	0.70	0.73	0.76	0.80	0.84	0.89	0.94	1.00	1.07
	0.9		0.23	0.27	0.31	0.34	0.38	0.42	0.46	0.49	0.54	0.57	0.61	0.64	0.67	0.71	0.75	0.78	0.82	0.86	0.90	0.95	1.00	1.06	1.13	1.20

FIG. X3.2 Corrected Loss from Observed Loss and Barometric Pressure mm Hg

X4. PROCEDURE TO EMULATE THE EMERGENT STEM ERROR OF A MERCURY-IN-GLASS THERMOMETER

X4.1 When an electronic or other sensor without an emergent stem error is used, the output of this sensor or the associated data system should emulate the output of a mercury-in-glass thermometer. Based on information supplied by four manufacturers of automated Test Method D 86 equipment, the averaged equations shown in X4.2 and X4.3 have been reported to be in use.

X4.1.1 The equations shown in X4.2 have limited applicability and are shown for information purposes only. In addition to the correction for the emergent stem, the electronic sensor and associated data system will also have to emulate the lag in response time observed for mercury-in-glass thermometers.

X4.2 When a low range thermometer would have been used, no stem correction is to be applied below 20°C. Above this temperature, the correction is calculated using the following formula:

$$ASTM\ 7C\ T_{elr} = T_t - 0.000162 \times (T_t - 20°C)^2 \quad (X4.1)$$

X4.3 When a high range thermometer would have been used, no stem correction is to be applied below 35°C. Above this temperature the correction is calculated using the following formula:

$$ASTM\ 8C\ T_{ehr} = T_t - 0.000131 \times (T_t - 35°C)^2 \quad (X4.2)$$

where:

T_{elr} = emulated temperature in °C for low range thermometers,
T_{ehr} = emulated temperature in °C for high range thermometers, and
T_t = true temperature in °C.

X5. EXPLANATORY REPORT FORMS

X5.1 Fig. X5.1 and Fig. X5.2 show report forms.

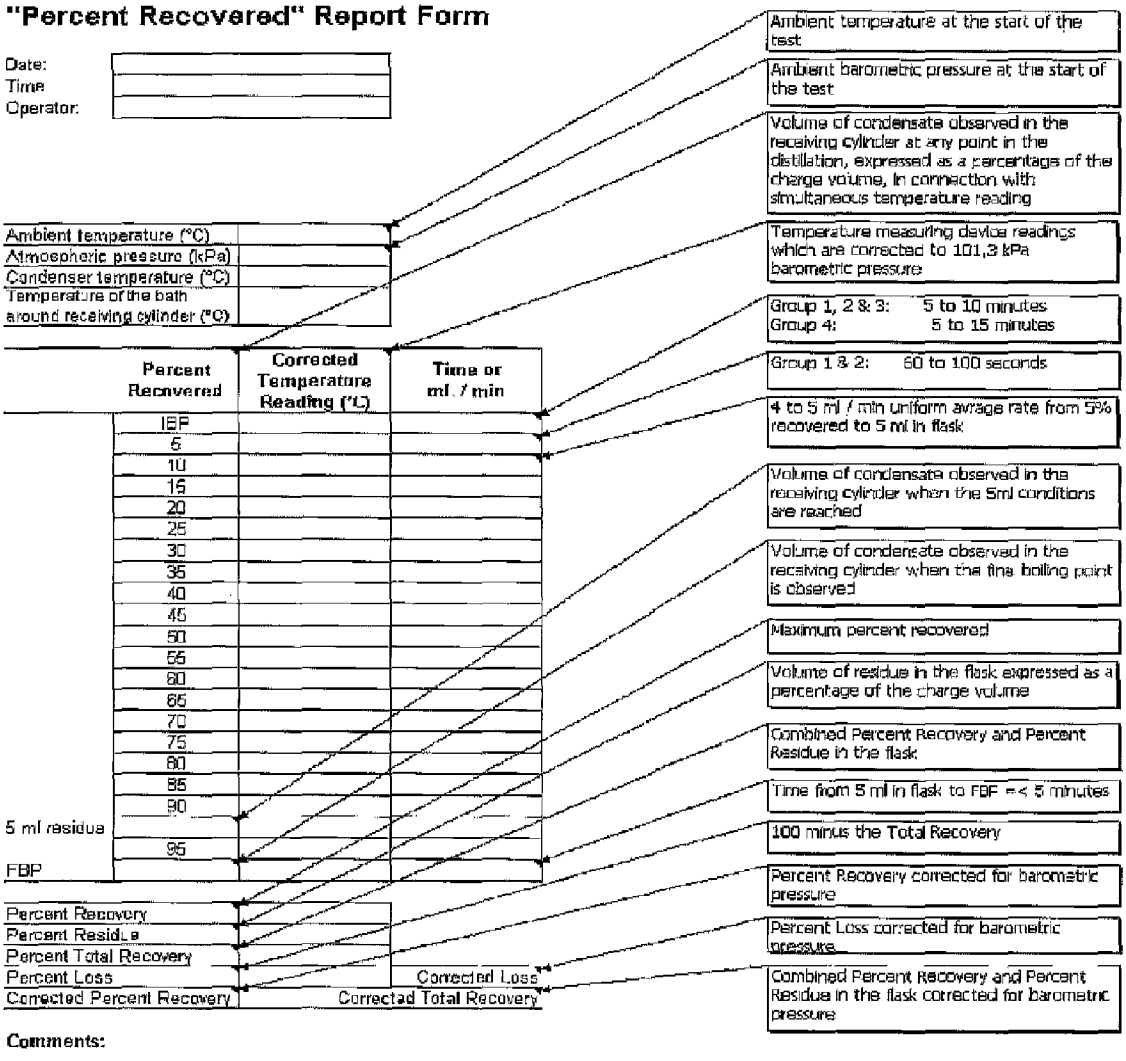


FIG. X5.1 Percent Recovered Report Form

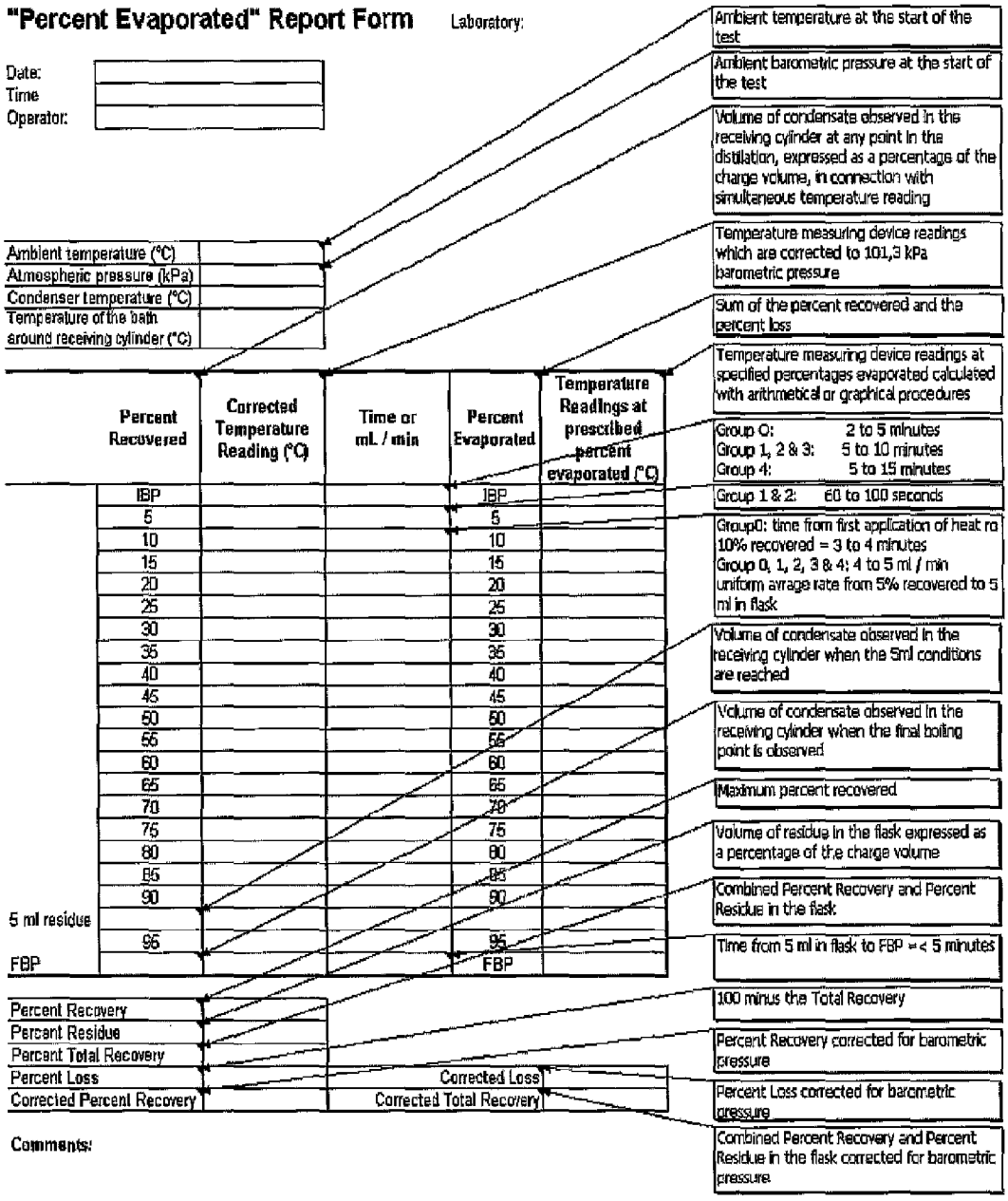


FIG. X5.2 Percent Evaporated Report Form

SUMMARY OF CHANGES

Subcommittee D02.08 has identified the location of selected changes to this standard since the last issue (D 86–05) that may impact the use of this standard. (Approved Jan. 15, 2007.)

(*1*) Deleted "natural gasolines" from 1.1.
(*2*) Deleted "Group 0" from the entire standard.
(*3*) Added Fig. 6.

Subcommittee D02.08 has identified the location of selected changes to this standard since the last issue, (D 86–04b), that may impact the use of this standard. (Approved July 1, 2005.)

(*1*) Replaced Table 4 with new values.
(*2*) Revised 9.1.2-9.1.2.2, 9.1.5, and Notes 9-11.
(*3*) Added 13.5.3 and footnote reference to the research report.
(*4*) Added Appendix X5, and cross-reference in Section 12.1.

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 D975: Standard Specification for Diesel Fuel Oils

CFR Section(s): 46 CFR 160.176-13(r)

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: D 975 – 98b

An American National Standard

Standard Specification for Diesel Fuel Oils[1]

This standard is issued under the fixed designation D 975; 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.

1. Scope

1.1 This specification covers five grades of diesel fuel oils suitable for various types of diesel engines. These grades are described as follows:

1.1.1 *Grade Low Sulfur No. 1-D*—A special-purpose, light distillate fuel for automotive diesel engines requiring low sulfur fuel and requiring higher volatility than that provided by Grade Low Sulfur No. 2-D.[2]

1.1.2 *Grade Low Sulfur No. 2-D*— A general-purpose, middle distillate fuel for automotive diesel engines requiring low sulfur fuel. It is also suitable for use in non-automotive applications, especially in conditions of varying speed and load.[2]

1.1.3 *Grade No. 1-D*—A special-purpose, light distillate fuel for automotive diesel engines in applications requiring higher volatility than that provided by Grade No. 2-D fuels.

1.1.4 *Grade No. 2-D*—A general-purpose, middle distillate fuel for automotive diesel engines, which is also suitable for use in non-automotive applications, especially in conditions of frequently varying speed and load.

1.1.5 *Grade No. 4-D*—A heavy distillate fuel, or a blend of distillate and residual oil, for low- and medium-speed diesel engines in non-automotive applications involving predominantly constant speed and load.

NOTE 1—A more detailed description of the grades of diesel fuel oils is given in Appendix X1.2.

1.2 This specification, unless otherwise provided by agreement between the purchaser and the supplier, prescribes the required properties of diesel fuels at the time and place of delivery.

1.2.1 Nothing in this specification shall preclude observance of federal, state, or local regulations which may be more restrictive.

NOTE 2—The generation and dissipation of static electricity can create problems in the handling of distillate diesel fuel oils. For more information on the subject, see Guide D 4865.

1.3 Values are stated in SI units and are regarded as the standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 56 Test Method for Flash Point by Tag Closed Tester[3]
D 86 Test Method for Distillation of Petroleum Products[3]
D 93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester[3]
D 97 Test Method for Pour Point of Petroleum Products[3]
D 129 Test Method for Sulfur in Petroleum Products (General Bomb Method)[3]
D 130 Test Method for Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test[3]
D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)[3]
D 482 Test Method for Ash from Petroleum Products[3]
D 524 Test Method for Ramsbottom Carbon Residue of Petroleum Products[3]
D 613 Test Method for Cetane Number of Diesel Fuel Oil[4]
D 976 Test Methods for Calculated Cetane Index of Distillate Fuels[3]
D 1266 Test Method for Sulfur in Petroleum Products (Lamp Method)[3]
D 1319 Test Method for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption[3]
D 1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)[3]
D 1552 Test Method for Sulfur in Petroleum Products (High-Temperature Method)[3]
D 1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)[3]
D 2274 Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)[3]
D 2276 Test Method for Particulate Contaminant in Aviation Fuel by Line Sampling[3]
D 2500 Test Method for Cloud Point of Petroleum Oils[3]
D 2622 Test Method for Sulfur in Petroleum Products by X-Ray Spectrometry[5]

[1] This specification is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.E on Burner, Diesel, Non-aviation Gas Turbine, and Marine Fuels.

Current edition approved Dec.10, 1998. Published February 1999. Originally published as D975 – 48 T. Last previous edition D975 – 98a.

[2] This fuel complies with 40 CFR Part 80—Regulation of Fuels and Fuel Additives: Fuel Quality Regulations for Highway Diesel Fuel Sold in 1993 and Later Calendar Years.

[3] *Annual Book of ASTM Standards*, Vol 05.01.

[4] *Annual Book of ASTM Standards*, Vol 05.04.

[5] *Annual Book of ASTM Standards*, Vol 05.02.

D 2709 Test Method for Water and Sediment in Distillate Fuels by Centrifuge[5]
D 2880 Specification for Gas Turbine Fuel Oils[5]
D 3117 Test Method for Wax Appearance Point of Distillate Fuels[5]
D 3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry[5]
D 3828 Test Methods for Flash Point by Small Scale Closed Tester[5]
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[5]
D 4294 Test Method for Sulfur in Petroleum Products by Energy–Dispersive X-Ray Fluorescence Spectrometry[5]
D 4539 Test Method for Filterability of Diesel Fuels by Low Temperature Flow Test (LTFT)[2]
D 4737 Test Method for Calculated Cetane Index by Four Variable Equation[6]
D 4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems[6]
D 5001 Test Method for Measurement of Lubricity of Aviation Turbine Fuels by the Ball-on-Cylinder Lubricity Evaluator (BOCLE)[6]
D 5771 Test Method for Cloud Point of Petroleum Products (Optical Detection Stepped Cooling Method)[6]
D 5772 Test Method for Cloud Point of Petroleum Products (Linear Cooling Rate Method)[6]
D 5773 Test Method for Cloud Point of Petroleum Products (Constant Cooling Rate Method)[6]
D 6078 Test Method for Evaluating Lubricity of Diesel Fuels by the Scuffing Load Ball–on–Cylinder Lubricity Evaluation (SLBOCLE)
D 6079 Test Method for Evaluating Lubricity of Diesel Fuels by the High–Fequency Reciprocating Rig (HFFR)

2.2 *Other Documents:*

26 CFR Part 48 Manufacturers and Realtors Excise Taxes
26 CFR Part 48 Diesel Fuel Excise Tax; Dye Color and Concentration
40 CFR Part 80 Regulation of Fuels and Fuel Additives
IP 309 Diesel and domestic heating fuels–Determination of cold filter plugging point

3. Test Methods

3.1 The requirements enumerated in this specification shall be determined in accordance with the following methods:

3.1.1 *Flash Point*—Test Method D 93, except where other methods are prescribed by law. For all grades, Test Method D 3828 can be used as an alternate with the same limits. For Grades Low Sulfur No. 1-D, Low Sulfur No. 2-D, No. 1-D, and No. 2-D, Test Method D 56 can be used as an alternate with the same limits, provided the flash point is below 93°C and the viscosity is below 5.5 mm^2/s at 40°C. This test method will give slightly lower values. In cases of dispute, Test Method D 93 shall be used as the referee method.

3.1.2 *Cloud Point*—Test Method D 2500. For all grades, the automatic Test Method D 5771, D 5772, or D 5773 can be used as alternates with the same limits.Test Method D 3117 can also be used since it is closely related to Test Method D 2500. In case of dispute, Test Method D 2500 shall be the referee method.

3.1.3 *Water and Sediment*—Test Method D 2709 is used for Grades Low Sulfur No. 1-D, Low Sulfur No. 2-D, No. 1-D, and No. 2-D. Test Method D 1796 is used for Grade No. 4-D.

3.1.4 *Carbon Residue*—Test Method D 524.

3.1.5 *Ash*—Test Method D 482.

3.1.6 *Distillation of Low Sulfur No. 1-D, Low Sulfur No. 2-D, No. 1-D, and No. 2-D Fuel Oils*—Test Method D 86.

3.1.7 *Viscosity*—Test Method D 445.

3.1.8 *Sulfur*—Test Method D 2622 is used for Grades Low Sulfur No. 1-D and Low Sulfur No. 2-D. Test Methods D 1266, D3120 and D4294 are also suitable for determining up to 0.05 % sulfur in diesel fuels. Test Method D 129 is used for Grades No. 1-D, No. 2-D and No. 4-D. Test Methods D 1552, D 2622, and D4294 can also be used for these grades. In addition, Test Method D 1266 can be used for Grade No. 1, but only with samples having sulfur contents of 0.4 mass % and less (down to 0.01 %). In case of dispute, Test Method D 2622 is the referee sulfur test method for Grades Low Sulfur No. 1-D and Low Sulfur No. 2-D. Test Method D 129 is the referee sulfur test method for Grades No. 1-D, No. 2-D, and No. 4-D.

3.1.9 *Corrosion*—Test Method D 130, 3 h test at 50°C.

3.1.10 *Cetane Number*—Test Method D 613.

3.1.11 *Cetane Index*—Method D 976 – 80.

3.1.12 *Aromaticity*—Test Method D 1319. This test method provides an indication of the aromatics content of fuels. For fuels with a maximum final boiling point of 315°C, this method is a measurement of the aromatic content of the fuel.

4. Workmanship

4.1 The diesel fuel shall be visually free of undissolved water, sediment, and suspended matter.

5. Requirements

5.1 The grades of diesel fuel oils herein specified shall be hydrocarbon oils conforming to the detailed requirements shown in Table 1.

6. Keywords

6.1 diesel; fuel oil; petroleum and petroleum products; specification

[6] *Annual Book of ASTM Standards*, Vol 05.03.

TABLE 1 Detailed Requirements for Diesel Fuel Oils[A]

Property	ASTM Test Method[B]	Grade Low Sulfur No. 1-D[C]	Grade Low Sulfur No. 2-D[C]	Grade No. 1-D[D]	Grade No. 2-D[D]	Grade No. 4-D[D]
Flash Point, °C, min.	D 93	38	52	38	52	55
Water and Sediment, % vol, max	D 2709	0.05	0.05	0.05	0.05	...
	D 1796	...	...	...	...	0.50
Distillation Temperature, °C 90 % % vol Recovered	D 86					
min		...	282[E]	...	282[E]	...
max		288	338	288	338	...
Kinematic Viscosity, mm^2/S at 40°C	D 445					
min.		1.3	1.9	1.3	1.9	5.5
max	...	2.4	4.1	2.4	4.1	24.0
Ash % mass, max	D 482	0.01	0.01	0.01	0.01	0.10
Sulfur, % mass, max[F]	D 2622[G]	0.05	0.05	...	...	...
	D 129	...	...	0.50	0.50	2.00
Copper strip corrosion rating max 3 h at 50°C	D 130	No. 3	No. 3	No. 3	No. 3	...
Cetane number, min[H]	D 613	40[I]	40[I]	40[I]	40[I]	30[I]
One of the following properties must be met:						
(1) Cetane index, min.	D 976[F]	40	40	...	...	...
(2) Aromaticity, % vol, max	D 1319[F]	35	35	...	...	...
Cloud point, °C, max	D 2500	[J]	[J]	[J]	[J]	[J]
Ramsbottom carbon residue on 10 % distillation residue, % mass, max	D 524	0.15	0.35	0.15	0.35	...

[A] To meet special operating conditions, modifications of individual limiting requirements may be agreed upon between purchaser, seller, and manufacturer.

[B] The test methods indicated are the approved referee methods. Other acceptable methods are indicated in 3.1.

[C] Under United States regulations, if Grades Low Sulfur No. 1-D or Low Sulfur No. 2-D are sold for tax exempt purposes then, at or beyond terminal storage tanks, they are required by 26 CFR Part 48 to contain the dye Solvent Red 164 at a concentration spectrally equivalent to 3.9 lbs per thousand barrels of the solid dye standard Solvent Red 26, or the tax must be collected.

[D] Under United States regulations, Grades No.1–D, No. 2–D, and No. 4–D are required by 40 CFR Part 80 to contain a sufficient amount of the dye Solvent Red 164 so its presence is visually apparent. At or beyond terminal storage tanks, they are required by 26 CFR Part 48 to contain the dye Solvent Red 164 at a concentration spectrally equivalent to 3.9 lbs per thousand barrels of the solid dye standard Solvent Red 26.

[E] When a cloud point less than – 12°C is specified, the minimum flash point shall be 38°C, the minimum viscosity at 40°C shall be 1.7 mm^2/s, and the minimum 90 % recovered temperature shall be waived.

[F] Other sulfur limits can apply in selected areas in the United States and in other countries.

[G] These test methods are specified in 40 CFR Part 80.

[H] Where cetane number by Test Method D 613 is not available, Test Method D 4737 can be used as an approximation.

[I] Low ambient temperatures as well as engine operation at high altitudes may require the use of fuels with higher cetane ratings.

[J] It is unrealistic to specify low temperature properties that will ensure satisfactory operation at all ambient conditions. In general, cloud point (or wax appearance point) may be used as an estimate of operating temperature limits for Grades Low Sulfur No. 1; Low Sulfur No. 2; and No. 1 and No. 2 diesel fuel oils. However, satisfactory operation below the cloud point (or wax appearance point) may be achieved depending on equipment design, operating conditions, and the use of flow-improver additives as described in X4.1.2. Tenth percentile minimum air temperatures for U.S. locations are provided in Appendix X4 as a means of estimating expected regional temperatures. This guidance is general. Some equipment designs or operation may allow higher or require lower cloud point fuels. Appropriate low temperature operability properties should be agreed upon between the fuel supplier and purchaser for the intended use and expected ambient temperatures.

APPENDIXES

(Nonmandatory Information)

X1. SIGNIFICANCE OF ASTM SPECIFICATION FOR DIESEL FUEL OILS

X1.1 Introduction

X1.1.1 The properties of commercial fuel oils depend on the refining practices employed and the nature of the crude oils from which they are produced. Distillate fuel oils, for example, may be produced within the boiling range of 150 and 400°C having many possible combinations of various properties, such as volatility, ignition quality, viscosity, and other characteristics.

X1.2 Grades

X1.2.1 This specification is intended as a statement of permissible limits of significant fuel properties used for specifying the wide variety of commercially available diesel fuel oils. Limiting values of significant properties are prescribed for five grades of diesel fuel oils. These grades and their general applicability for use in diesel engines are broadly indicated as follows:

X1.2.2 *Grade Low Sulfur No. 1-D*—Grade Low Sulfur No. 1-D comprises the class of low-sulfur, volatile fuel oils from kerosine to the intermediate distillates. Fuels within this grade are applicable for use in high-speed engines that require low sulfur fuel and in services involving frequent and relatively wide variations in loads and speeds, and also for use in cases where abnormally low fuel temperatures are encountered.

X1.2.3 *Grade Low Sulfur No. 2-D*—Grade Low Sulfur No. 2-D includes the class of low-sulfur, distillate gas oils of lower volatility than Grade Low Sulfur No. 1-D. These fuels are applicable for use in high-speed engines that require low sulfur

fuels and in services involving relatively high loads and uniform speeds, or in engines not requiring fuels having the higher volatility or other properties specified for Grade Low Sulfur No. 1-D.

X1.2.4 *Grade No. 1-D*— Grade No. 1-D comprises the class of volatile fuel oils from kerosine to the intermediate distillates. Fuels within this grade are applicable for use in high-speed engines in services involving frequent and relatively wide variations in loads and speeds, and also for use in cases where abnormally low fuel temperatures are encountered.

X1.2.5 *Grade No. 2-D*— Grade No. 2-D includes the class of distillate gas oils of lower volatility. These fuels are applicable for use in high-speed engines in services involving relatively high loads and uniform speeds, or in engines not requiring fuels having the higher volatility or other properties specified for Grade No. 1-D.

X1.2.6 *Grade No. 4-D*— Grade No. 4-D covers the class of more viscous distillates and blends of these distillates with residual fuel oils. These fuels are applicable for use in low- and medium-speed engines employed in services involving sustained loads at substantially constant speed.

X1.3 Selection of Particular Grade

X1.3.1 The selection of a particular diesel fuel oil from one of these three ASTM grades for use in a given engine requires consideration of the following factors:

X1.3.1.1 Fuel price and availability,
X1.3.1.2 Maintenance considerations,
X1.3.1.3 Engine size and design,
X1.3.1.4 Emission control systems,
X1.3.1.5 Speed and load ranges,
X1.3.1.6 Frequency of speed and load changes, and
X1.3.1.7 Atmospheric conditions. Some of these factors can influence the required fuel properties outlined as follows:

X1.4 Cetane Number

X1.4.1 Cetane number is a measure of the ignition quality of the fuel and influences combustion roughness. The cetane number requirements depend on engine design, size, nature of speed and load variations, and on starting and atmospheric conditions. Increase in cetane number over values actually required does not materially improve engine performance. Accordingly, the cetane number specified should be as low as possible to assure maximum fuel availability.

X1.5 Distillation

X1.5.1 The fuel volatility requirements depend on engine design, size, nature of speed and load variations, and starting and atmospheric conditions. For engines in services involving rapidly fluctuating loads and speeds as in bus and truck operation, the more volatile fuels may provide best performance, particularly with respect to smoke and odor. However, best fuel economy is generally obtained from the heavier types of fuels because of their higher heat content.

X1.6 Viscosity

X1.6.1 For some engines it is advantageous to specify a minimum viscosity because of power loss due to injection pump and injector leakage. Maximum viscosity, on the other hand, is limited by considerations involved in engine design and size, and the characteristics of the injection system.

X1.7 Carbon Residue

X1.7.1 Carbon residue gives a measure of the carbon depositing tendencies of a fuel oil when heated in a bulb under prescribed conditions. While not directly correlating with engine deposits, this property is considered an approximation.

X1.8 Sulfur

X1.8.1 The effect of sulfur content on engine wear and deposits appears to vary considerably in importance and depends largely on operating conditions. Fuel sulfur can affect emission control systems performance. To assure maximum availability of fuels, the permissible sulfur content should be specified as high as is practicable, consistent with maintenance considerations.

X1.9 Flash Point

X1.9.1 The flash point as specified is not directly related to engine performance. It is, however, of importance in connection with legal requirements and safety precautions involved in fuel handling and storage, and is normally specified to meet insurance and fire regulations.

X1.10 Cloud Point

X1.10.1 Cloud point is of importance in that it defines the temperature at which a cloud or haze of wax crystals appears in the oil under prescribed test conditions which generally relates to the temperature at which wax crystals begin to precipitate from the oil in use.

X1.11 Ash

X1.11.1 Ash-forming materials may be present in fuel oil in two forms: *(1)* abrasive solids, and *(2)* soluble metallic soaps. Abrasive solids contribute to injector, fuel pump, piston and ring wear, and also to engine deposits. Soluble metallic soaps have little effect on wear but may contribute to engine deposits.

X1.12 Copper Strip Corrosion

X1.12.1 This test serves as a measure of possible difficulties with copper and brass or bronze parts of the fuel system.

X1.13 Aromaticity

X1.13.1 This test is used as an indication of the aromatic contents of diesel fuel. Aromatic content is specified to prevent an increase in the average aromatics in Grades Low Sulfur No. 1-D and Low Sulfur No. 2-D fuels. Increases in aromatic content of fuels over current levels may have a negative impact on emissions.

X1.14 Cetane Index

X1.14.1 Cetane Index is specified as a limitation on the amount of high aromatic components in Grades Low Sulfur No. 1-D and Low Sulfur No. 2-D.

X2. LONG-TERM STORAGE OF DISTILLATE FUELS

X2.1 Scope

X2.1.1 This appendix provides guidance for consumers of distillate fuels who may wish to store quantities of fuels for extended periods. Fuels containing residual components are excluded. Consistently successful long-term fuel storage requires attention to fuel selection, storage conditions, and monitoring of properties prior to and during storage.

X2.1.2 Normally produced fuels have adequate stability properties to withstand normal storage without the formation of troublesome amounts of insoluble degradation products. Fuels that are to be stored for prolonged periods should be selected to avoid formation of sediments, which can overload filters or plug combustor nozzles or injectors. Selection of these fuels should result from supplier-user discussions.

X2.1.3 These suggested practices are general in nature and should not be considered substitutes for any requirements imposed by the warranty of the distillate fuel equipment manufacturer or by federal, state, or local government regulations. Although they cannot replace a knowledge of local conditions or good engineering and scientific judgment, these suggested practices do provide guidance in developing an individual fuel management system for the distillate fuel user. They include suggestions in the operation and maintenance of existing fuel storage and handling facilities and for identifying where, when, and how fuel quality should be monitored.

X2.2 Definitions

X2.2.1 *long-term storage*—storage of fuel for longer than 12 months after it is received by the user.

X2.2.2 *bulk fuel*—fuel in the storage facility.

X2.2.3 *combustor fuel*— fuel entering the combustion zone of the burner or engine after filtration or other treatment of bulk fuel.

X2.2.4 *fuel contaminants*—foreign materials that make fuel less suitable or unsuitable for the intended use. Fuel contaminants include materials introduced subsequent to the manufacture of fuel and fuel degradation products.

X2.2.5 *fuel-degradation products*—those materials that are formed in fuel during extended storage. Insoluble degradation products may combine with other fuel contaminants to reinforce deleterious effects. Soluble degradation products (soluble gums) are less volatile than fuel and may carbonize to form in fuels due to complex interactions and oxidation of small amounts of olefinic, sulfurous, oxygenated, and nitrogenous compounds present in fuels. The formation of degradation products may be catalyzed by dissolved metals, especially copper salts.

X2.3 Fuel Selection

X2.3.1 Certain distilled refinery products are generally more suitable for long-term storage than others. The stability properties of distillates are highly dependent on the crude oil sources, severity of processing, and whether additional refinery treatment has been carried out.

X2.3.2 The composition and stability properties of distillate fuels produced at specific refineries may be different. Any special requirements of the user, such as long-term storage, should be discussed with the supplier.

X2.3.3 Blends of fuels from various sources may interact to give stability properties worse than expected based on the characteristics of the individual fuels.

X2.4 Fuel Additives

X2.4.1 Available fuel additives can improve the suitability of marginal fuels for long-term storage but may be unsuccessful for fuels with markedly poor stability properties. Most additives should be added at the refinery or during the early weeks of storage to obtain maximum benefits.

X2.4.2 Biocides or biostats destroy or inhibit the growth of fungi and bacteria, which can grow at fuel-water interfaces to give high particulate concentrations in the fuel. Available biocides are soluble in both the fuel and water or in the water phase only.

X2.5 Tests for Fuel Quality

X2.5.1 At the time of manufacture, the storage stability of fuel may be assessed using Test Method D 2274. However, this accelerated stability test may not correlate well with field storage stability due to varying field conditions and to fuel composition.

X2.5.2 Performance criteria for accelerated stability tests that assure satisfactory long-term storage of fuels have not been established.

X2.6 Fuel Monitoring

X2.6.1 A plan for monitoring the quality of bulk fuel during prolonged storage is an integral part of a successful program. A plan to replace aged fuel with fresh product at established intervals is also desirable.

X2.6.2 Stored fuel should be periodically sampled and its quality assessed. Practice D 4057 provides guidance for sampling. Fuel contaminants and degradation products will usually settle to the bottom of a quiescent tank. A "Bottom" or "Clearance" sample, as defined in Practice D 4057, should be included in the evaluation along with an "All Level" sample.

X2.6.3 The quantity of insoluble fuel contaminants present in fuel can be determined using Test Method D 2276, Procedure A.

X2.6.4 Other quality tests like fuel color (see Test Method D 1500) and stability tests (see Test Method D 2274) after storage may have value. Correlations of these tests with fuel suitability are tenuous.

X2.7 Fuel Storage Conditions

X2.7.1 Contamination levels in fuel can be reduced by storage in tanks kept free of water, and tankage should have provisions for water draining on a scheduled basis. Water promotes corrosion, and microbiological growth may occur at a fuel-water interface. Underground storage is preferred to avoid temperature extremes; above-ground storage tanks should be sheltered or painted with reflective paint. High storage temperatures accelerate fuel degradation. Fixed roof

tanks should be kept full to limit oxygen supply and tank breathing.

X2.7.2 Copper and copper-containing alloys should be avoided. Copper can promote fuel degradation and may produce mercaptide gels. Zinc coatings can react with water or organic acids in the fuel to form gels which rapidly plug filters.

X2.7.3 Appendix X3 of Specification D 2880 discusses fuel contaminants as a general topic.

X2.8 Use of Degraded Fuels

X2.8.1 Fuels that have undergone mild-to-moderate degradation can often be consumed in a normal way, depending on the fuel system requirements. Filters and other cleanup equipment can require special attention and increased maintenance. Burner nozzle or injector fouling can occur more rapidly.

X2.8.2 Fuels containing very large quantities of fuel degradation products and other contaminants or with runaway microbiological growth require special attention. Consultation with experts in this area is desirable. It can be possible to drain the sediment or draw off most of the fuel above the sediment layer and use it with the precautions described in X2.8.1. However, very high soluble gum levels or corrosion products from microbiological contamination can cause severe operational problems.

X3. DIESEL FUEL LUBRICITY

X3.1 Introduction

X3.1.1 Diesel fuel functions as a lubricant in certain items of fuel injection equipment such as rotary/distributor fuel pumps and injectors. In limited cases, fuel with very specific properties can have insufficient lubricating properties which can lead to a reduction in the normal service life of fuel pumps or injectors.

X3.2 Fuel Characteristics Affecting Equipment Wear

X3.2.1 Two fuel characteristics that affect equipment wear are low viscosity and lack of sufficient quantities of trace components, which have an affinity for metal surfaces. If fuel viscosity meets the requirements of a particular engine, a fuel film is maintained between the moving surfaces of the fuel system components. This prevents excessive metal-to-metal contact and avoids premature failure due to wear. Similarly, certain surface active molecules in the fuel adhere to, or combine with, metallic surfaces to produce a protective film which also can protect surfaces against excessive wear.

X3.3 Fuel Lubricity

X3.3.1 The concern about fuel lubricity is limited to situations in which fuels with lower viscosities than those specified for a particular engine are used or with fuels which have been processed in a manner that results in the elimination of the trace levels of the surface active species that act as lubricating agents. Presently the only fuels of the latter type shown to have lubricity problems resulted from sufficiently severe processing to reduce aromatics substantially below current levels. Research is in progress to identify the characteristics of such fuels and where the use of a lubricity improver additive is required, to ensure satisfactory operation in the sensitive areas of the vehicle fuel system.

X3.3.2 Work in the area of diesel fuel lubricity is ongoing by several organizations, such as the International Standard Organization (ISO) and the ASTM Diesel Fuel Lubricity Task Force. These groups include representatives from the fuel injection equipment manufacturers, fuel producers, and additive suppliers. The charge of the ASTM task force has been the recommendation of test methods and a fuel specification for Specification D 975. Two test methods were proposed and approved. These are Test Method D 6078, a scuffing load ball-on-cylinder lubricity evaluator method, SLBOCLE, and Test Method D 6079, a high frequency reciprocating rig method, HFRR. The inclusion of a single fuel specification in the main table for Grade No. 2 requires further research because 1) the correlation of the data among the two test methods and the fuel injection equipment needs further clarification, 2) both methods in their current from do not apply to all fuel-additive combinations, and 3) the reproducibility values for both test methods are large. In the meantime, the following information may be of use and serve as a general guideline to fuel suppliers and users.

X3.3.3 SAE Technical Paper 952369[7] indicates that users should monitor their fuel injection pumps for possible trends of abnormal wear rates if the fuel has a scuffing load value between 2000 and 2800 g in Test Method D 6078. According to this paper, fuels with values below 2000 g will in all probability cause accelerated wear in fuel lubricated rotary-type fuel injection pumps. It should be noted that fuels with a sufficient level of an effective lubricity additive may protect the equipment, but may not be recognized by the test method.

X3.3.4 Work at ISO, documented in SAE Technical Paper 952372,[8] indicates that a fuel with a 450-micron wear scar diameter or lower value at 60 °C in Test Method D 6079 (380 micron at 25 °C) should protect all fuel injection equipment. Other SAE publications present data to show that some fuels and fuel/additive combinations can have values above this level and still provide sufficient lubricity protection to the equipment. The current ISO test program is addressing this issue.

X3.3.5 Pump stand testing of fuels, although more expensive and time consuming, is a more accurate means of evaluating the lubricity of diesel fuel. Although several fuel injection manufacturers have pump-stand tests, no single test method has become standardized and no standard industry-approved type procedure is available at this time.

[7] Westbrook, S.R., "Survey of Low Sulfur Diesel Fuels and Aviation Kerosenes from U.S. Military Installations", SAE Technical Paper 952369, 1995.

[8] Nikanjam, M., "ISO Diesel Fuel Lubricity Round Robin Program", SAE Technical Paper 952372, 1995.

X4. TENTH PERCENTILE MINIMUM AMBIENT AIR TEMPERATURES FOR THE UNITED STATES (EXCEPT HAWAII)

X4.1 Introduction

X4.1.1 The tenth percentile minimum ambient air temperatures shown on the following maps (Figs. X4.1-X4.12) and in Table X4.1 were derived from an analysis of historical hourly temperature readings recorded over a period of 15 to 21 years from 345 weather stations in the United States. This study was conducted by the U.S. Army Mobility Equipment Research and Development Center (USAMERDC), Coating and Chemical Laboratory, Aberdeen Proving Ground, MD 21005. The tenth percentile minimum ambient air temperature is defined as the lowest ambient air temperature which will not go lower on average more than 10 % of the time. In other words, the daily minimum ambient air temperature would on average not be expected to go below the monthly tenth percentile minimum ambient air temperature more than 3 days for a 30 day month.

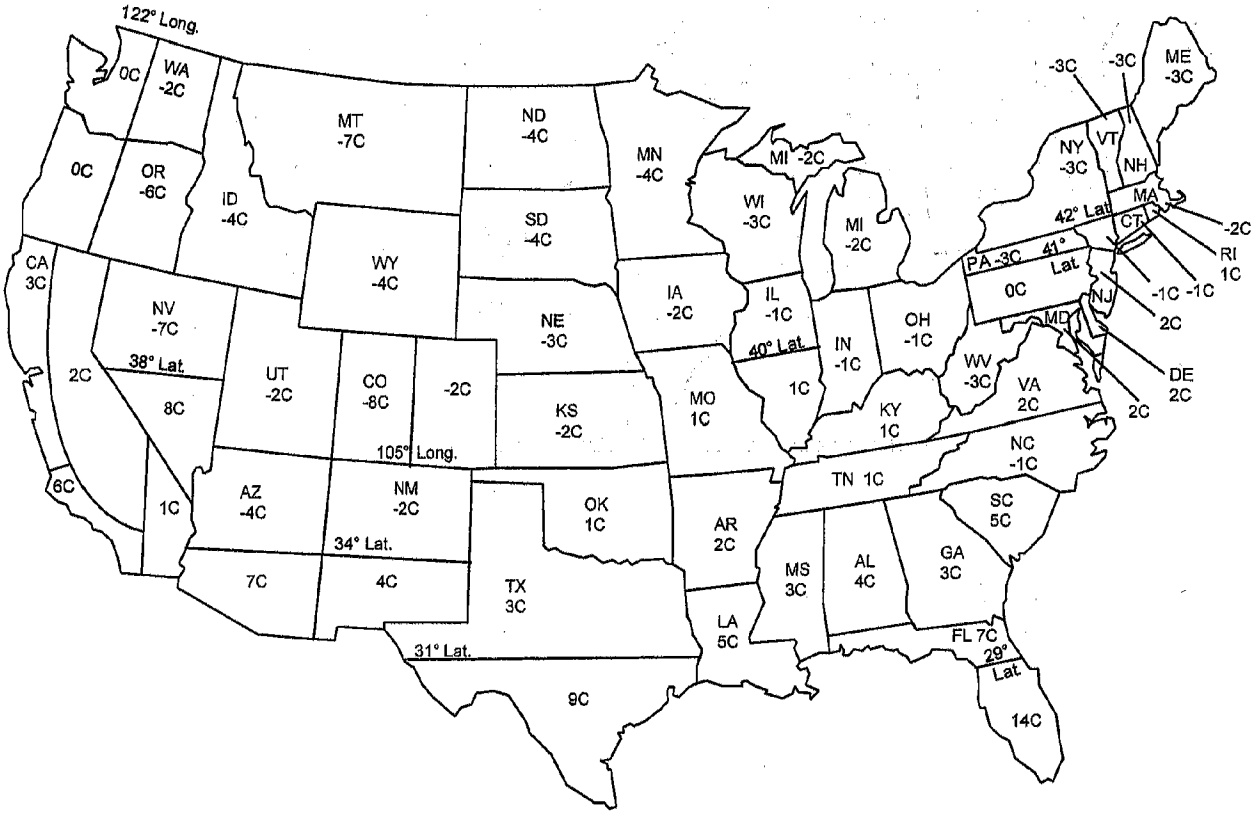


FIG. X4.1 October—10th Percentile Minimum Temperatures

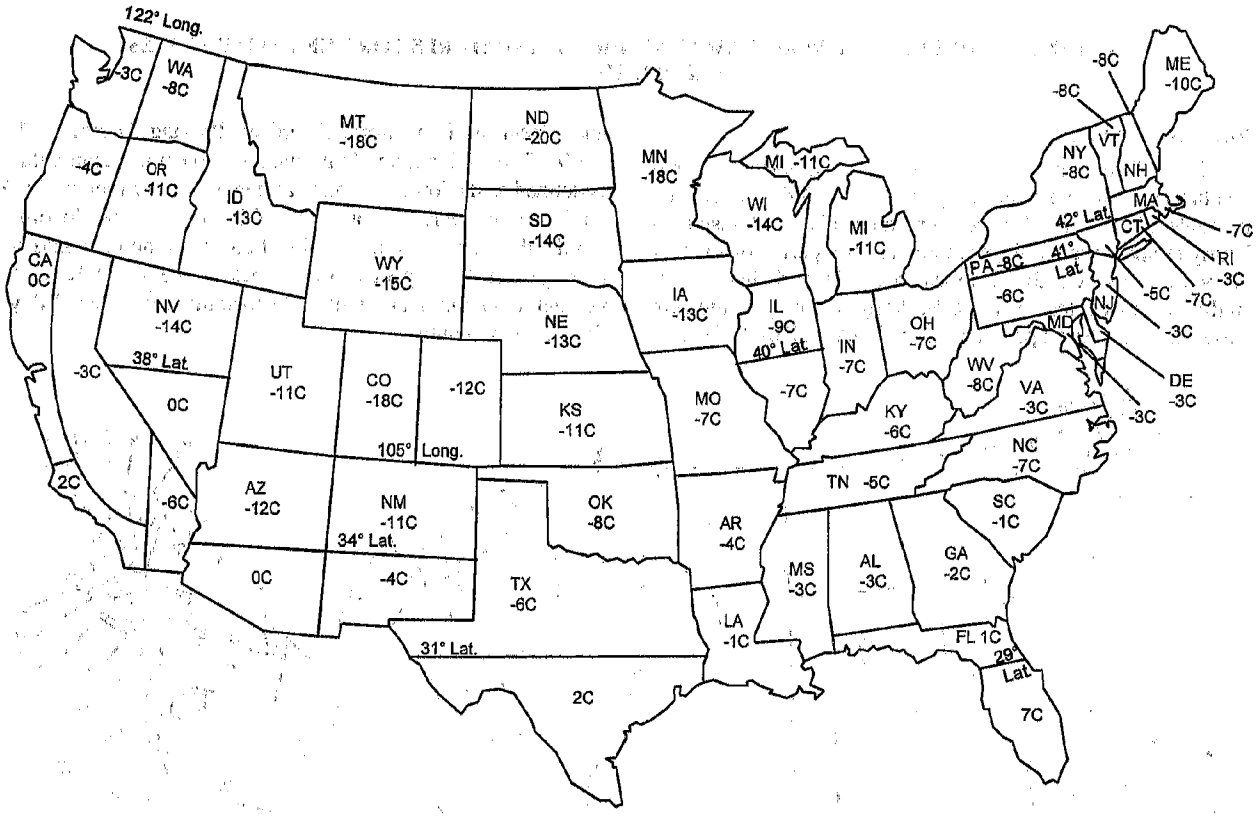


FIG. X4.2 November—10th Percentile Minimum Ambient Air Temperatures

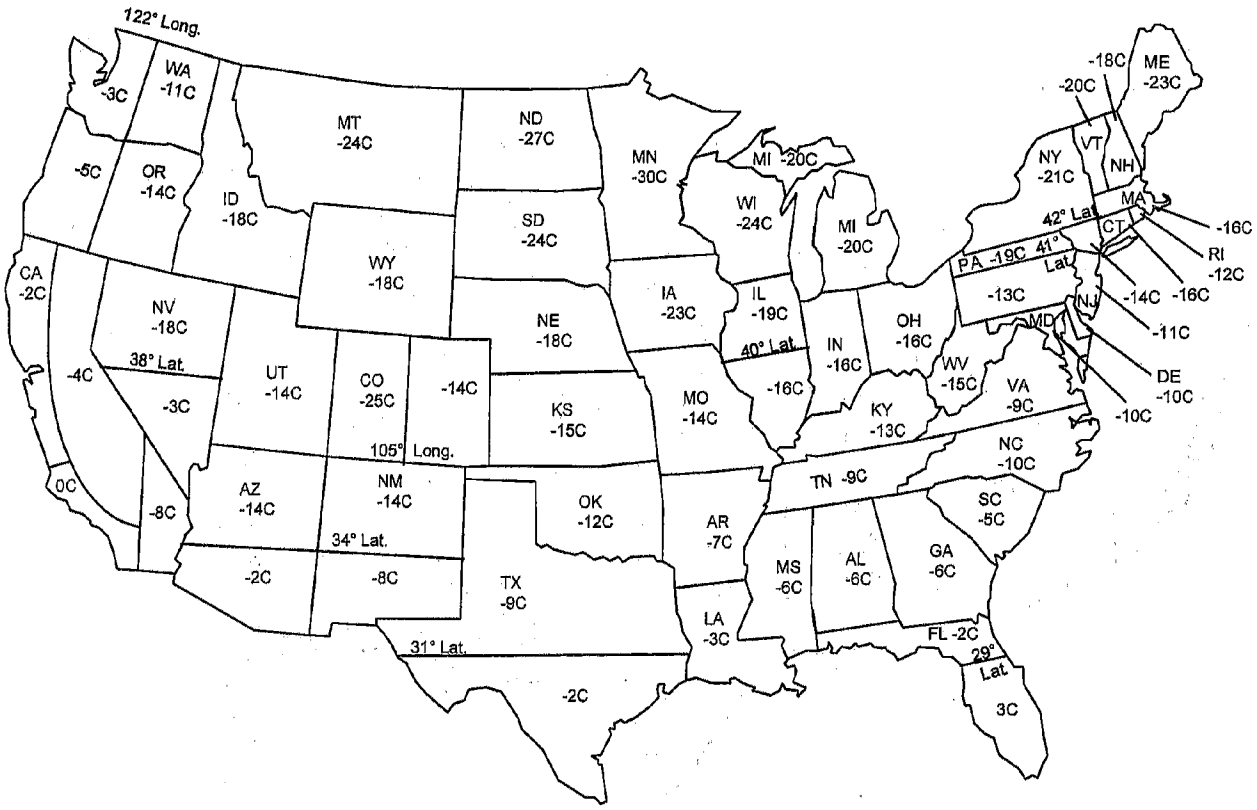


FIG. X4.3 December—10th Percentile Minimum Ambient Air Temperatures

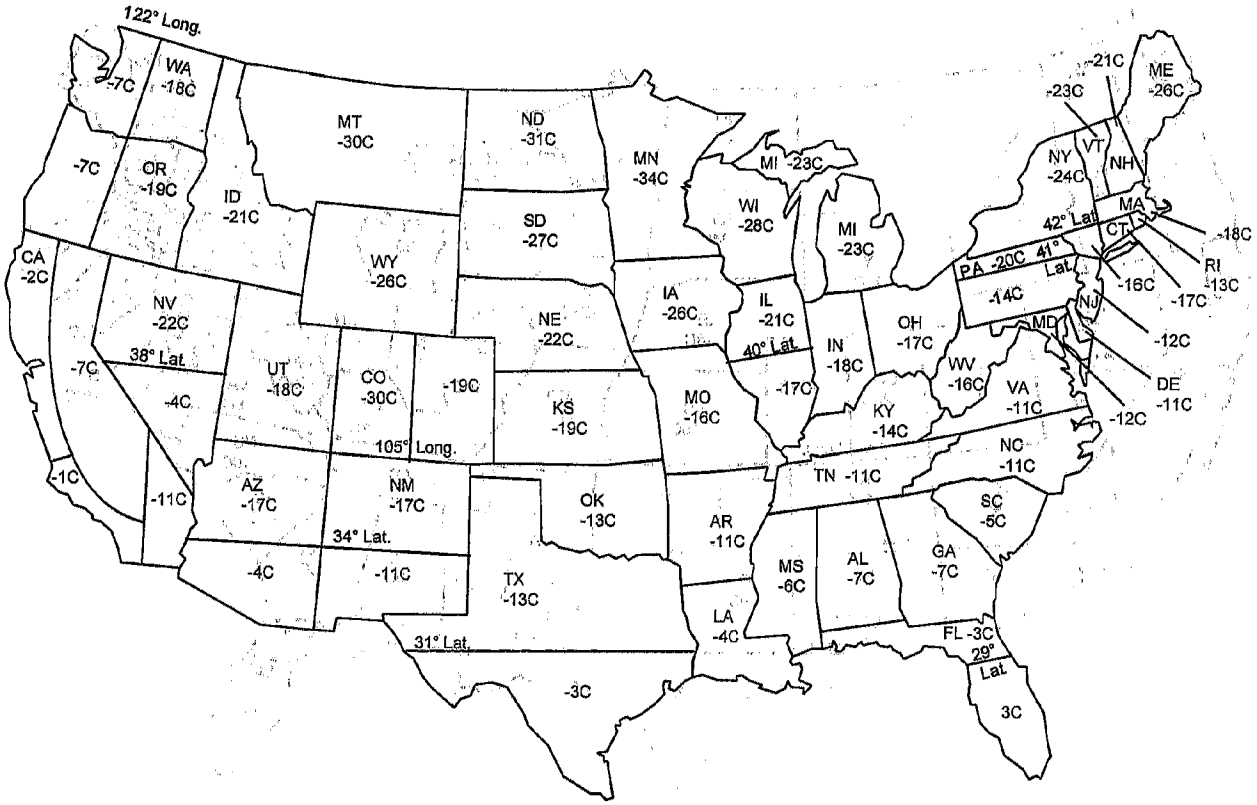


FIG. X4.4 January—10th Percentile Minimum Ambient Air Temperatures

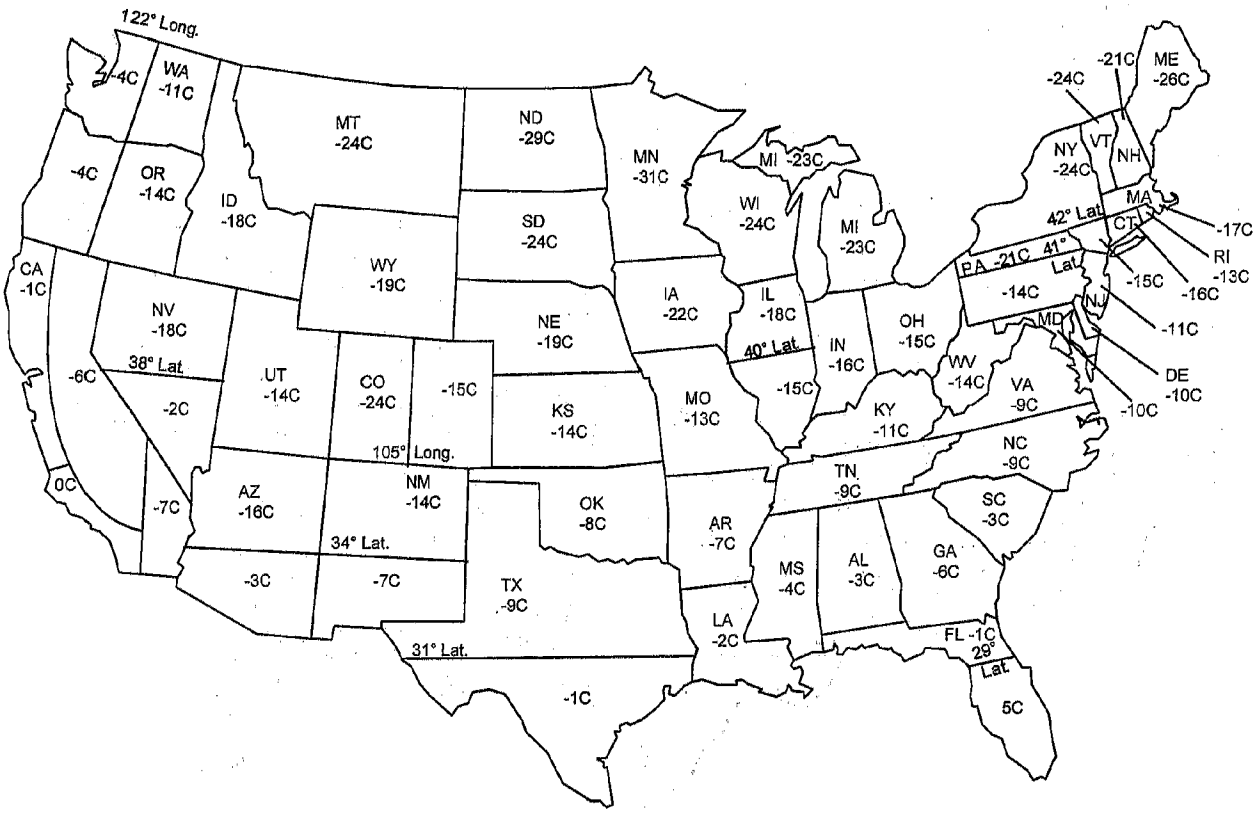


FIG. X4.5 February—10th Percentile Minimum Ambient Air Temperatures

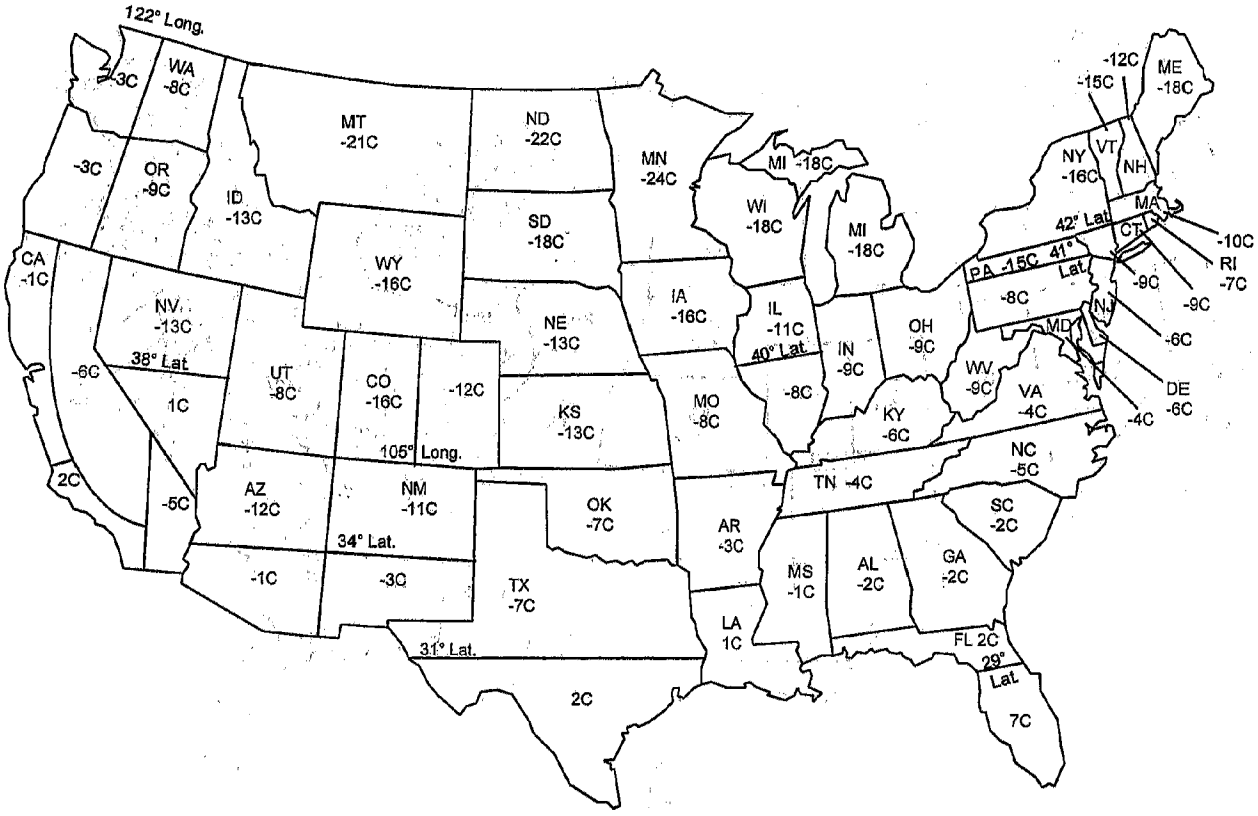


FIG. X4.6 March—10th Percentile Minimum Ambient Air Temperatures

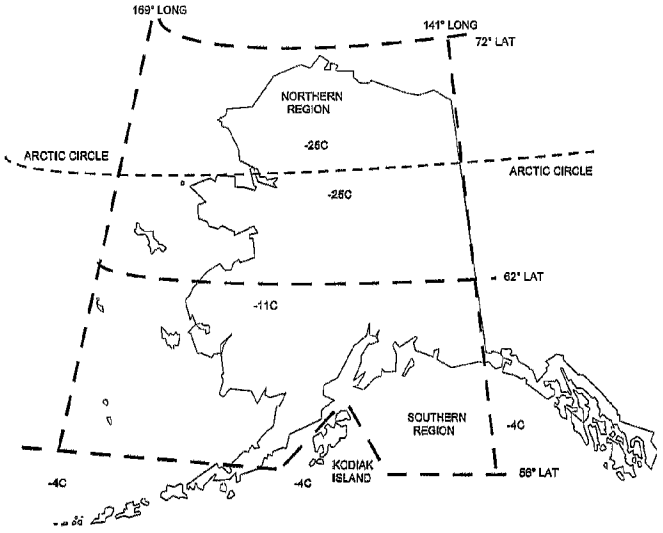


FIG. X4.7 October—10th Percentile Minimum Ambient Air Temperatures

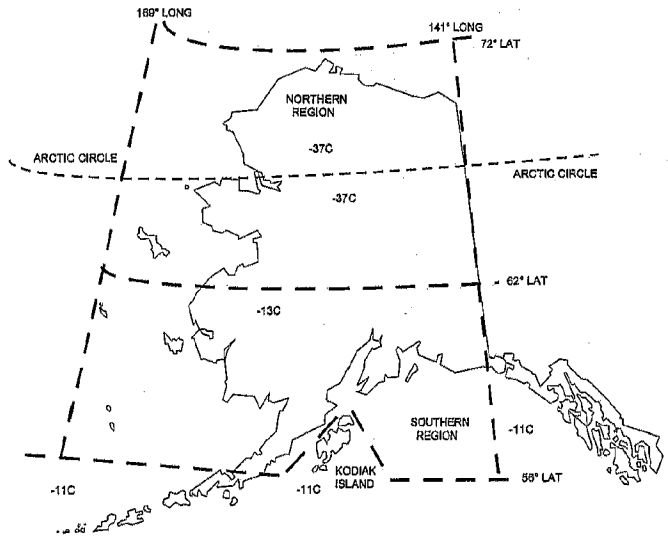


FIG. X4.8 November—10th Percentile Minimum Ambient Air Temperatures

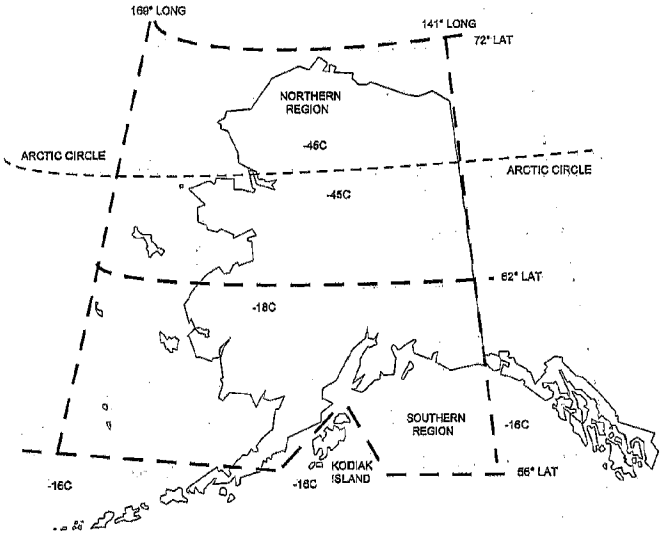


FIG. X4.9 December—10th Percentile Minimum Ambient Air Temperatures

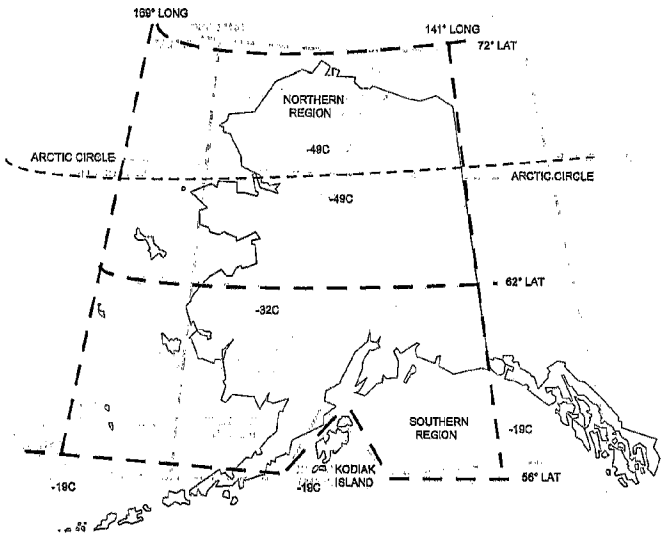


FIG. X4.10 January—10th Percentile Minimum Ambient Air Temperatures

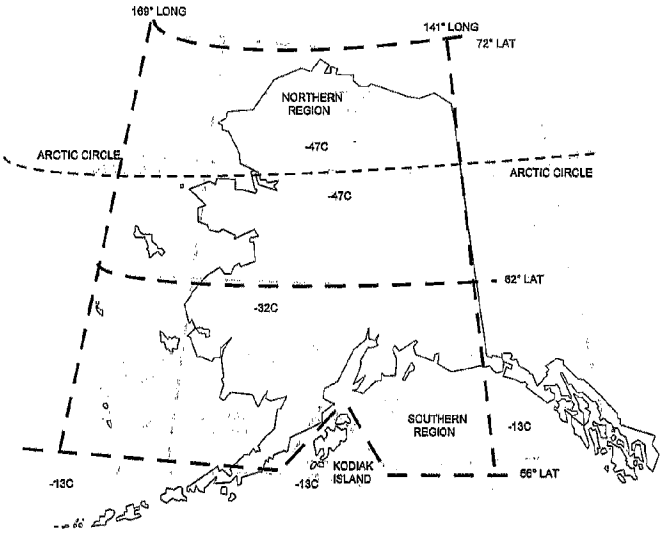


FIG. X4.11 February—10th Percentile Minimum Ambient Air Temperatures

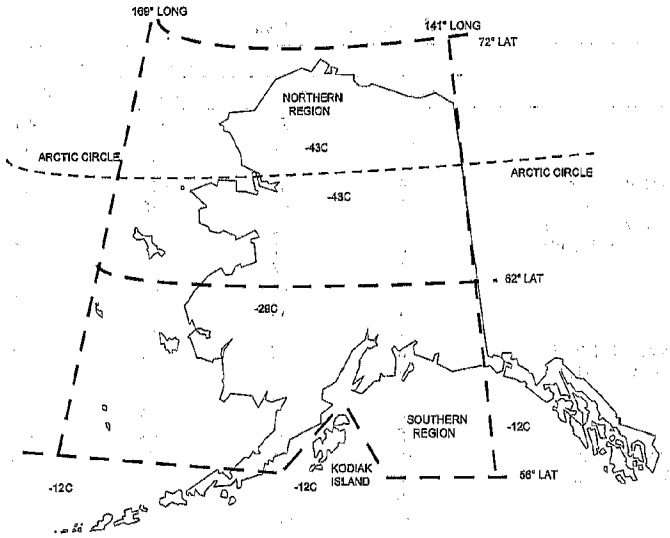


FIG. X4.12 March—10th Percentile Minimum Ambient Air Temperatures

TABLE X4.1 Tenth Percentile Minimum Ambient Air Temperatures for the United States (except Hawaii)

State		10th Percentile Temperature°C, min					
		Oct.	Nov.	Dec.	Jan.	Feb.	March
Alabama		4	−3	−6	−7	−3	−2
Alaska	Northern	−25	−37	−45	−49	−47	−43
	Southern	−11	−13	−18	−32	−32	−29
	South East	−4	−11	−16	−19	−13	−12
Arizona	North 34° latitude	−4	−12	−14	−17	−16	−12
	South 34° latitude	7	0	−2	−4	−3	−1
Arkansas		2	−4	−7	−11	−7	−3
California	North Coast	3	0	−2	−2	−1	−1
	Interior	2	−3	−4	−7	−6	−6
	South Coast	6	2	0	−1	0	2
	Southeast	1	−6	−8	−11	−7	−5
Colorado	East 105° long	−2	−12	−14	−19	−15	−12
	West 105° long	−8	−18	−25	−30	−24	−16
Connecticut		−1	−7	−16	−17	−16	−9
Delaware		2	−3	−10	−11	−10	−6
Florida	North 29° latitude	7	1	−2	−3	−1	2
	South 29° latitude	14	7	3	3	5	7
Georgia		3	−2	−6	−7	−6	−2
Idaho		−4	−13	−18	−21	−18	−13
Illinois	North 40° latitude	−1	−9	−19	−21	−18	−11
	South 40° latitude	1	−7	−16	−17	−15	−8
Indiana		−1	−7	−16	−18	−16	−9
Iowa		−2	−13	−23	−26	−22	−16
Kansas		−2	−11	−15	−19	−14	−13
Kentucky		1	−6	−13	−14	−11	−6
Louisiana		5	−1	−3	−4	−2	1
Maine		−3	−10	−23	−26	−26	−18
Maryland		2	−3	−10	−12	−10	−4
Massachusetts		−2	−7	−16	−18	−17	−10
Michigan		−2	−11	−20	−23	−23	−18
Minnesota		−4	−18	−30	−34	−31	−24
Mississippi		3	−3	−6	−6	−4	−1
Missouri		1	−7	−14	−16	−13	−8
Montana		−7	−18	−24	−30	−24	−21
Nebraska		−3	−13	−18	−22	−19	−13
Nevada	North 38° latitude	−7	−14	−18	−22	−18	−13
	South 38° latitude	8	0	−3	−4	−2	1
New Hampshire		−3	−8	−18	−21	−21	−12
New Jersey		2	−3	−11	−12	−11	−6
New Mexico	North 34° latitude	−2	−11	−14	−17	−14	−11
	South 34° latitude	4	−4	−8	−11	−7	−3
New York	North 42° latitude	−3	−8	−21	−24	−24	−16
	South 42° latitude	−1	−5	−14	−16	−15	−9
North Carolina		−1	−7	−10	−11	−9	−5

TABLE X4.1 *Continued*

State		10th Percentile Temperature°C, min					
		Oct.	Nov.	Dec.	Jan.	Feb.	March
North Dakota		−4	−20	−27	−31	−29	−22
Ohio		−1	−7	−16	−17	−15	−9
Oklahoma		1	−8	−12	−13	−8	−7
Oregon	East 122° long	−6	−11	−14	−19	−14	−9
	West 122° long	0	−4	−5	−7	−4	−3
Pennsylvania	North 41° latitude	−3	−8	−19	−20	−21	−15
	South 41° latitude	0	−6	−13	−14	−14	−8
Rhode Island		1	−3	−12	−13	−13	−7
South Carolina		5	−1	−5	−5	−3	−2
South Dakota		−4	−14	−24	−27	−24	−18
Tennessee		1	−5	−9	−11	−9	−4
Texas	North 31° latitude	3	−6	−9	−13	−9	−7
	South 31° latitude	9	2	−2	−3	−1	2
Utah		−2	−11	−14	−18	−14	−8
Vermont		−3	−8	−20	−23	−24	−15
Virginia		2	−3	−9	−11	−9	−4
Washington	East 122° long	−2	−8	−11	−18	−11	−8
	West 122° long	0	−3	−3	−7	−4	−3
West Virginia		−3	−8	−15	−16	−14	−9
Wisconsin		−3	−14	−24	−28	−24	−18
Wyoming		−4	−15	−18	−26	−19	−16

X4.1.2 These data may be used to estimate low temperature operability requirements. In establishing low temperature operability requirements, consideration should be given to the following. These factors, or any combination, may make low temperature operability more or less severe than normal. As X4.1.2.1-X4.1.2.12 indicate, field work suggests that cloud point (or was appearance point) is a fair indication of the low temperature operability limit of fuels without cold flow additives in most vehicles.

X4.1.2.1 Long term weather patterns (Average winter low temperatures will be exceeded on occasion).

X4.1.2.2 Short term local weather conditions (Unusual cold periods do occur).

X4.1.2.3 Elevation (High locations are usually colder than surrounding lower areas).

X4.1.2.4 Specific engine design.

X4.1.2.5 Fuel system design (Recycle rate, filter location, filter capacity, filter porosity, and so forth.)

X4.1.2.6 Fuel viscosity at low temperatures

X4.1.2.7 Equipment add-ons (Engine heaters, radiator covers, fuel line and fuel filter heaters and so forth.)

X4.1.2.8 Types of operation (Extensive idling, engine shutdown, or unusual operation).

X4.1.2.9 Low temperature flow improver additives in fuel.

X4.1.2.10 Geographic area for fuel use and movement between geographical areas.

X4.1.2.11 General housekeeping (Dirt and/or water in fuel or fuel supply system).

X4.1.2.12 Impact failure for engine to start or run (Critical vs. non-critical application).

X4.1.3 *Historical Background*—A field test conducted by the CRC in 1981 resulted in two documents that provide insight into correlating laboratory tests to vehicle performance in the field. Prior to the 1981 field test, it was thought that in most cases vehicle would operate 6°C below the cloud point (or wax appearance point) due to the fuel temperature lagging behind the air temperature and the premise that more wax than would be produced at the cloud point would be necessary to block a filter or plug a fuel line. CRC Report No. 537[9] indicated that in overnight cool down, 30 % of the vehicles tested had final fuel tank temperatures within 2°C of the overnight minimum ambient temperature. CRC Report No. 528[10] concluded that of the several laboratory tests examined (cloud point, pour point, CEPP, I.TET), cloud point (see Test Method D 2500) predicted the bahavior of the untreated fuels. The combination of these reports would suggest the cloud point (or wax appearance point), in most cases, would be closer to the vehicle low temperature operability limit for untreated fuels. In addition, CRC Report No. 528 found that the Low Temperature Flow Test (LTFT), (see Test Method D 4539) provided the best correlation with vehicle performance for untreated fuel and flow improver treated fuel. Pour point, (see Test Method D 97) and Cold Filter Plugging Point (CFPP), (see Test Method IP 309) tests were not sufficiently severe enough to predict the performance of the test fuels in the diesel vehicles used in the study.

X4.1.3.1 *Current Practices*—It is recognized that fuel distributors, producers, and end users in the United States use cloud point, wax appearance point, CFPP, and LTFT to estimate vehicle low temperature operability limits for diesel fuel. No independent data has been published in recent years to determine test applicability for today's fuels and vehicles.

X4.2 Maps

X4.2.1 The maps in the following figures were derived from CCL Report No. 316, "A Predictive Study for Defining Limiting Temperatures and Their Application in Petroleum Product Specifications," by John P. Doner. This report was published by the U.S. Army Mobility Equipment Research and Development Center (USAMERDC), Coating and Chemical Laboratory, and it is available from the National Technical Information Service, Springfield, VA 22151, by requesting Publication No. AD756-420.

X4.2.2 Where states are divided the divisions are noted on the maps and table with the exception of California, which is divided by counties as follows:

California, North Coast—Alameda, Contra Costa, Del Norte, Humbolt, Lake, Marin, Mendocino, Monterey, Napa, San Benito, San Francisco, San Mateo, Santa Clara, Santa Cruz, Solano, Sonoma, Trinity.

California, Interior—Lassen, Modoc, Plumas, Sierra, Siskiyou, Alpine, Amador, Butte, Calaveras, Colusa, El Dorado, Fresno, Glenn, Kern (except that portion lying east of the Los Angeles County Aqueduct), Kings, Madera, Mariposa, Merced, Placer, Sacramento, San Joaquin, Shasta, Stanislaus, Sutter, Tehama, Tulare, Tuolumne, Yolo, Yuba, Nevada.

California, South Coast—Orange, San Diego, San Luis Obispo, Santa Barbara, Ventura, Los Angeles (except that portion north of the San Gabriel Mountain range and east of the Los Angeles County Aqueduct).

California, Southeast—Imperial, Riverside, San Bernardino, Los Angeles (that portion north of the San Gabriel Mountain range and east of the Los Angeles County Aqueduct), Mono, Inyo, Kern (that portion lying east of the Los Angeles County Aqueduct).

X4.2.3 The temperatures in CCL Report No. 316 were in degrees Fahrenheit. The degree Celsius temperatures in Appendix X4 were obtained by converting the original degree Fahrenheit temperatures.

[9] CRC Report No. 537, "The Relationship Between Vehicle Fuel Temperature and Ambient Temperature, 1981 CRC Kapuskasing Field Test", December 1983.

[10] CRC Report No. 528, "1981 CRC Diesel Fuel Low-Temperature Operability Field Test", September 1983.

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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WASHINGTON, D.C.

Designation: D 975 – 07

An American National Standard

Standard Specification for Diesel Fuel Oils[1]

This standard is issued under the fixed designation D 975; 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.

1. Scope*

1.1 This specification covers seven grades of diesel fuel oils suitable for various types of diesel engines. These grades are described as follows:

1.1.1 *Grade No. 1-D S15*—A special-purpose, light middle distillate fuel for use in diesel engine applications requiring a fuel with 15 ppm sulfur (maximum) and higher volatility than that provided by Grade No. 2-D S15 fuel.[2]

1.1.2 *Grade No. 1-D S500*—A special-purpose, light middle distillate fuel for use in diesel engine applications requiring a fuel with 500 ppm sulfur (maximum) and higher volatility than that provided by Grade No. 2-D S500 fuel.[2]

1.1.3 *Grade No. 1-D S5000*—A special-purpose, light middle distillate fuel for use in diesel engine applications requiring a fuel with 5000 ppm sulfur (maximum) and higher volatility than that provided by Grade No. 2-D S5000 fuels.

1.1.4 *Grade No. 2-D S15*—A general purpose, middle distillate fuel for use in diesel engine applications requiring a fuel with 15 ppm sulfur (maximum). It is especially suitable for use in applications with conditions of varying speed and load.[2]

1.1.5 *Grade No. 2-D S500*—A general-purpose, middle distillate fuel for use in diesel engine applications requiring a fuel with 500 ppm sulfur (maximum). It is especially suitable for use in applications with conditions of varying speed and load.[2]

1.1.6 *Grade No. 2-D S5000*—A general-purpose, middle distillate fuel for use in diesel engine applications requiring a fuel with 5000 ppm sulfur (maximum), especially in conditions of varying speed and load.

1.1.7 *Grade No. 4-D*—A heavy distillate fuel, or a blend of distillate and residual oil, for use in low- and medium-speed diesel engines in applications involving predominantly constant speed and load.

NOTE 1—A more detailed description of the grades of diesel fuel oils is given in X1.2.

NOTE 2—The Sxxx designation has been adopted to distinguish grades by sulfur rather than using words such as "Low Sulfur" as previously because the number of sulfur grades is growing and the word descriptions were thought to be not precise. S5000 grades correspond to the so-called "regular" sulfur grades, the previous No. 1-D and No. 2-D. S500 grades correspond to the previous "Low Sulfur" grades. S15 grades were not in the previous grade system and are commonly referred to as "Ultra-Low Sulfur" grades or ULSD.

1.2 This specification, unless otherwise provided by agreement between the purchaser and the supplier, prescribes the required properties of diesel fuels at the time and place of delivery.

1.2.1 Nothing in this specification shall preclude observance of federal, state, or local regulations which may be more restrictive.

NOTE 3—The generation and dissipation of static electricity can create problems in the handling of distillate diesel fuel oils. For more information on the subject, see Guide D 4865.

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

2. Referenced Documents

2.1 *ASTM Standards:* [3]

D 56 Test Method for Flash Point by Tag Closed Cup Tester

D 86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure

D 93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester

D 129 Test Method for Sulfur in Petroleum Products (General Bomb Method)

D 130 Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D 482 Test Method for Ash from Petroleum Products

[1] This specification is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.E0.02 on Diesel Fuel Oils.

Current edition approved Feb. 1, 2007. Published March 2007. Originally approved in 1948. Last previous edition approved in 2006 as D 975–06b.

[2] This fuel complies with 40 CFR Part 80—Control of Air Pollution from New Motor Vehicles: Heavy-Duty Engines and Vehicle Standards and Highway Diesel Fuel Sulfur Control Requirements: Final Rule. Regulation of Fuels and Fuel Additives: Fuel Quality Regulations for Highway Diesel Fuel Sold in 1993 and Later Calendar Years.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

***A Summary of Changes section appears at the end of this standard.**

D 524 Test Method for Ramsbottom Carbon Residue of Petroleum Products
D 613 Test Method for Cetane Number of Diesel Fuel Oil
D 1266 Test Method for Sulfur in Petroleum Products (Lamp Method)
D 1319 Test Method for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption
D 1552 Test Method for Sulfur in Petroleum Products (High-Temperature Method)
D 1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)
D 2274 Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)
D 2500 Test Method for Cloud Point of Petroleum Products
D 2622 Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry
D 2709 Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge
D 2880 Specification for Gas Turbine Fuel Oils
D 2887 Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography
D 3117 Test Method for Wax Appearance Point of Distillate Fuels
D 3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry
D 3828 Test Methods for Flash Point by Small Scale Closed Cup Tester
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D 4294 Test Method for Sulfur in Petroleum and Petroleum Products by Energy-Dispersive X-ray Fluorescence Spectrometry
D 4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination
D 4539 Test Method for Filterability of Diesel Fuels by Low-Temperature Flow Test (LTFT)
D 4737 Test Method for Calculated Cetane Index by Four Variable Equation
D 4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems
D 5453 Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence
D 5771 Test Method for Cloud Point of Petroleum Products (Optical Detection Stepped Cooling Method)
D 5772 Test Method for Cloud Point of Petroleum Products (Linear Cooling Rate Method)
D 5773 Test Method for Cloud Point of Petroleum Products (Constant Cooling Rate Method)
D 5842 Practice for Sampling and Handling of Fuels for Volatility Measurement
D 5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products
D 6078 Test Method for Evaluating Lubricity of Diesel Fuels by the Scuffing Load Ball-on-Cylinder Lubricity Evaluator (SLBOCLE)
D 6079 Test Method for Evaluating Lubricity of Diesel Fuels by the High-Frequency Reciprocating Rig (HFRR)
D 6217 Test Method for Particulate Contamination in Middle Distillate Fuels by Laboratory Filtration
D 6371 Test Method for Cold Filter Plugging Point of Diesel and Heating Fuels
D 6468 Test Method for High Temperature Stability of Distillate Fuels
D 6469 Guide for Microbial Contamination in Fuels and Fuel Systems
D 6890 Test Method for Determination of Ignition Delay and Derived Cetane Number (DCN) of Diesel Fuel Oils by Combustion in a Constant Volume Chamber
D 6898 Test Method for Evaluating Diesel Fuel Lubricity by an Injection Pump Rig

2.2 *Other Documents:*

26 CFR Part 48 Manufacturers and Realtors Excise Taxes[4]
40 CFR Part 80 Regulation of Fuels and Fuel Additives[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *S(numerical specification maximum)*—indicates the maximum sulfur content, in weight ppm (μg/g), allowed by this specification in a diesel fuel grade.

3.1.1.1 *Discussion*—Of the seven diesel fuel grades specified in this standard, six have important distinguishing maximum sulfur regulatory requirements. These are Grades No. 1-D S15, No. 1-D S500, No. 1-D S5000, No. 2-D S15, No. 2-D S500 and No. 2-D S5000. The seventh grade, No. 4-D, is distinguished from these other grades by many major properties in addition to sulfur (unregulated maximum), and therefore is not included in this designation system. Thus, Grade No. 4-D does not have the designation S20000 as part of its grade name.

4. Sampling, Containers, and Sample Handling

4.1 It is strongly advised to review all test methods prior to sampling to understand the importance and effects of sampling technique, proper containers, and special handling required for each test method.

4.2 Correct sampling procedures are critical to obtaining a representative sample of the diesel fuel oil to be tested. Refer to Appendix X2 for recommendations. The recommended procedures or practices provide techniques useful in the proper sampling or handling of diesel fuels.

5. Test Methods

5.1 The requirements enumerated in this specification shall be determined in accordance with the following methods:

5.1.1 *Flash Point*—Test Methods D 93, except where other methods are prescribed by law. For all grades, Test Method D 3828 may be used as an alternate with the same limits. For Grades No. 1-D S15, No. 1-D S500 , No. 1-D S5000, No. 2-D

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

S15, No. 2-D S500, and No. 2-D S5000, Test Method D 56 may be used as an alternate with the same limits, provided the flash point is below 93°C and the viscosity is below 5.5 mm^2/s at 40°C. This test method will give slightly lower values. In cases of dispute, Test Methods D 93 shall be used as the referee method. Test Method D 56 can not be used as the alternate method for Grade No. 4-D because its minimum viscosity limit is 5.5 mm^2/s at 40°C.

5.1.2 *Cloud Point*—Test Method D 2500. For all fuel grades in Table 1, the automatic Test Methods D 5771, D 5772, or D 5773 can be used as alternates with the same limits. Test Method D 3117 can also be used since it is closely related to Test Method D 2500. In case of dispute, Test Method D 2500 shall be the referee method.

5.1.3 *Water and Sediment*—Test Method D 2709 is used for fuel Grades No. 1-D S15, No. 1-D S500, No. 1-D S5000, No. 2-D S15, No. 2-D S500, and No. 2-D S5000. Test Method D 1796 is used for Grade No. 4-D.

5.1.4 *Carbon Residue*—Test Method D 524 is used for fuel Grades No. 1-D S15, No. 1-D S500, No. 1-D S5000, No. 2-D

TABLE 1 Detailed Requirements for Diesel Fuel Oils[A]

Property	ASTM Test Method[B]	Grade						
		No. 1-D S15	No. 1-D S500[C]	No. 1-D S5000[D]	No. 2-D S15	No. 2-D S500[C,E]	No. 2-D S5000[D,E]	No. 4-D[D]
Flash Point, °C, min.	D 93	38	38	38	52[E]	52[E]	52[E]	55
Water and Sediment, % vol, max	D 2709	0.05	0.05	0.05	0.05	0.05	0.05	...
	D 1796	...	...	...	...	...	...	0.50
Distillation: one of the following requirements shall be met:								
1. Physical Distillation	D 86							
Distillation Temperature, °C 90 % , % vol recovered								
min		...	...	...	282[E]	282[E]	282[E]	...
max		288	288	288	338	338	338	...
2. Simulated Distillation	D 2887							
Distillation Temperature, °C 90 %, % vol recovered								
min						300[E]	300[E]	
max			304	304		356	356	
Kinematic Viscosity, mm^2/S at 40°C	D 445							
min		1.3	1.3	1.3	1.9[E]	1.9[E]	1.9[E]	5.5
max	...	2.4	2.4	2.4	4.1	4.1	4.1	24.0
Ash % mass, max	D 482	0.01	0.01	0.01	0.01	0.01	0.01	0.10
Sulfur, ppm (µg/g)[F] max	D 5453	15	...	...	15	...	...	...
% mass, max	D 2622[G]	...	0.05	...	...	0.05	...	...
% mass, max	D 129	...	...	0.50	...	...	0.50	2.00
Copper strip corrosion rating max 3 h at 50°C	D 130	No. 3	No. 3	No. 3	No. 3	No. 3	No. 3	...
Cetane number, min[H]	D 613	40[I]	40[I]	40[I]	40[I]	40[I]	40[I]	30[I]
One of the following properties must be met:								
(1) Cetane index, min.	D 976-80[G]	40	40	...	40	40	...	...
(2) Aromaticity, % vol, max	D 1319[G]	35	35	...	35	35	...	...
Operability Requirements								
Cloud point, °C, max	D 2500	J	J	J	J	J	J	...
or								
LTFT/CFPP, °C, max	D 4539/ D 6371							
Ramsbottom carbon residue on 10 % distillation residue, % mass, max	D 524	0.15	0.15	0.15	0.35	0.35	0.35	...
Lubricity, HFRR @ 60°C, micron, max	D 6079	520	520	520	520	520	520	...

[A] To meet special operating conditions, modifications of individual limiting requirements may be agreed upon between purchaser, seller, and manufacturer.

[B] The test methods indicated are the approved referee methods. Other acceptable methods are indicated in 5.1.

[C] Under United States regulations, if Grades No. 1–D S500 or No. 2–D S500 are sold for tax exempt purposes then, at or beyond terminal storage tanks, they are required by 26 CFR Part 48 to contain the dye Solvent Red 164 at a concentration spectrally equivalent to 3.9 lb per thousand barrels of the solid dye standard Solvent Red 26, or the tax must be collected.

[D] Under United States regulations, Grades No.1–D S5000, No. 2–D S5000, and No. 4–D are required by 40 CFR Part 80 to contain a sufficient amount of the dye Solvent Red 164 so its presence is visually apparent. At or beyond terminal storage tanks, they are required by 26 CFR Part 48 to contain the dye Solvent Red 164 at a concentration spectrally equivalent to 3.9 lb per thousand barrels of the solid dye standard Solvent Red 26.

[E] When a cloud point less than –12°C is specified, as can occur during cold months, it is permitted and normal blending practice to combine Grades No. 1 and No. 2 to meet the low temperature requirements. In that case, the minimum flash point shall be 38°C, the minimum viscosity at 40°C shall be 1.7 mm^2/s, and the minimum 90 % recovered temperature shall be waived.

[F] Other sulfur limits can apply in selected areas in the United States and in other countries.

[G] These test methods are specified in 40 CFR Part 80.

[H] Where cetane number by Test Method D 613 is not available, Test Method D 4737 can be used as an approximation.

[I] Low ambient temperatures as well as engine operation at high altitudes may require the use of fuels with higher cetane ratings.

[J] It is unrealistic to specify low temperature properties that will ensure satisfactory operation at all ambient conditions. In general, cloud point (or wax appearance point) Low Temperature Flow Test, and Cold Filter Plugging Point Test may be used as an estimate of operating temperature limits for Grades No. 1–D S500; No. 2–D S500; and No. 1–D S5000 and No. 2–D S5000 diesel fuel oils. However, satisfactory operation below the cloud point (or wax appearance point) may be achieved depending on equipment design, operating conditions, and the use of flow-improver additives as described in X5.1.2. Appropriate low temperature operability properties should be agreed upon between the fuel supplier and purchaser for the intended use and expected ambient temperatures. Test Methods D 4539 and D 6371 may be especially useful to estimate vehicle low temperature operability limits when flow improvers are used. Due to fuel delivery system, engine design, and test method differences, low temperature operability tests may not provide the same degree of protection in various vehicle operating classes. Tenth percentile minimum air temperatures for U.S. locations are provided in Appendix X5 as a means of estimating expected regional temperatures. The tenth percentile minimum air temperatures may be used to estimate expected regional target temperatures for use with Test Methods D 2500, D 4539, and D 6371. Refer to X5.1.3 for further general guidance on test application.

S15, No. 2-D S500 and No. 2-D S5000. Grade No. 4-D does not have a limit for carbon residue.

5.1.5 *Ash*—Test Method D 482 is used for all grades in Table 1.

5.1.6 *Distillation*—Test Method D 86 is used for Grades No. 1-D S15, No. 1-D S500, No. 1-D S5000, No. 2-D S15, No. 2-D S500 and No. 2-D S5000. For all grades, Test Method D 2887 can be used as an alternate with the limits listed in Table 1. In case of dispute, Test Method D 86 shall be the referee method. Grade No. 4-D does not have distillation requirements.

5.1.7 *Viscosity*—Test Method D 445 is used for all fuel grades in Table 1.

5.1.8 *Sulfur*—The following list shows the referee test methods and alternate test methods for sulfur, the range over which each test method applies and the corresponding fuel grades.

Sulfur Test Method	Range	Grades
D 129 (referee)	>0.1 mass %	No. 1-D S5000, No. 2-D S5000, No. 4-D
D 1266	0.0005 to 0.4 mass % 5 to 4000 mg/kg (wt ppm)	No. 1-D S500, No. 2-D S500
D 1552	>0.06 mass %	No. 1- D S5000, No. 2-D S5000, No. 4-D
D 2622 (referee for S500 Grades)	0.0003 to 5.3 mass % 3 to 53 000 mg/kg (wt ppm)	All Grades
D 3120	3.0 to 100 mg/kg (wt ppm)	No. 1-D S15, No. 2-D S15 No. 1-D S500, No. 2-D S500 (S500 grades must be diluted before testing)
D 4294	0.0150 to 5.00 mass % 150 to 50 000 mg/kg (wt ppm)	No. 1- D S5000, No. 2-D S5000, No. 4-D
D 5453 (referee for S15 grades)	0.0001 to 0.8 mass % 1.0 to 8000 mg/kg (wt ppm)	All Grades

NOTE 4—The units used to report results in the above test methods are:

D 129	mass %
D 1266	mass %
D 1552	mass %
D 2622	mass %
D 3120	ppm (μg/g)
D 4294	mass %
D 5453	ppm (μg/g)

Results reported in mg/kg and in ppm (μg/g) are numerically the same. The units used in Table 1 for the sulfur requirements are the units in which results for the referee test are reported.

5.1.9 *Copper Corrosion*—Test Method D 130, 3 h test at 50°C. This test method is used for fuel Grades No. 1-D S15, No. 1-D S500, No. 1-D S5000, No. 2-D S15, No. 2-D S500 and No. 2-D S5000. Grade No. 4-D does not have a copper corrosion requirement.

5.1.10 *Cetane Number*—Test Method D 613 is used for all fuel grades in Table 1. Test Method D 6890 is used for all No. 1-D and No. 2-D grades with the DCN result being compared to the cetane number specification requirement of 40. Test Method D 613 shall be the referee method.

5.1.11 *Cetane Index*—Test Methods D 976–80 is used for fuel Grades No. 1-D S15, No. 1-D S500, No. 2-D S15 and No. 2-D S500. Grades No. 1-D S5000, No. 2-D S5000 and No. 4-D do not have an aromatics content requirement, so do not use this test method as a surrogate for aromatics content.

5.1.12 *Aromaticity*—Test Method D 1319. This test method provides an indication of the aromatics content of fuels. For fuels with a maximum final boiling point of 315°C, this method is a measurement of the aromatic content of the fuel. This test method is used for fuel Grades No. 1-D S15, No. 1-D S500, No. 2-D S15 and No. 2-D S500. Grades No. 1-D S5000, No. 2-D S5000 and No. 4-D do not have an aromatics content requirement.

5.1.13 *Lubricity*—Test Method D 6079.

6. Workmanship

6.1 The diesel fuel shall be visually free of undissolved water, sediment, and suspended matter.

7. Requirements

7.1 The grades of diesel fuel oils herein specified shall be hydrocarbon oils conforming to the detailed requirements shown in Table 1.

7.2 *Grades No. 2-D S15, No. 2-D S500 and No. 2–D S5000*—When a cloud point less than −12°C is specified, as can occur during cold months, it is permitted and normal blending practice to combine Grades No. 1 and No. 2 to meet the low temperature requirements. In that case, the minimum flash point shall be 38°C, the minimum viscosity at 40°C shall be 1.7 mm^2/s, and the minimum 90 % recovered temperature shall be waived.

8. Keywords

8.1 diesel; fuel oil; petroleum and petroleum products

APPENDIXES

(Nonmandatory Information)

X1. SIGNIFICANCE OF ASTM SPECIFICATION FOR DIESEL FUEL OILS

X1.1 Introduction

X1.1.1 The properties of commercial fuel oils depend on the refining practices employed and the nature of the crude oils from which they are produced. Distillate fuel oils, for example, may be produced within the boiling range of 150 and 400°C having many possible combinations of various properties, such as volatility, ignition quality, viscosity, and other characteristics.

X1.2 Grades

X1.2.1 This specification is intended as a statement of permissible limits of significant fuel properties used for specifying the wide variety of commercially available diesel fuel oils. Limiting values of significant properties are prescribed for seven grades of diesel fuel oils. These grades and their general applicability for use in diesel engines are broadly indicated as follows:

X1.2.2 *Grade No. 1-D S15*—Grade No. 1-D S15 comprises the class of very low sulfur, volatile fuel oils from kerosine to the intermediate middle distillates. Fuels within this grade are applicable for use in *(1)* high-speed diesel engines and diesel engine applications that require ultra-low sulfur fuels, *(2)* applications necessitating frequent and relatively wide variations in loads and speeds, and *(3)* applications where abnormally low operating temperatures are encountered.

X1.2.3 *Grade No. 1-D S500*—Grade No. 1-D S500 comprises the class of low-sulfur, volatile fuel oils from kerosine to the intermediate middle distillates. Fuels within this grade are applicable for use in *(1)* high-speed diesel engines that require low sulfur fuels, *(2)* in applications necessitating frequent and relatively wide variations in loads and speeds, and *(3)* in applications where abnormally low operating temperatures are encountered.

X1.2.4 *Grade No. 1-D S5000*—Grade No. 1-D S5000 comprises the class of volatile fuel oils from kerosine to the intermediate middle distillates. Fuels within this grade are applicable for use in high-speed diesel engines applications necessitating frequent and relatively wide variations in loads and speeds, and also for use in cases where abnormally low operating temperatures are encountered.

X1.2.5 *Grade No. 2-D S15*—Grade No. 2-D S15 includes the class of very low sulfur, middle distillate gas oils of lower volatility than Grade No. 1-D S15. These fuels are applicable for use in *(1)* high speed diesel engines and diesel engine applications that require ultra-low sulfur fuels, *(2)* applications necessitating relatively high loads and uniform speeds, or *(3)* diesel engines not requiring fuels having higher volatility or other properties specified in Grade No. 1-D S15.

X1.2.6 *Grade No. 2-D S500*—Grade No. 2-D S500 includes the class of low-sulfur, middle distillate gas oils of lower volatility than Grade No. 1-D S500. These fuels are applicable for use in *(1)* high-speed diesel engine applications that require low sulfur fuels, *(2)* applications necessitating relatively high loads and uniform speeds, or *(3)* diesel engines not requiring fuels having higher volatility or other properties specified for Grade No. 1-D S500.

X1.2.7 *Grade No. 2-D S5000*—Grade No. 2-D S5000 includes the class of middle distillate gas oils of lower volatility than Grade No. 1-D S5000. These fuels are applicable for use in *(1)* high-speed diesel engines in applications necessitating relatively high loads and uniform speeds, or *(2)* in diesel engines not requiring fuels having higher volatility or other properties specified for Grade No. 1-D S5000.

X1.2.8 *Grade No. 4-D*—Grade No. 4-D comprises the class of more viscous middle distillates and blends of these middle distillates with residual fuel oils. Fuels within this grade are applicable for use in low- and medium-speed diesel engines in applications necessitating sustained loads at substantially constant speed.

X1.3 Selection of Particular Grade

X1.3.1 The selection of a particular diesel fuel oil from one of these seven ASTM grades for use in a given engine requires consideration of the following factors:

X1.3.1.1 Fuel price and availability,

X1.3.1.2 Maintenance considerations,

X1.3.1.3 Engine size and design,

X1.3.1.4 Emission control systems,

X1.3.1.5 Speed and load ranges,

X1.3.1.6 Frequency of speed and load changes, and

X1.3.1.7 Atmospheric conditions. Some of these factors can influence the required fuel properties outlined as follows:

X1.4 Cetane Number

X1.4.1 Cetane number is a measure of the ignition quality of the fuel and influences combustion roughness. The cetane number requirements depend on engine design, size, nature of speed and load variations, and on starting and atmospheric conditions. Increase in cetane number over values actually required does not materially improve engine performance. Accordingly, the cetane number specified should be as low as possible to assure maximum fuel availability.

X1.5 Distillation

X1.5.1 The fuel volatility requirements depend on engine design, size, nature of speed and load variations, and starting and atmospheric conditions. For engines in services involving rapidly fluctuating loads and speeds as in bus and truck operation, the more volatile fuels may provide best performance, particularly with respect to smoke and odor. However, best fuel economy is generally obtained from the heavier types of fuels because of their higher heat content.

X1.6 Viscosity

X1.6.1 For some engines it is advantageous to specify a minimum viscosity because of power loss due to injection pump and injector leakage. Maximum viscosity, on the other hand, is limited by considerations involved in engine design and size, and the characteristics of the injection system.

X1.7 Carbon Residue

X1.7.1 Carbon residue gives a measure of the carbon depositing tendencies of a fuel oil when heated in a bulb under prescribed conditions. While not directly correlating with engine deposits, this property is considered an approximation.

X1.8 Sulfur

X1.8.1 The effect of sulfur content on engine wear and deposits appears to vary considerably in importance and depends largely on operating conditions. Fuel sulfur can affect emission control systems performance. To assure maximum availability of fuels, the permissible sulfur content should be specified as high as is practicable, consistent with maintenance considerations.

X1.9 Flash Point

X1.9.1 The flash point as specified is not directly related to engine performance. It is, however, of importance in connection with legal requirements and safety precautions involved in fuel handling and storage, and is normally specified to meet insurance and fire regulations.

X1.10 Cloud Point

X1.10.1 Cloud point is of importance in that it defines the temperature at which a cloud or haze of wax crystals appears in the oil under prescribed test conditions which generally relates to the temperature at which wax crystals begin to precipitate from the oil in use.

X1.11 Ash

X1.11.1 Ash-forming materials may be present in fuel oil in two forms: *(1)* abrasive solids, and *(2)* soluble metallic soaps. Abrasive solids contribute to injector, fuel pump, piston and ring wear, and also to engine deposits. Soluble metallic soaps have little effect on wear but may contribute to engine deposits.

X1.12 Copper Strip Corrosion

X1.12.1 This test serves as a measure of possible difficulties with copper and brass or bronze parts of the fuel system.

X1.13 Aromaticity

X1.13.1 This test is used as an indication of the aromatics content of diesel fuel. Aromatics content is specified to prevent an increase in the average aromatics content in Grades No. 1-D S15, No. 1-D S500, No. 2-D S15 and No. 2-D S500 fuels and is required by 40 CFR Part 80. Increases in aromatics content of fuels over current levels may have a negative impact on emissions.

X1.14 Cetane Index

X1.14.1 Cetane Index is specified as a limitation on the amount of high aromatic components in Grades No. 1-D S15, No. 1-D S500, No. 2-D S15 and No. 2-D S500.

X1.15 Other

X1.15.1 *Microbial Contamination*—Refer to Guide D 6469 for a discussion of this form of contamination.

X2. SAMPLING, CONTAINERS AND SAMPLE HANDLING

X2.1 Introduction

X2.1.1 This appendix provides guidance on methods and techniques for the proper sampling of diesel fuel oils. As diesel fuel oil specifications become more stringent and contaminants and impurities become more tightly controlled, even greater care needs to be taken in collecting and storing samples for quality assessment.

X2.2 Sampling, Containers and Sample Handling Recommendations

X2.2.1 Appropriate manual method sampling procedures can be found in Practice D 4057 and automatic method sampling is covered in Practice D 4177.

X2.2.2 The correct sample volume and appropriate container selection are also important decisions that can impact test results. Practice D 4306 for aviation fuel container selection for tests sensitive to trace contamination may be useful. Practice D 5854 for procedures on container selection and sample mixing and handling is recommended. For cetane number determination protection from light is important. Collection and storage of diesel fuel oil samples in an opaque container, such as a dark brown glass bottle, metal can, or a minimally reactive plastic container to minimize exposure to UV emissions from sources such as sunlight or fluorescent lamps, is recommended. According to Paragraph 8.2 of Test Method D 6079, "Because of sensitivity of lubricity measurements to trace materials, sample containers shall be only fully epoxy-lined metal, amber borosilicate glass, or polytetrafluoroethylene as specified in Practice D 4306."

X2.2.3 For volatility determination of a sample, Practice D 5842 for special precautions recommended for representative sampling and handling techniques may be appropriate.

X3. STORAGE AND THERMAL STABILITY OF DIESEL FUELS

X3.1 Scope

X3.1.1 This appendix provides guidance for consumers of diesel fuels who may wish to store quantities of fuels for extended periods or use the fuel in severe service or high temperature applications. Fuels containing residual components are excluded. Consistently successful long-term fuel storage or use in severe applications requires attention to fuel selection, storage conditions, handling and monitoring of properties during storage and prior to use.

X3.1.2 Normally produced fuels have adequate stability properties to withstand normal storage and use without the formation of troublesome amounts of insoluble degradation products. Fuels that are to be stored for prolonged periods or used in severe applications should be selected to avoid formation of sediments or gums, which can overload filters or plug injectors. Selection of these fuels should result from supplier-user discussions.

X3.1.3 These suggested practices are general in nature and should not be considered substitutes for any requirements imposed by the warranty of the distillate fuel equipment manufacturer or by federal, state, or local government regulations. Although they cannot replace a knowledge of local conditions or good engineering and scientific judgment, these suggested practices do provide guidance in developing an individual fuel management system for the middle distillate fuel user. They include suggestions in the operation and maintenance of existing fuel storage and handling facilities and for identifying where, when, and how fuel quality should be monitored or selected for storage or severe use.

X3.2 Definitions

X3.2.1 *bulk fuel*—fuel in the storage facility.

X3.2.2 *fuel contaminants*—foreign materials that make fuel less suitable or unsuitable for the intended use.

X3.2.2.1 *Discussion*—Fuel contaminants include materials introduced subsequent to the manufacture of fuel and fuel degradation products.

X3.2.3 *fuel-degradation products*—those materials that are formed in fuel during extended storage or exposure to high temperatures.

X3.2.3.1 *Discussion*—Insoluble degradation products may combine with other fuel contaminants to reinforce deleterious effects. Soluble degradation products (soluble gums) are less volatile than fuel and may carbonize to form deposits due to complex interactions and oxidation of small amounts of olefinic or sulfur-, oxygen- or nitrogen-containing compounds present in fuels. The formation of degradation products may be catalyzed by dissolved metals, especially copper salts. When dissolved copper is present it can be deactivated with metal deactivator additives.

X3.2.4 *long-term storage*—storage of fuel for longer than 12 months after it is received by the user.

X3.2.5 *severe use*—use of the fuel in applications which may result in engines operating under high load conditions that may cause the fuel to be exposed to excessive heat.

X3.3 Fuel Selection

X3.3.1 Certain distilled refinery products are generally more suitable for long-term storage and severe service than others. The stability properties of middle distillates are highly dependent on the crude oil sources, severity of processing, use of additives and whether additional refinery treatment has been carried out.

X3.3.2 The composition and stability properties of middle distillate fuels produced at specific refineries may be different. Any special requirements of the user, such as long-term storage or severe service, should be discussed with the supplier.

X3.3.3 Blends of fuels from various sources may interact to give stability properties worse than expected based on the characteristics of the individual fuels.

X3.4 Fuel Additives

X3.4.1 Available fuel additives can improve the suitability of marginal fuels for long-term storage and thermal stability, but may be unsuccessful for fuels with markedly poor stability properties. Most additives should be added at the refinery or during the early weeks of storage to obtain maximum benefits.

X3.4.2 Biocides or biostats destroy or inhibit the growth of fungi and bacteria, which can grow at fuel-water interfaces to give high particulate concentrations in the fuel. Available biocides are soluble in both the fuel and water or in the water phase only.

X3.5 Tests for Fuel Quality

X3.5.1 At the time of manufacture, the storage stability of fuel may be assessed using Test Method D 2274 or D 5304. However, these accelerated stability tests may not correlate well with field storage stability due to varying field conditions and to fuel composition.

X3.5.2 Performance criteria for accelerated stability tests that assure satisfactory long-term storage of fuels have not been established.

X3.5.3 Test Method D 6468, provides an indication of thermal oxidative stability of middle distillate fuels when heated to temperatures near 150°C.

X3.6 Fuel Monitoring

X3.6.1 A plan for monitoring the quality of bulk fuel during prolonged storage is an integral part of a successful program. A plan to replace aged fuel with fresh product at established intervals is also desirable.

X3.6.2 Stored fuel should be periodically sampled and its quality assessed. Practice D 4057 provides guidance for sampling. Fuel contaminants and degradation products will usually settle to the bottom of a quiescent tank. A "Bottom" or "Clearance" sample, as defined in Practice D 4057, should be included in the evaluation along with an "All Level" sample.

X3.6.3 The quantity of insoluble fuel contaminants present in fuel can be determined using Test Method D 6217.

X3.6.4 Test Method D 6468, can be used for investigation of operational problems that might be related to fuel thermal

stability. Testing samples from the fuel tank or from bulk storage may give an indication as to the cause of filter plugging. It is more difficult to monitor the quality of fuels in vehicle tanks since operation may be on fuels from multiple sources.

X3.6.5 Some additives exhibit effects on fuels tested in accordance with Test Method D 6468 that may or may not be observed in the field. Data have not been developed that correlate results from the test method for various engine types and levels of operating severity.

X3.7 Fuel Storage Conditions

X3.7.1 Contamination levels in fuel can be reduced by storage in tanks kept free of water, and tankage should have provisions for water draining on a scheduled basis. Water promotes corrosion, and microbiological growth may occur at a fuel-water interface. Underground storage is preferred to avoid temperature extremes; above-ground storage tanks should be sheltered or painted with reflective paint. High storage temperatures accelerate fuel degradation. Fixed roof tanks should be kept full to limit oxygen supply and tank breathing.

X3.7.2 Copper and copper-containing alloys should be avoided. Copper can promote fuel degradation and may produce mercaptide gels. Zinc coatings can react with water or organic acids in the fuel to form gels that rapidly plug filters.

X3.7.3 Appendix X2 of Specification D 2880 discusses fuel contaminants as a general topic.

X3.8 Fuel Use Conditions

X3.8.1 Many diesel engines are designed so that the diesel fuel is used for heat transfer. In modern heavy-duty diesel engines, for example, only a portion of the fuel that is circulated to the fuel injectors is actually delivered to the combustion chamber. The remainder of the fuel is circulated back to the fuel tank, carrying heat with it. Thus adequate high temperature stability can be a necessary requirement in some severe applications or types of service.

X3.8.2 Inadequate high temperature stability may result in the formation of insoluble degradation products.

X3.9 Use of Degraded Fuels

X3.9.1 Fuels that have undergone mild-to-moderate degradation can often be consumed in a normal way, depending on the fuel system requirements. Filters and other cleanup equipment can require special attention and increased maintenance. Burner nozzle or injector fouling can occur more rapidly.

X3.9.2 Fuels containing very large quantities of fuel degradation products and other contaminants or with runaway microbiological growth require special attention. Consultation with experts in this area is desirable. It can be possible to drain the sediment or draw off most of the fuel above the sediment layer and use it with the precautions described in X3.9.1. However, very high soluble gum levels or corrosion products from microbiological contamination can cause severe operational problems.

X3.10 Thermal Stability Guidelines

X3.10.1 Results from truck fleet experience suggests that Test Method D 6468 can be used to qualitatively indicate whether diesel fuels have satisfactory thermal stability performance properties.[5,6]

X3.10.2 Performance in engines has not been sufficiently correlated with results from Test Method D 6468 to provide definitive specification requirements. However, the following guidelines are suggested.

X3.10.2.1 Fuels giving a Test Method D 6468 reflectance value of 70 % or more in a 90 minute test at the time of manufacture should give satisfactory performance in normal use.

X3.10.2.2 Fuels giving a Test Method D 6468 reflectance value of 80 % or more in a 180 minute test at the time of manufacture should give satisfactory performance in severe use.

X3.10.3 Thermal stability as determined by Test Method D 6468 is known to degrade during storage.[7] The guidance above is for fuels used within six months of manufacture.

[5] Bacha, John D., and Lesnini, David G., "Diesel Fuel Thermal Stability at 300°F," *Proceedings of the 6th International Conference on Stability and Handling of Liquid Fuels*, Vancouver, B.C., October 1997.

[6] Schwab, Scott D., Henly, Timothy J., Moxley, Joel F., and Miller, Keith, "Thermal Stability of Diesel Fuel," *Proceedings of the 7th International Conference on Stability and Handling of Liquid Fuels* , Graz, Austria September 2000.

[7] Henry, C. P., "The DuPont F21 149°C (300°F) Accelerated Stability Test," *Distillate Fuel Stability and Cleanliness, ASTM STP 751*, 1981, pp. 22-33.

X4. DIESEL FUEL LUBRICITY

X4.1 Introduction

X4.1.1 Diesel fuel functions as a lubricant in most components of fuel injection equipment such as pumps and injectors. In limited cases, fuel with specific properties will have insufficient lubricating properties which will lead to a reduction in the normal service life and functional performance of diesel fuel injection systems.

X4.2 Fuel Characteristics Affecting Equipment Wear

X4.2.1 Currently, two fuel characteristics affect equipment wear. These are low viscosity and lack of sufficient quantities of trace components that have an affinity for surfaces. If fuel viscosity meets the requirements of a particular engine, a fuel film is maintained between the moving surfaces of the fuel system components. This prevents excessive metal-to-metal

contact and avoids premature failure due to wear. Similarly, certain surface active molecules in the fuel adhere to, or combine with, surfaces to produce a protective film which also can protect surfaces against excessive wear.

X4.3 Fuel Lubricity

X4.3.1 The concern about fuel lubricity is limited to situations in which fuels with lower viscosities than those specified for a particular engine are used or in which fuels that have been processed in a manner that results in severe reduction of the trace levels of the surface active species that act as surface protecting agents. Presently the only fuels of the latter type shown to have lubricity problems resulted from sufficiently severe processing to reduce aromatics or sulfur.

X4.3.2 Work in the area of diesel fuel lubricity is ongoing by several organizations, such as the International Organization for Standardization (ISO), the ASTM Diesel Fuel Lubricity Task Force, and the Coordinating Research Council (CRC) Diesel Performance Group. These groups include representatives from the fuel injection equipment manufacturers, fuel producers, and additive suppliers. The charge of the ASTM task force has been the recommendation of test methods and fuel lubricity requirements for Specification D 975. Two test methods were proposed and approved. These are Test Method D 6078, a scuffing load ball-on-cylinder lubricity evaluator method, SLBOCLE, and Test Method D 6079, a high frequency reciprocating rig (HFRR) method. Use of these tests raises three issues: 1) The correlation of the data among the two test methods and the fuel injection equipment is not perfect, 2) Both methods in their current form do not apply to all fuel-additive combinations, and 3) The reproducibility values for both test methods are large. In order to protect diesel fuel injection equipment, an HFRR Wear Scar Diameter (WSD) of 520 microns has been placed in Specification D 975.[8]

X4.3.3 Most experts agree that fuels having a SLBOCLE lubricity value below 2000 g might not prevent excessive wear in injection equipment[9] while fuels with values above 3100 g should provide sufficient lubricity in all cases.[10] Experts also agree that if HFFR test at 60°C is used, fuels with values above 600 microns might not prevent excessive wear,[11] while fuels with values below 450 microns should provide sufficient lubricity in all cases.[10] More accurately, an industry-accepted long-term durability pump test, such as Test Method D 6898, can be used to evaluate the lubricity of a diesel fuel. A poor result in such a test indicates that the fuel has low lubricity and may not be able to provide sufficient protection.

NOTE X4.1—Some injection equipment can be fitted with special components that can tolerate low lubricity fuels.

[8] Mitchell, K., "Diesel Fuel Lubricity—Base Fuel Effects," SAE Technical Paper 2001-01-1928, 2001.

[9] Westbrook, S. R., "Survey of Low Sulfur Diesel Fuels and Aviation Kerosenes from U.S. Military Installations," SAE Technical Paper 952369, 1995.

[10] Nikanjam, M., "ISO Diesel Fuel Lubricity Round Robin Program," SAE Technical Paper 952372, 1995.

[11] Nikanjam, M., "*Diesel Fuel Lubricity: On the Path to Specifications*," SAE Technical Paper 1999-01-1479, 1999.

X5. TENTH PERCENTILE MINIMUM AMBIENT AIR TEMPERATURES FOR THE UNITED STATES (EXCEPT HAWAII)

X5.1 Introduction

X5.1.1 The tenth percentile minimum ambient air temperatures shown on the following maps (Figs. X5.1-X5.12) and in Table X5.1 were derived from an analysis of historical hourly temperature readings recorded over a period of 15 to 21 years from 345 weather stations in the United States. This study was conducted by the U.S. Army Mobility Equipment Research and Development Center (USAMERDC), Coating and Chemical Laboratory, Aberdeen Proving Ground, MD 21005. The tenth percentile minimum ambient air temperature is defined as the lowest ambient air temperature which will not go lower on average more than 10 % of the time. In other words, the daily minimum ambient air temperature would on average not be expected to go below the monthly tenth percentile minimum ambient air temperature more than 3 days for a 30-day month. See Table X5.1.

X5.1.2 These data may be used to estimate low temperature operability requirements. In establishing low temperature operability requirements, consideration should be given to the following. These factors, or any combination, may make low temperature operability more or less severe than normal. As X5.1.2.1 through X5.1.2.12 indicate, field work suggests that cloud point (or wax appearance point) is a fair indication of the low temperature operability limit of fuels without cold flow additives in most vehicles.

X5.1.2.1 Long term weather patterns (Average winter low temperatures will be exceeded on occasion).

X5.1.2.2 Short term local weather conditions (Unusual cold periods do occur).

X5.1.2.3 Elevation (High locations are usually colder than surrounding lower areas).

X5.1.2.4 Specific engine design.

X5.1.2.5 Fuel system design (Recycle rate, filter location, filter capacity, filter porosity, and so forth.)

X5.1.2.6 Fuel viscosity at low temperatures

X5.1.2.7 Equipment add-ons (Engine heaters, radiator covers, fuel line and fuel filter heaters and so forth.)

X5.1.2.8 Types of operation (Extensive idling, engine shutdown, or unusual operation).

X5.1.2.9 Low temperature flow improver additives in fuel.

X5.1.2.10 Geographic area for fuel use and movement between geographical areas.

X5.1.2.11 General housekeeping (Dirt and/or water in fuel or fuel supply system).

X5.1.2.12 Impact failure for engine to start or run (Critical vs. non-critical application).

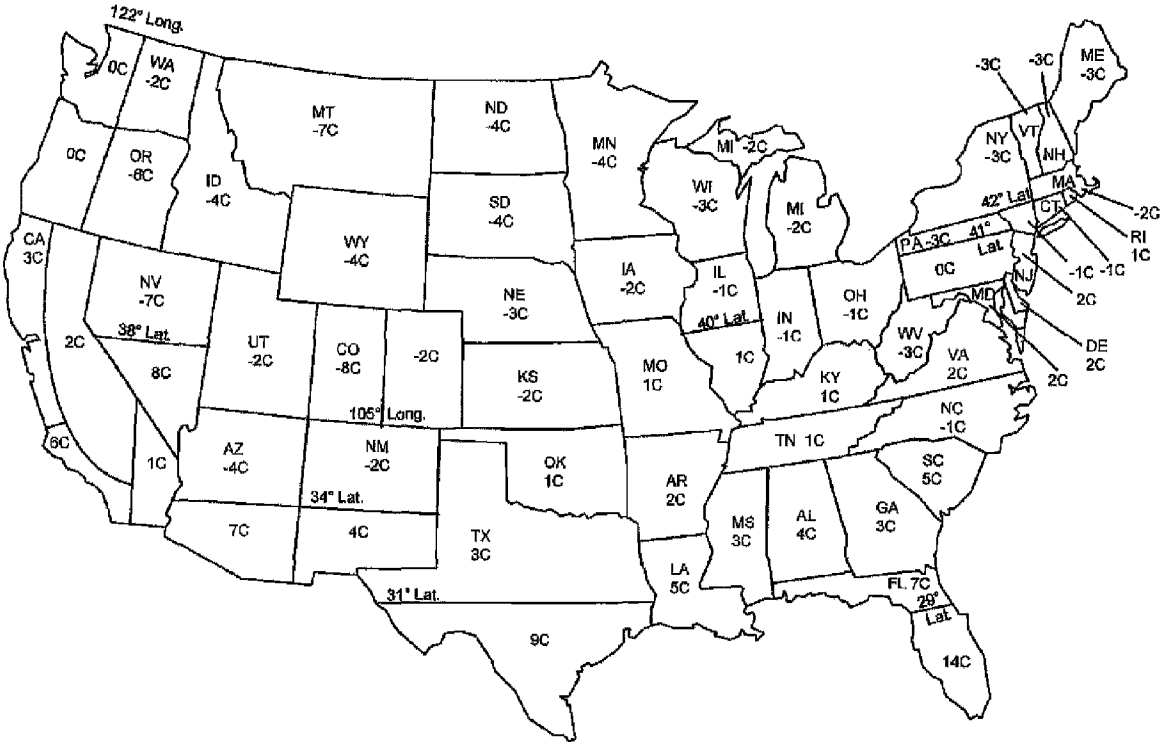

FIG. X5.1 October—10th Percentile Minimum Temperatures

X5.1.3 *Historical Background*—Three test methods have been widely used to estimate or correlate with low temperature vehicle operability. Cloud point, Test Method D 2500, is the oldest of the three and most conservative of the tests. The cloud point test indicates the earliest appearance of wax precipitation that might result in plugging of fuel filters or fuel lines under prescribed cooling conditions. Although not 100 % failsafe, it is the most appropriate test for applications that can not tolerate much risk. The Cold Filter Plugging Point (CFPP) test, Test Method D 6371, was introduced in Europe in 1965. The CFPP was designed to correlate with the majority of European vehicles. Under rapid cooling conditions, 20 cc fuel is drawn through a 45 micron screen then allowed to flow back through the screen for further cooling. This process is continued every 1°C until either the 20 cc fuel fails to be drawn through the screen in 60 s or it fails to return through the screen in 60 s. It was field tested many times in Europe[12] before being widely accepted as a European specification. Field tests have also shown CFPP results more than 10°C below the cloud point should be viewed with caution because those results did not necessarily reflect the true vehicle low temperature operability limits.[13] CFPP has been applied to many areas of the world where similar vehicle designs are used. The Low Temperature Flow Test (LTFT), Test Method D 4539, was designed to correlate with the most severe and one of the most common fuel delivery systems used in North American Heavy Duty trucks. Under prescribed slow cool conditions (1°C/h), similar to typical field conditions, several 200 cc fuel specimens in glass containers fitted with 17 µm screen assemblies are cooled. At 1°C intervals one specimen is drawn through the screen under a 20 kPa vacuum. Approximately 90 % of the fuel must come over in 60 s or less for the result to be a pass. This process is continued at lower temperatures (1°C increments) until the fuel fails to come over in the allotted 60 s. The lowest passing temperature is defined as the LTFT for that fuel. In 1981, a CRC program was conducted to evaluate the efficacy of cloud point, CFPP, pour point, and LTFT for protecting the diesel vehicle population in North America and to determine what benefit flow-improvers could provide. The field test consisted of 3 non-flow improved diesel fuels, 5 flow improved diesel fuels, 4 light-duty passenger cars, and 3 heavy-duty trucks. The field trial resulted in two documents[14,15] that provide insight into correlating laboratory tests to North

[12] "Low Temperature Operability of Diesels. A Report by CEC Investigation Group IGF-3," CEC P-171–82.

[13] "SFPP-A New Laboratory Test for Assessment of Low Temperature Operability of Modern Diesel Fuels," CEC/93/EF 15, 5–7, May 1993.

[14] CRC Report No. 537, "The Relationship Between Vehicle Fuel Temperature and Ambient Temperature, 1981 CRC Kapuskasing Field Test," December 1983.

[15] CRC Report No. 528, "1981 CRC Diesel Fuel Low-Temperature Operability Field Test," September 1983.

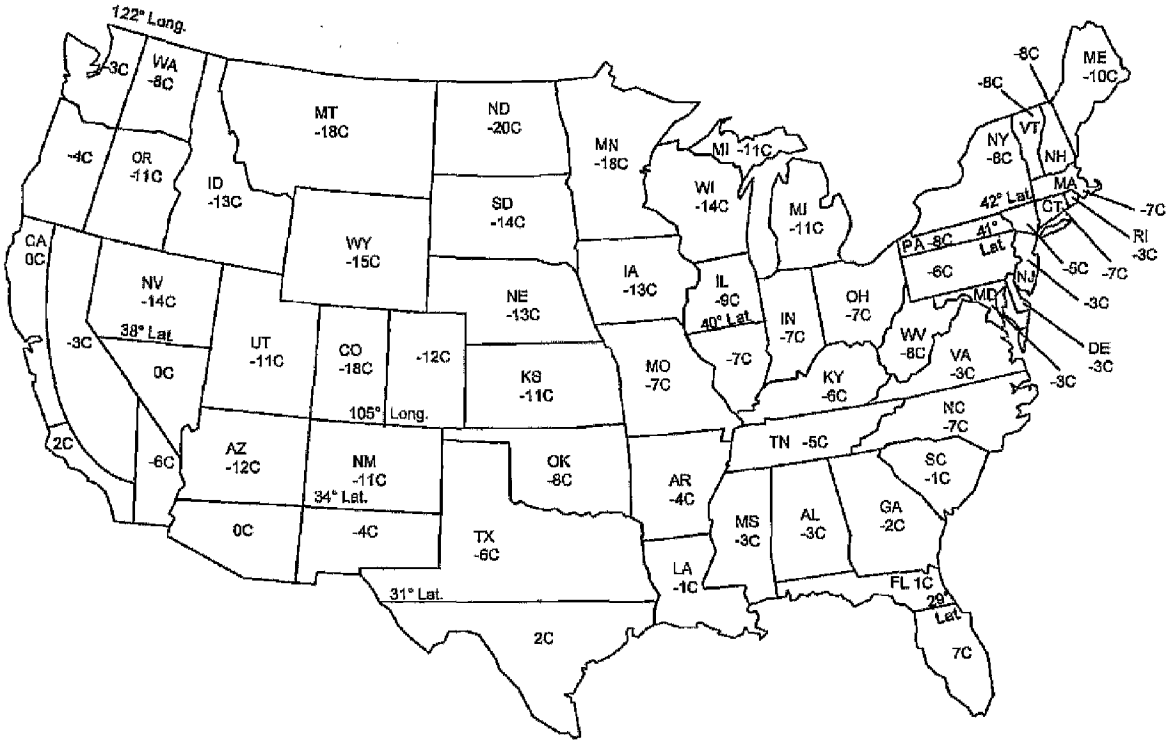

FIG. X5.2 November—10th Percentile Minimum Ambient Air Temperatures

American vehicle performance in the field. The general conclusions of the study were:

(1) In overnight cool down, 30 % of the vehicles tested had a final fuel tank temperature within 2°C of the overnight minimum ambient temperature.

(2) The use of flow-improved diesel fuel permits some vehicles to operate well below the fuel cloud point.

(3) Significant differences exist in the severity of diesel vehicles in terms of low temperature operation.

(4) No single laboratory test was found that adequately predicts the performance of all fuels in all vehicles.

(5) CFPP was a better predictor than pour point, but both methods over-predicted, minimum operating temperatures in many vehicles. For this reason, these tests were judged inadequate predictors of low-temperature performance and dismissed from further consideration.

(6) Cloud point and LTFT showed varying degrees of predictive capability, and offered distinctively different advantages. Both predicted the performance of the base fuels well, but LTFT more accurately predicted the performance of the flow-improved fuels. On the other hand, cloud point came closest to a fail-safe predictor of vehicle performance for all vehicles.

Since the 1981 field test, non-independent studies[16] using newer vehicles verified the suitability of the LTFT for North American heavy-duty trucks. Users are advised to review these and any more recent publications when establishing low temperature operability requirements and deciding upon test methods.

X5.1.3.1 *Current Practices*—It is recognized that fuel distributors, producers, and end users in the United States use cloud point, wax appearance point, CFPP, and LTFT to estimate vehicle low temperature operability limits for diesel fuel. No independent data has been published in recent years to determine test applicability for today's fuels and vehicles.

X5.2 Maps

X5.2.1 The maps in the following figures were derived from CCL Report No. 316, "A Predictive Study for Defining Limiting Temperatures and Their Application in Petroleum Product Specifications," by John P. Doner. This report was published by the U.S. Army Mobility Equipment Research and Development Center (USAMERDC), Coating and Chemical Laboratory, and it is available from the National Technical

[16] SAE 962197, SAE 982576, SAE 2000-01-2883.

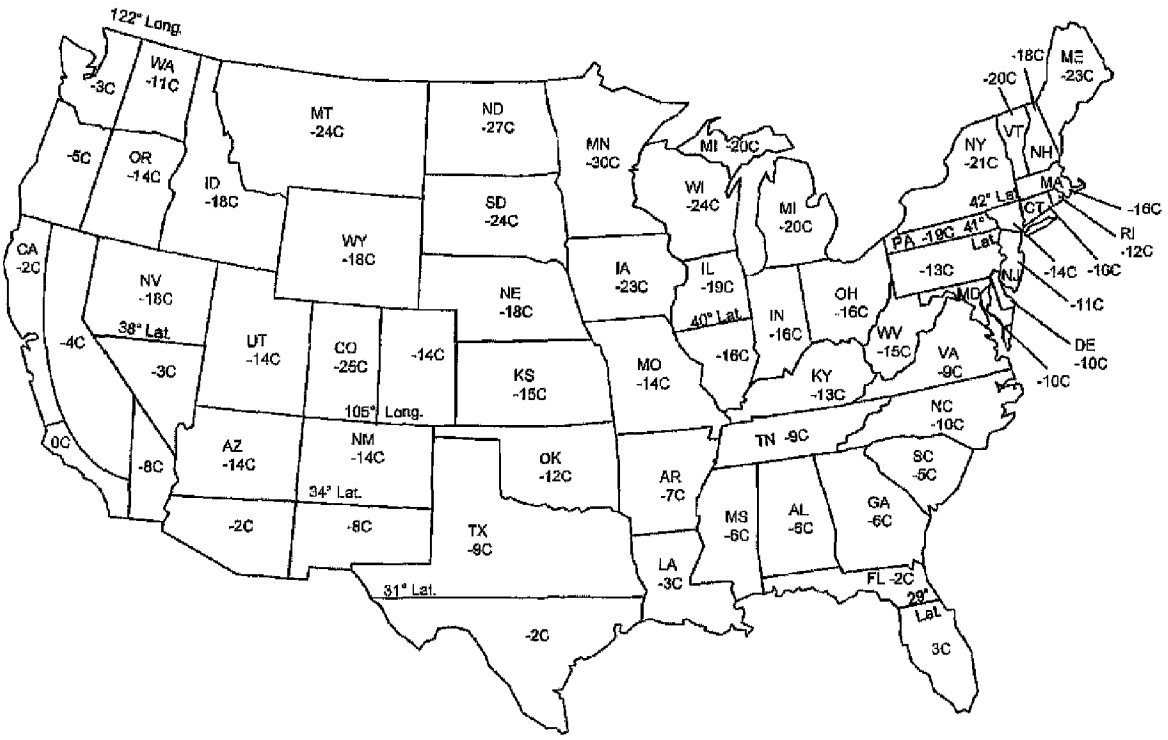

FIG. X5.3 December—10th Percentile Minimum Ambient Air Temperatures

Information Service, Springfield, VA 22151, by requesting Publication No. AD756-420.

X5.2.2 Where states are divided the divisions are noted on the maps and table with the exception of California, which is divided by counties as follows:

California, North Coast—Alameda, Contra Costa, Del Norte, Humbolt, Lake, Marin, Mendocino, Monterey, Napa, San Benito, San Francisco, San Mateo, Santa Clara, Santa Cruz, Solano, Sonoma, Trinity.

California, Interior—Lassen, Modoc, Plumas, Sierra, Siskiyou, Alpine, Amador, Butte, Calaveras, Colusa, El Dorado, Fresno, Glenn, Kern (except that portion lying east of the Los Angeles County Aqueduct), Kings, Madera, Mariposa, Merced, Placer, Sacramento, San Joaquin, Shasta, Stanislaus, Sutter, Tehama, Tulare, Tuolumne, Yolo, Yuba, Nevada.

California, South Coast—Orange, San Diego, San Luis Obispo, Santa Barbara, Ventura, Los Angeles (except that portion north of the San Gabriel Mountain range and east of the Los Angeles County Aqueduct).

California, Southeast—Imperial, Riverside, San Bernardino, Los Angeles (that portion north of the San Gabriel Mountain range and east of the Los Angeles County Aqueduct), Mono, Inyo, Kern (that portion lying east of the Los Angeles County Aqueduct).

X5.2.3 The temperatures in CCL Report No. 316 were in degrees Fahrenheit. The degree Celsius temperatures in Appendix X5 were obtained by converting the original degree Fahrenheit temperatures.

FIG. X5.4 January—10th Percentile Minimum Ambient Air Temperatures

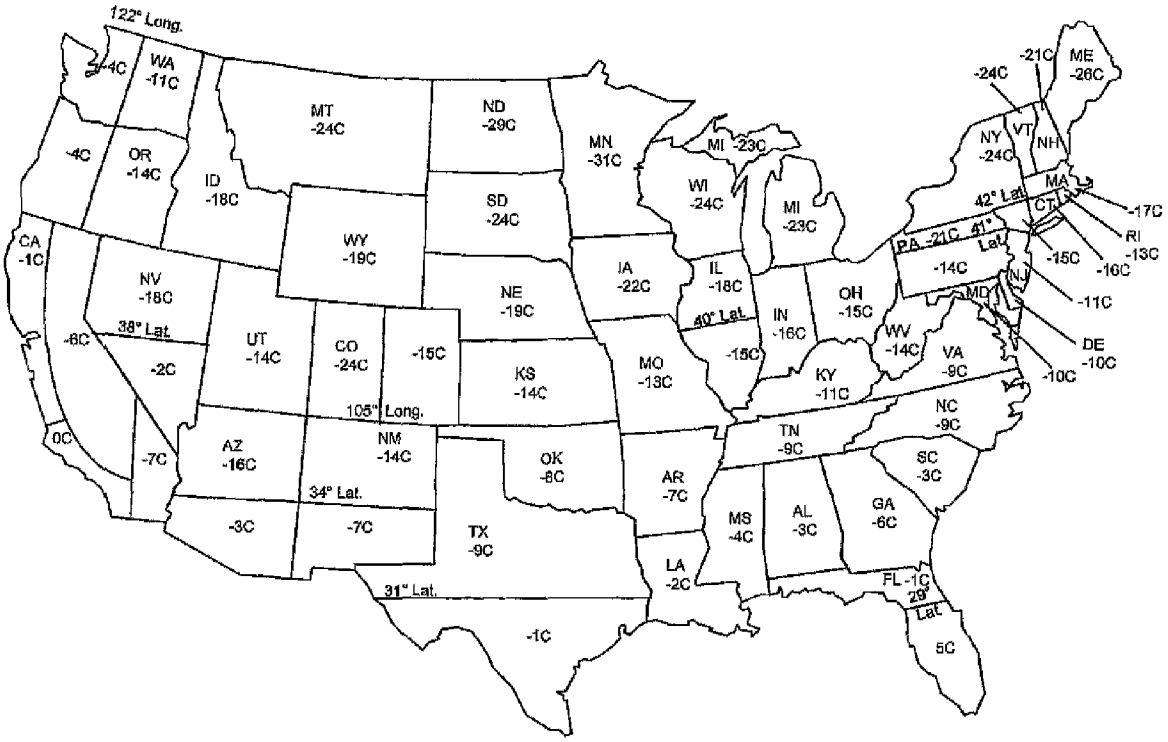

FIG. X5.5 February—10th Percentile Minimum Ambient Air Temperatures

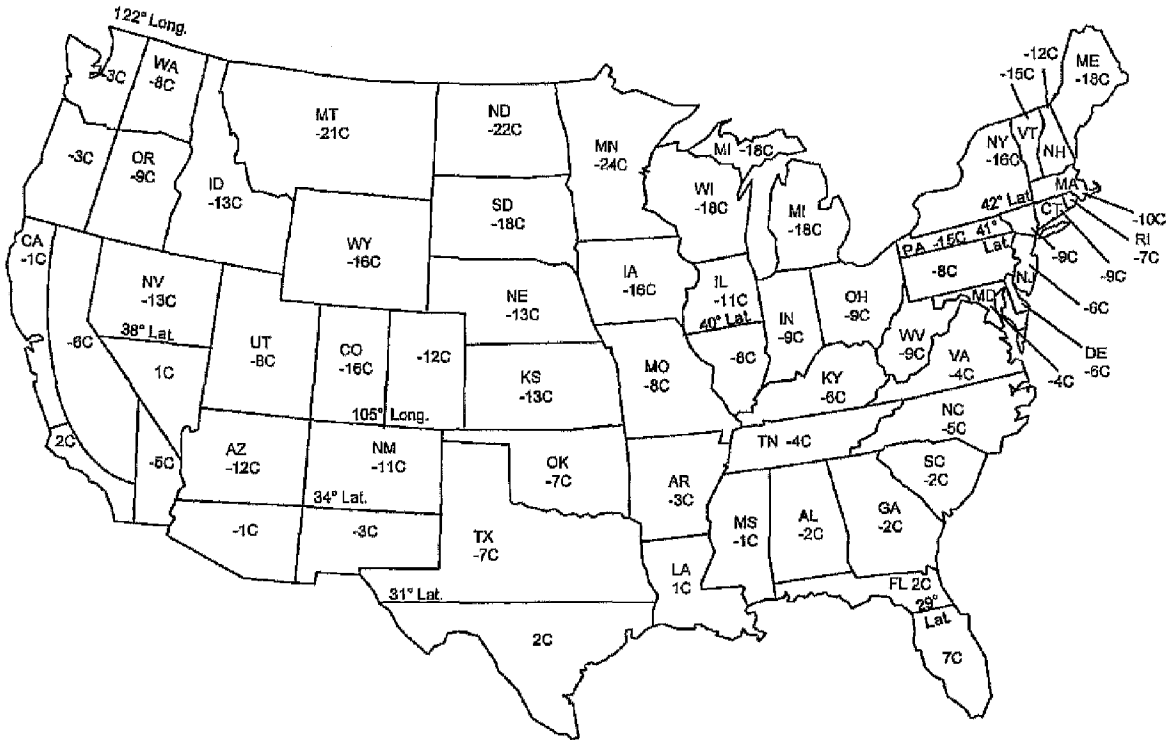

FIG. X5.6 March—10th Percentile Minimum Ambient Air Temperatures

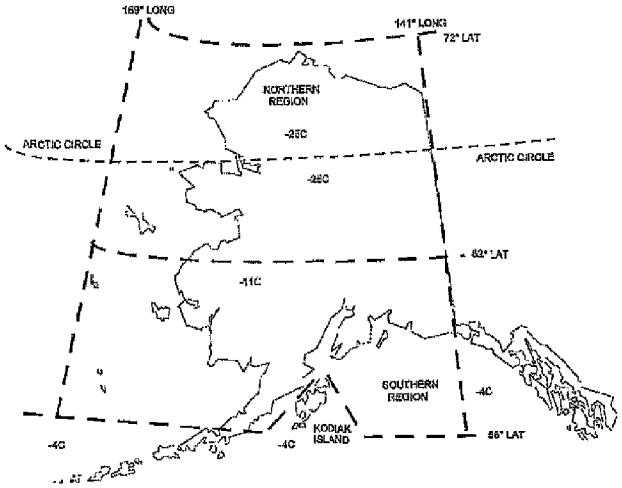

FIG. X5.7 October—10th Percentile Minimum Ambient Air Temperatures

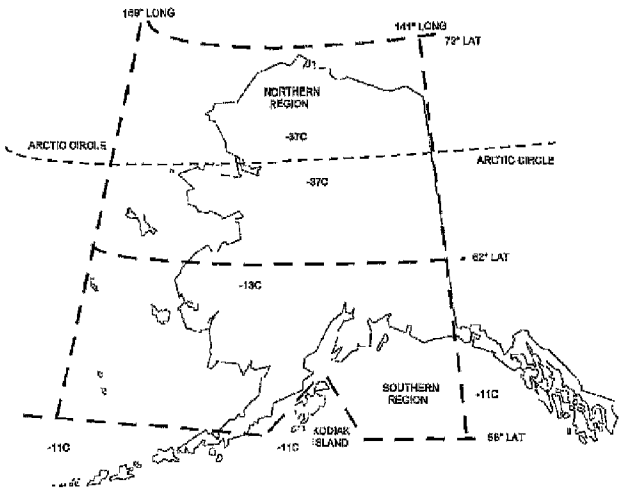

FIG. X5.8 November—10th Percentile Minimum Ambient Air Temperatures

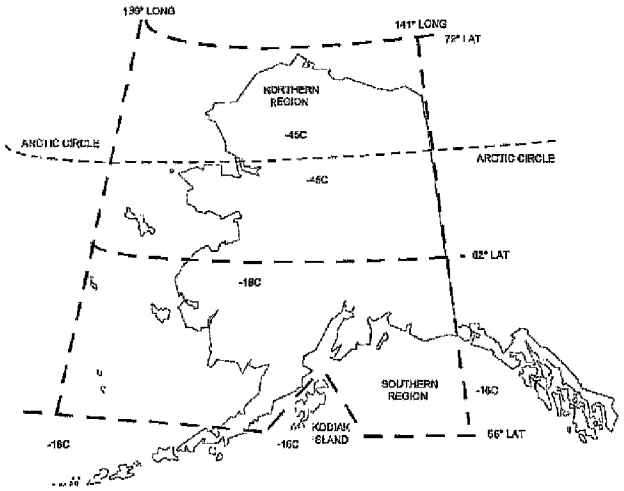

FIG. X5.9 December—10th Percentile Minimum Ambient Air Temperatures

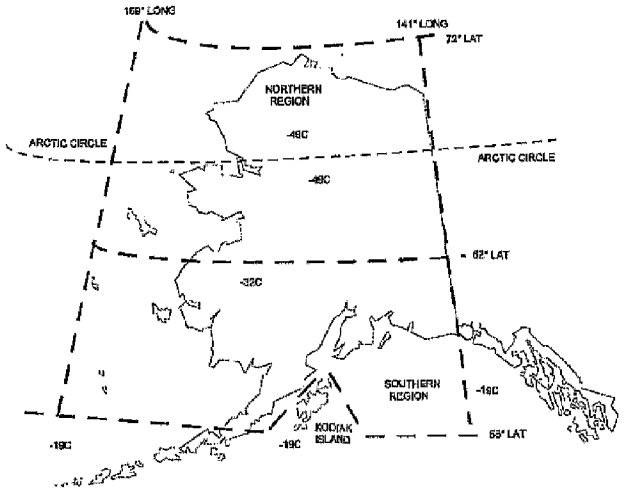

FIG. X5.10 January—10th Percentile Minimum Ambient Air Temperatures

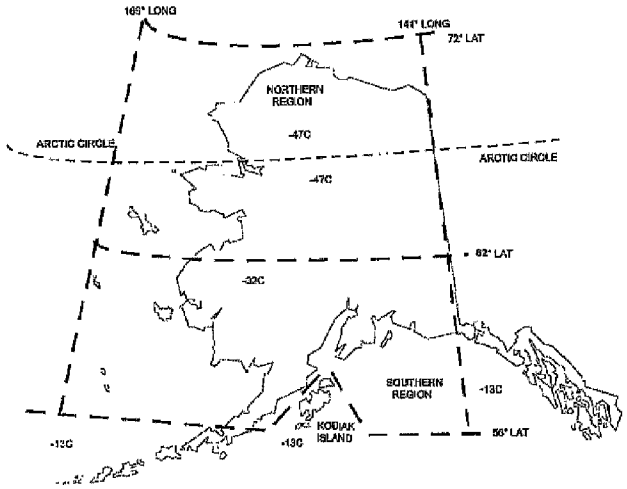

FIG. X5.11 February—10th Percentile Minimum Ambient Air Temperatures

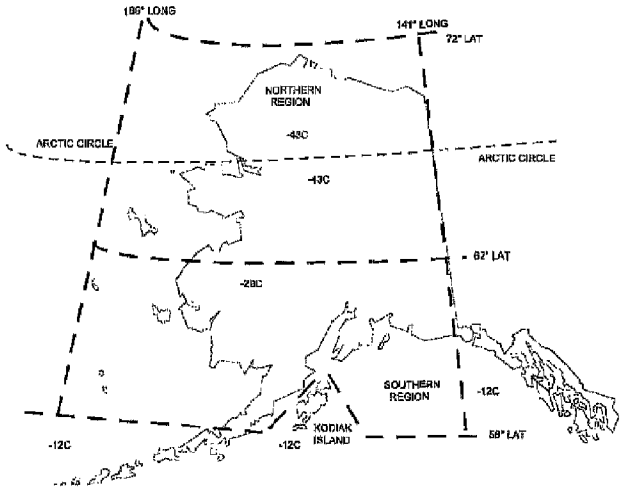

FIG. X5.12 March—10th Percentile Minimum Ambient Air Temperatures

TABLE X5.1 Tenth Percentile Minimum Ambient Air Temperatures for the United States (except Hawaii)

State		10th Percentile Temperature°C, min					
		Oct.	Nov.	Dec.	Jan.	Feb.	March
Alabama		4	−9	−6	−7	−3	−2
Alaska	Northern	−25	−37	−45	−49	−47	−43
	Southern	−11	−13	−18	−32	−32	−29
	South East	−4	−11	−16	−19	−13	−12
Arizona	North 34° latitude	−4	−12	−14	−17	−16	−12
	South 34° latitude	7	0	−2	−4	−3	−1
Arkansas		2	−4	−7	−11	−7	−3
California	North Coast	3	0	−2	−2	−1	−1
	Interior	2	−3	−4	−7	−6	−6
	South Coast	6	2	0	−1	0	2
	Southeast	1	−6	−8	−11	−7	−5
Colorado	East 105° long	−2	−12	−14	−19	−15	−12
	West 105° long	−8	−18	−25	−30	−24	−16
Connecticut		−1	−7	−16	−17	−16	−9
Delaware		2	−3	−10	−11	−10	−6
Florida	North 29° latitude	7	1	−2	−3	−1	2
	South 29° latitude	14	7	3	3	5	7
Georgia		3	−2	−6	−7	−6	−2
Idaho		−4	−13	−18	−21	−18	−13
Illinois	North 40° latitude	−1	−9	−19	−21	−18	−11
	South 40° latitude	1	−7	−16	−17	−15	−8
Indiana		−1	−7	−16	−18	−16	−9
Iowa		−2	−13	−23	−26	−22	−16
Kansas		−2	−11	−15	−19	−14	−13
Kentucky		1	−6	−13	−14	−11	−6
Louisiana		5	−1	−3	−4	−2	1
Maine		−3	−10	−23	−26	−26	−18
Maryland		2	−3	−10	−12	−10	−4
Massachusetts		−2	−7	−16	−18	−17	−10
Michigan		−2	−11	−20	−23	−23	−18
Minnesota		−4	−18	−30	−34	−31	−24
Mississippi		3	−3	−6	−6	−4	−1
Missouri		1	−7	−14	−16	−13	−8
Montana		−7	−18	−24	−30	−24	−21
Nebraska		−3	−13	−18	−22	−19	−13
Nevada	North 38° latitude	−7	−14	−18	−22	−18	−13
	South 38° latitude	8	0	−3	−4	−2	1
New Hampshire		−3	−8	−18	−21	−21	−12
New Jersey		2	−3	−11	−12	−11	−6
New Mexico	North 34° latitude	−2	−11	−14	−17	−14	−11
	South 34° latitude	4	−4	−8	−11	−7	−3
New York	North 42° latitude	−3	−8	−21	−24	−24	−16
	South 42° latitude	−1	−5	−14	−16	−15	−9
North Carolina		−1	−7	−10	−11	−9	−5
North Dakota		−4	−20	−27	−31	−29	−22
Ohio		−1	−7	−16	−17	−15	−9
Oklahoma		1	−8	−12	−13	−8	−7
Oregon	East 122° long	−6	−11	−14	−19	−14	−9
	West 122° long	0	−4	−5	−7	−4	−3
Pennsylvania	North 41° latitude	−3	−8	−19	−20	−21	−15
	South 41° latitude	0	−6	−13	−14	−14	−8
Rhode Island		1	−3	−12	−13	−13	−7
South Carolina		5	−1	−5	−5	−3	−2
South Dakota		−4	−14	−24	−27	−24	−18
Tennessee		1	−5	−9	−11	−9	−4
Texas	North 31° latitude	3	−6	−9	−13	−9	−7
	South 31° latitude	9	2	−2	−3	−1	2
Utah		−2	−11	−14	−18	−14	−8
Vermont		−3	−8	−20	−23	−24	−15
Virginia		2	−3	−9	−11	−9	−4
Washington	East 122° long	−2	−8	−11	−18	−11	−8
	West 122° long	0	−3	−3	−7	−4	−3
West Virginia		−3	−8	−15	−16	−14	−9
Wisconsin		−3	−14	−24	−28	−24	−18
Wyoming		−4	−15	−18	−26	−19	−16

SUMMARY OF CHANGES

Subcommittee D02.E0.02 has identified the location of selected changes to this standard since the last issue (D 975–06b) that may impact the use of this standard. (Approved Feb. 1, 2007.)

(*1*) Added standards to the Referenced Documents.
(*2*) Added Section 4.
(*3*) Added X2.2.2.

Subcommittee D02.E0.02 has identified the location of selected changes to this standard since the last issue (D 975–06a) that may impact the use of this standard. (Approved Nov. 1, 2006.)

(*1*) Revised Appendix X4.

Subcommittee D02.E0.02 has identified the location of selected changes to this standard since the last issue (D 975–06) that may impact the use of this standard. (Approved Oct. 1, 2006.)

(*1*) Added Test Method D 6890.
(2) Revised 5.1.10.

Subcommittee D02.E0.02 has identified the location of selected changes to this standard since the last issue (D 975–05) that may impact the use of this standard. (Approved May 15, 2006.)

(*1*) Deleted Test Method D 6920 from this standard.

Subcommittee D02.E0.02 has identified the location of selected changes to this standard since the last issue (D 975–04c$^{\varepsilon 1}$) that may impact the use of this standard. (Approved June 1, 2005.)

(*1*) Removed footnote J from Grade No. 4–D in Table 1.

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:

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Standards Body:

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: E 11 – 95

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 $4\sqrt{2}$:1.

4.3 All measurements of openings and wire diameters

[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.

Current edition approved Jan. 15, 1995. Published March 1995. Originally published as E 11 – 25T. Last previous edition E 11 – 87.

[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.

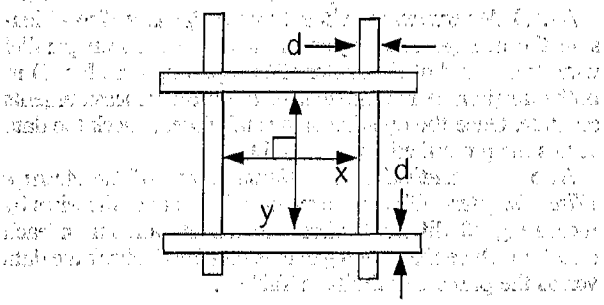

FIG. 1 Proper Dimensioning of Wire Cloth Mesh

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.

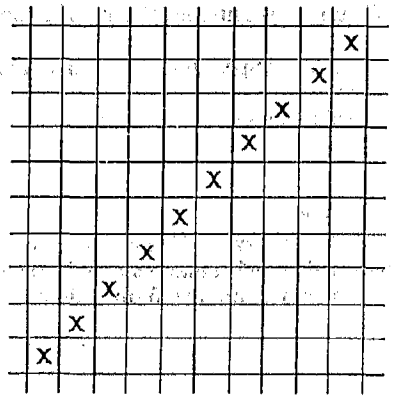

FIG. A1.1 Orientation of Openings to be Measured in Each Field

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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OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: E 1337 – 90 (Reapproved 1996)

Standard Test Method for Determining Longitudinal Peak Braking Coefficient of Paved Surfaces Using a Standard Reference Test Tire[1]

This standard is issued under the fixed designation E 1337; 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 measurement of peak braking coefficient of paved surfaces using a standard reference test tire (SRTT) as described in Specification E 1136 that represents current technology passenger car radial tires. General test procedures and limitations are presented for determining peak braking coefficient independent of surface conditions. Actual surface test conditions are determined and controlled by the user at the time of test. Test and surface condition documentation procedures and details are specified. This measurement quantifies the peak braking coefficient at the time of test and does not necessarily represent a maximum or fixed value.

1.2 This test method utilizes a measurement representing the peak braking force on a braked test tire passing over a road surface. This test is conducted with a tire under a nominal vertical load at a constant speed while its major plane is parallel to its direction of motion and perpendicular to the pavement.

1.3 The measured peak braking coefficient obtained with the equipment and procedures stated herein may not necessarily agree or correlate directly with those obtained by other surface coefficient measuring methods.

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:*

E 274 Test Method for Skid Resistance of Paved Surfaces Using A Full-Scale Tire[2]

E 556 Test Method for Calibrating a Wheel Force or Torque Transducer Using a Calibration Platform (User Level)[2]

E 867 Terminology Relating to Traveled Surface Characteristics[2]

E 1136 Specification for a Radial Standard Reference Test Tire[2]

F 377 Practice for Calibration of Braking/Tractive Measuring Devices for Testing Tires[3]

F 408 Test Method for Tires for Wet Traction in Straight-Ahead Braking, Using a Towed Trailer[3]

F 457 Method for Speed and Distance Calibration of a Fifth Wheel Equipped with Either Analog or Digital Instrumentation[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *chirp test*—the progressive application of brake torque required to produce the maximum value of longitudinal braking force that will occur prior to wheel lockup, with subsequent brake release to prevent any wheel lockup (tire slide).

3.1.2 For other definitions pertaining to this standard, see Terminology E 867 and Method F 408.

3.2 *Descriptions of Terms:*

3.2.1 *braking force coefficient, tire*—the ratio of braking force to vertical load.

3.2.2 *braking force coefficient, tire, peak*—the maximum value, as defined in 12.2, of tire braking force coefficient that occurs prior to wheel lockup as the braking torque is progressively increased.

3.2.3 *braking force coefficient, tire, slide*—the value of the braking force coefficient obtained on a locked wheel.

3.2.4 *braking force, tire*—the negative longitudinal force resulting from braking torque application.

3.2.5 *braking torque*—the negatively directed wheel torque.

3.2.6 *longitudinal force, tire (F_x)*—the component of a tire force vector in the X' direction.

3.2.7 *tire-axis system*—the origin of the tire-axis system is the center of the tire contact. The X' axis is the intersection of the wheel plane and the road plane with a positive direction forward. The Z′ axis is perpendicular to the road plane with a positive direction downward. The Y′ axis is in the road plane, its direction being chosen to make the axis system orthogonal and right-hand (see Fig. 1 in Method F 408).

3.2.8 *tire forces*—the external forces acting on the tire by the road.

3.2.9 *torque wheel (T)*—The external torque applied to a tire from a vehicle about the wheel spin axis. Driving torque is positive wheel torque; braking torque is negative wheel torque.

[1] This test method is under the jurisdiction of ASTM Committee E-17 on Vehicle-Pavement Systems and is the direct responsibility of Subcommittee E17.21 on Field Methods for Measuring Tire Pavement Friction.

Current edition approved Feb. 23, 1990. Published April 1990.

[2] *Annual Book of ASTM Standards*, Vol 04.03.

[3] *Annual Book of ASTM Standards*, Vol 09.02.

3.2.10 *vertical load (F_z)*—the downward vertical component of force between the tire and the road.

4. Summary of Test Method

4.1 The measurements are conducted with a standard reference test tire (Specification E 1136) mounted on a test trailer towed by a vehicle. The trailer contains a transducer, instrumentation, and actuation controls for the braking of the test tire. See 6.6 for trailer instrumentation.

4.2 The test apparatus is normally brought to a test speed of 40 mph (64 km/h). The brake is progressively applied until sufficient braking torque results to produce the maximum braking force that will occur prior to wheel lockup. Longitudinal force, vertical load, and vehicle speed are recorded with the aid of suitable instrumentation and data acquisition equipment.

4.3 The peak braking coefficient of the road surface is determined from the ratio of the maximum value of braking force to the simultaneous vertical load occurring prior to wheel lockup as the braking torque is progressively increased.

5. Significance and Use

5.1 Pavement surfaces have different traction characteristics, depending on many factors. Surface texture, binder content, usage, environmental exposure, and surface conditions (that is, wet, dry) are some of the factors.

5.2 The measured values represent peak braking coefficients for tires of the general type in operation on passenger vehicles, obtained with a towed test trailer on a prescribed road surface, under user defined surface conditions. Such surface conditions may include the water depth used to wet the road surface and the type of water application method. Variations in these conditions may influence the test results.

6. Apparatus

6.1 The apparatus consists of a tow vehicle and test trailer. The vehicle and trailer must comply with all legal requirements applicable to state laws when operated on public roads.

6.2 *Tow Vehicle*—The vehicle shall have the capability of maintaining a test speed of 40 mph (64 km/h) within ±0.5 mph (±0.8 km/h) even at maximum level of application of braking forces.

6.3 *Test Trailer*—The test wheel shall be equipped with a sufficient braking torque to produce the maximum value of braking test wheel longitudinal force at the conditions specified.

6.3.1 Each of the trailer wheels shall have a suspension capable of holding toe and camber changes to within ±0.05° with maximum vertical suspension displacements under both static and dynamic conditions.

6.3.2 The rate of brake application shall be sufficient to control the time interval between initial brake application and peak longitudinal force to be between 0.3 and 0.5 s.

6.4 *Vertical Load*—The trailer shall be of such a design as to provide a static load of 1031± 15 lbf (4586 ± 67 N) to the test wheel and on detachable trailers a static down load of 100 to 200 lbf (445 to 890 N) at the hitch point.

6.5 *Tire and Rim*—The test tire shall be the standard reference test tire (SRTT) for pavement tests, as specified in Specification E 1136, mounted on a suitable 14 by 6-in. rim.

6.5.1 When irregular wear or damage results from tests, or when wear or usage influences the test results, the use of the tire should be discontinued.

6.6 *Instrumentation*:

6.6.1 *General Requirements for Measuring System*—The instrumentation system shall conform to the following overall requirements at ambient temperatures between 40 and 100°F (4 and 38°C):

6.6.1.1 Overall system accuracy of ±1.5 % of applied load from 200 lbf (890 N) to full scale; for example, at 200 lbf (890 N), applied calibration force of the system output shall be determinable within ±3 lbf (±13 N).

6.6.1.2 The exposed portions of the system shall tolerate 100 % relative humidity (rain or spray) and all other adverse conditions, such as dust, shock, and vibrations which may be encountered in highway operations.

6.6.1.3 *Braking Forces*—The braking force measuring transducer shall measure longitudinal reaction force within a range between 0 and 2000 lbf (0 and 8.9 kN) generated at the tire-pavement interface as a result of brake application. The tire force-measuring transducer shall be of such design as to measure the tire-pavement interface force with minimum inertial effects. Transducers are recommended to provide an output directly proportional to force with hysteresis less than 1 % of the applied load, nonlinearity less than 1 % of the applied load up to the maximum expected loading, and sensitivity to any expected cross-axis loading or torque loading less than 1 % of the applied load. The force transducer shall be mounted in such a manner as to experience less than 1° angular rotation with respect to its measuring plane at the maximum expected loading.

6.6.1.4 *Vertical Load*—The vertical load measuring transducer shall measure the vertical load at the test wheel during brake application. The transducer shall have the same specifications as those described in 6.6.1.3.

NOTE 1—Other transducer systems may be used to determine peak braking coefficients if they can be shown to correlate with the force-measuring transducer system with the same overall accuracy.

6.6.1.5 *Vehicle Speed-Measuring Transducers*—Transducers such as "fifth wheel" or a free-rolling wheel coupled tachometer shall provide speed resolution and accuracy of ±1.5 % of the indicated speed or ±0.5 mph (±0.8 km/h), whichever is greater. Output shall be directly viewable by the driver and shall be simultaneously recorded. Fifth wheel systems shall conform to Method F 457.

6.6.1.6 *Signal Conditioning and Data Acquisition*—All signal conditioning and recording equipment shall provide linear output and shall allow data reading resolution to meet the requirements of 6.6.1.1.

6.6.1.7 All strain-gage transducers shall be equipped with resistance shunt calibration resistors or equivalent that can be connected before or after test sequences. The calibration signal shall be at least 50 % of the normal vertical load and shall be recorded.

6.6.1.8 A digital data acquisition system shall be employed to individually digitize the braking force, vertical load, and vehicle speed analog outputs. The braking force, vertical load,

and test wheel speed input signals to be digitized shall be sampled (as close to simultaneous as possible to minimize phase shifting) at 100 samples per second for each channel from unfiltered analog signals. Vehicle speed can be analog filtered, if necessary, to remove noise since this is a steady-state signal.

NOTE 2—Experience indicates that data sampling at 100 samples per second of unfiltered analog skid trailer data will properly describe the significant frequencies. To prevent "aliasing," caution must be exercised in digitizing skid trailer data which contains any significant frequencies above 50 Hz or other types of analog data.

7. Hazards

7.1 The test vehicle, as well as all attachments to it, shall comply with all applicable state and federal laws. All necessary precautions shall be taken beyond those imposed by laws and regulations to ensure maximum safety of operating personnel and other traffic. No test shall be made when there is danger that dispersed water may freeze on the pavement.

8. Preparation of Apparatus

8.1 *Preparation of Test Tire*:

8.1.1 Trim the test tires to remove all protuberances in the tread area caused by mold air vents or flashes at mold junctions.

8.1.2 Test tires should be stored in such a location that they all have the same ambient temperature prior to testing and shield them from the sun to avoid excessive heating by solar radiation.

8.1.3 Mount the test tire on Tire and Rim Association (TRA) recommended rim[4] (6.5) by using conventional mounting methods. **Caution:** Assure proper bead seating by the use of a suitable lubricant. Excessive use of lubricant should be avoided to prevent slipping of the tire on the wheel rim.

8.1.4 Check the test tires for the specified inflation pressure at ambient temperature (cold), just prior to testing. The test tire inflation pressure shall be 35 ± 0.5 psi. (241 ± 3 kPa)

9. Calibration

9.1 *Vehicle Speed*—Calibrate the test vehicle speed indicator at the test speed by determining the time for traversing at constant speed a reasonably level and straight, accurately measured pavement of a length appropriate for the method of timing. Load the test trailer to its specified operating weight for this calibration. Record speed variations during a traverse with the test system. Make a minimum of three runs at each test speed to complete the calibration. Other methods of equivalent accuracy may be used. Calibration of a fifth wheel shall be performed in accordance with Method F 457.

10. Conditioning

10.1 *Pretest Tire Conditioning*:

10.1.1 Test tire pretest conditioning shall be performed to precondition all tires prior to initial testing. Pretest conditioning is to be done only once per tire and prior to any actual test measurements. This process is recommended because the new tire burnish effect may have an influence on the peak braking coefficient obtained and to minimize test variability caused by transient, non-preconditioned, tire braking performance.

10.1.2 Pretest tire conditioning shall be conducted on a dry and level surface. Each tire shall be chirped ten times at 20 mph (32 km/h) under test load.

10.2 *General Test Conditions*:

10.2.1 The test surface shall be free of loose material or foreign deposits.

10.2.2 Do not test when wind conditions interfere with wetted test repeatability. Test results may be influenced by wind speed, or direction, or both. The magnitude of this dependence is a function of the water depth, application procedures, and surface wind protection.

11. Procedure

11.1 Warm up electronic test equipment as required for stabilization.

11.2 Install an SRTT (Specification E 1136) in the test position of the test trailer. A tire with a similar loaded radius and high cornering properties should be used on the opposite side to level the axle and to minimize trailer yaw during brake torque applications.

11.3 Check and, if necessary, adjust the test trailer static weight on the test tire to the specified test load (see 6.4).

11.4 Check and adjust tire inflation pressure as required immediately before testing to specified value (see 8.1.4).

11.5 When testing on an externally wetted test surface, offset the trailer test wheel sufficiently to prevent "tracking" of the towing vehicle. Twelve to sixteen in. (305 to 406 mm) is suggested.

11.6 Record tire identification and other data, including date, time, ambient temperature, test surface temperature, tire durometer, test surface type, and water depth (if wetted surface is used). Measure the water depth with a variable height probe type device.

11.7 Record electrical calibration signals prior to and after testing each surface, or as needed to ensure valid data.

11.8 Perform pretest tire conditioning (10.1) if using a new tire.

11.9 Conduct test at the required test vehicle speed. It is recommended that peak braking coefficient measurement tests be conduoted using the chirp test methodology to minimize tire damage due to tire sliding.

11.10 Make at least eight determinations of the peak braking coefficient evenly distributed over the test surface with the test system at the specified test speed.

11.11 *Lateral Positioning of Test Vehicle on Highway Surfaces*—Normally, testing shall be done in the center of either wheel track of a traffic lane on a highway. The specific details regarding lane and the wheel-path used should be provided when reporting the data.

11.12 *Test Speeds*:

11.12.1 The standard test speed shall be 40 mph (64 km/h), and tests shall normally be conducted at that speed. Where the legal maximum speed is less than 40 mph (64 km/h), the tests may have to be conducted at a lower speed. Where the legal speed is considerably in excess of 40 mph (64 km/h), tests may

[4] Current recommendations available from the Tire and Rim Association, 3200 Market St., Akron, OH 44313.

be made at the prevailing traffic speed, but it is recommended that at the same locations, additional tests be made at 40 mph (64 km/h). Maintain test speeds within ±1 mph (1.5 km/h).

11.12.2 The test speed must be given when the peak braking coefficient is quoted. This may be done by adding the numerals of the actual test speed in miles per hour in parentheses to the coefficient, for example, 0.50(50) indicates the peak braking coefficient was obtained at a test speed of 50 mph (80 km/h).

12. Calculation

12.1 *Data Reduction*:

12.1.1 Digitally filter the digitized input analog signals of braking force, vertical load, and vehicle speed using a five point moving average technique.

12.1.2 *Digital Filtering Methodology*—Calculate an average value for the first five digital data points. Drop the first data point and add the sixth data point, calculate another five point average value. Repeat this procedure for all remaining data points. This sequence is done individually on all the above digitized input analog signals. The following example computations illustrate the method using one channel.

$$(pt1 + pt2 + pt3 + pt4 + pt5)/5 = PT1$$
$$(pt2 + pt3 + pt4 + pt5 + pt6)/5 = PT2$$
$$(pt3 + pt4 + pt5 + pt6 + pt7)/5 = PT3$$

A new set of data points (indicated by capital letters) are then defined to represent the filtered data for each channel (that is, Avg ptx = PTy).

PT1, PT2, PT3, etc. – braking force
PT1, PT2, PT3, etc. – vertical force

12.2 Determining and Calculating Peak Braking Coefficient.

12.2.1 The peak braking coefficient shall be determined for each run (brake application).

12.2.2 Using the digitally filtered data (PT1, PT2, PT3, etc.), scan the longitudinal channel and determine the highest absolute filtered value (PTy) prior to wheel lock up. Calculate an average peak braking force value using the highest filtered value (PTy) and one filtered point directly before (PTy_{-1}) and directly after it (PTy_{+1}). This three point average is the peak braking force value developed for this individual lock up.

12.2.3 Determine the vertical load value from its respective digitally filtered data that corresponds to the highest absolute value for braking force, from 12.2.2. Calculate an average vertical load value using this corresponding value and one point directly before and directly after it. This three point average is the vertical load value that corresponds to the average peak braking force for this individual lock up.

12.2.4 Calculate the peak braking coefficient by dividing the three point average peak braking force, determined from 12.2.2, by the three point average vertical load, as determined in 12.2.3. The peak braking coefficient should be reported to two (2) decimal places.

12.3 For each test (11.10) the mean and standard deviation for peak braking coefficient are calculated from the individual determinations.

13. Report

13.1 *Field Report*—The field report for each test section shall contain data on the following items:

13.1.1 Identify test procedure used,
13.1.2 Location and identification of test section,
13.1.3 Date and time of day,
13.1.4 Weather conditions,
13.1.5 Lane and wheel-path tested,
13.1.6 Speed of test vehicle (for each test),
13.1.7 Peak braking coefficient (for each test),
13.1.8 Water depth, if wetted surface is used, and
13.1.9 Ambient and surface temperature.

13.2 *Summary Report*—The summary report shall include, for each test section, data on the following items insofar as they are pertinent to the variables or combinations of variables under investigation:

13.2.1 Location and identification of test section,
13.2.2 Number of lanes and presence of lane separators,
13.2.3 Grade and alignment,
13.2.4 Pavement type, mix design of surface course, condition, and aggregate type (specific source, if available),
13.2.5 Age of pavement,
13.2.6 Average daily traffic,
13.2.7 Posted speed limit,
13.2.8 Date and time of day,
13.2.9 Weather conditions,
13.2.10 Lane and wheel-path tested,
13.2.11 Ambient and surface temperature, and
13.2.12 Average, high, and low peak braking coefficient for the test section and speed at which the tests were made. (If values are reported that were not used in computing the average, this fact should be reported.)

14. Precision and Bias

14.1 *Precision*—Data are not yet available for making a statement on the precision of this test method. When such data become available, a precision statement will be included in this test method.

14.2 *Bias*—There are no standards or references with which the results of this test can be compared. The function of the test as indicated above is to be able to make comparisons among road surfaces tested with the same tire. It is believed that the results of the test method are adequate for making such comparisons without an external reference for assessing accuracy. It must be noted that surface friction is affected by many variables such as environmental conditions, usage, age, surface contamination (externally applied water), etc., and measured values are only valid until one of these conditions significantly changes.

15. Recommendations for Tire Use and Operational Requirements

15.1 When irregular wear or damage results from tests, or when wear or usage influences the test results, the use of the tire should be discontinued.

NOTE 3—Test results such as measured braking force may be influenced by tire groove depth or tread hardness, or both. The magnitude of this dependence is a function of the water depth, pavement characteristics, test speed, tire aging effects, and break-in (preconditioning).

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.

Document Name: ASTM E145: Standard Specification for Gravity-Convection and Forced- Ventilation Ovens

CFR Section(s): 40 CFR 63.14

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: E 145 – 94ε1

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.

ε1 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

[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.

Current edition approved Aug. 15, 1994. Published October 1994. Originally published as E 145 – 59 T. Last previous edition E 145 – 68 (1992).

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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Document Name:

CFR Section(s):

Standards Body:

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: E 169 – 87

Standard Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis[1]

This standard is issued under the fixed designation E 169; 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 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:

$$A = a_1bc_1 + a_2bc_2 + \ldots + a_nbc_n$$

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

[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)$^{\epsilon 1}$.

[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-

[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
Isooctane	<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™ 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.

$$c = A/ab$$

$$C, \% = (c/C_s) \times 100 = (A/abC_s) \times 100$$

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:

$$C, \% = \frac{A_1 - \left[A_s + [A_2 - A_3] \times \frac{\lambda_3 - \lambda_1}{\lambda_3 - \lambda_2}\right] f}{abC_c} \times 100$$

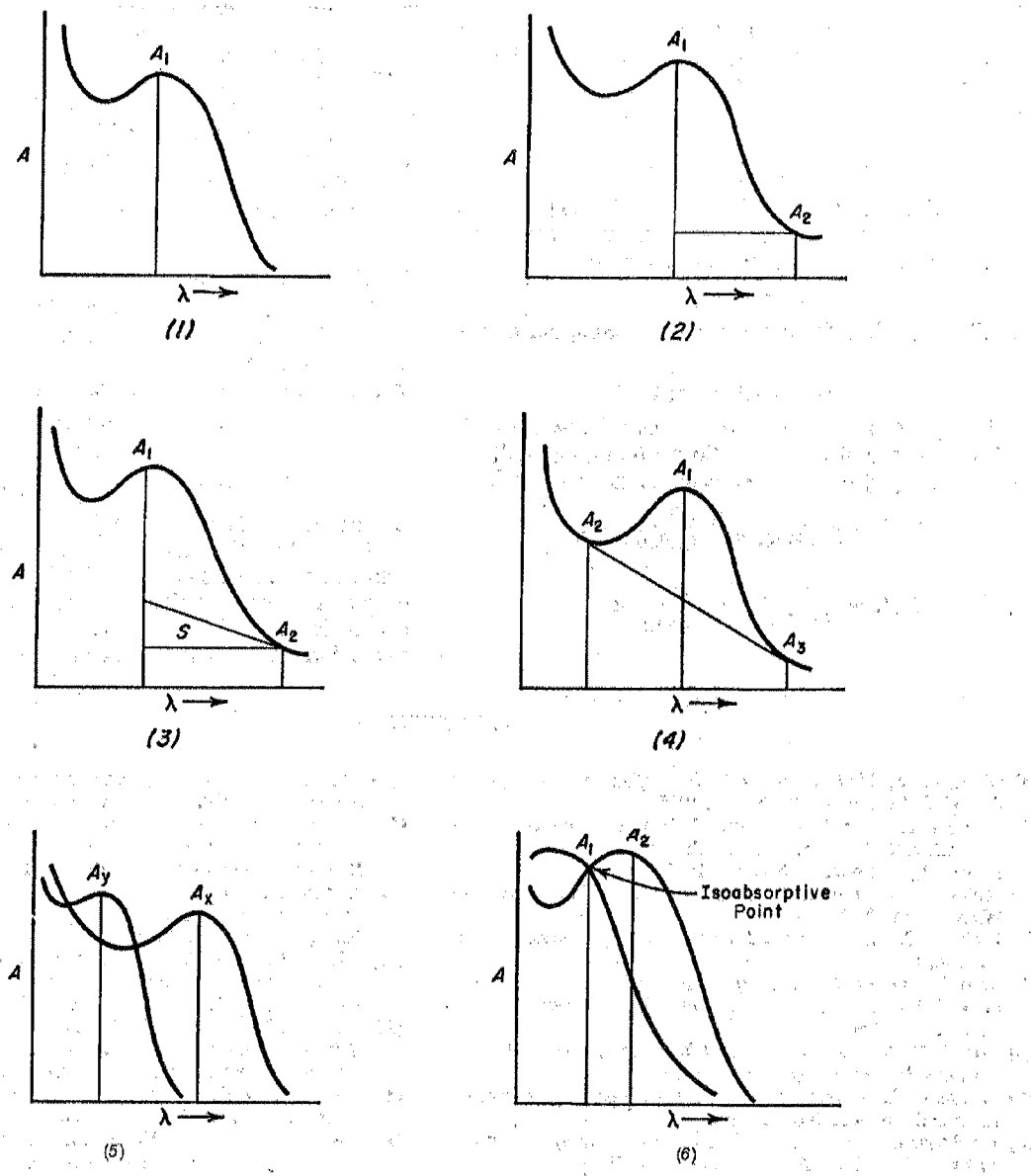
The absorptivity a is here the effective absorptivity as determined on a pure sample, using the corrections, and is somewhat lower than the true or absolute absorptivity.

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:

$$C, \% = f\left[\frac{A_1 - \frac{A_2 + A_3}{2}}{abC_s}\right] \times 100$$

10.4.5 *One Component, With Background Correction from Outside Data:*



(1) One Component, No Background Correction.
(2) One Component, Simple Background Correction.
(3) One Component, with Slope-Type Background Correction.
(4) One Component, with Linear Background Correction.
(5) Two Components, with Overlapping Absorption for Only One Component.
(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:

$$C_x, \% = \frac{[a_{y2}A_1 - a_{y1}A_2]f}{bC_s \times (a_{y2}a_{x1} - a_{y1}a_{x2})} \times 100$$

$$C_y, \% = \frac{[a_{x2}A_1 - a_{x1}A_2]f}{bC_s \times (a_{y1}a_{x2} - a_{y2}a_{x1})} \times 100$$

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.)

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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.

[ε] 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.

Current edition approved July 1, 1982. Published September 1982. Originally published as E 185 – 61 T. Last previous edition E 185 – 79.

[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

[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.

[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.

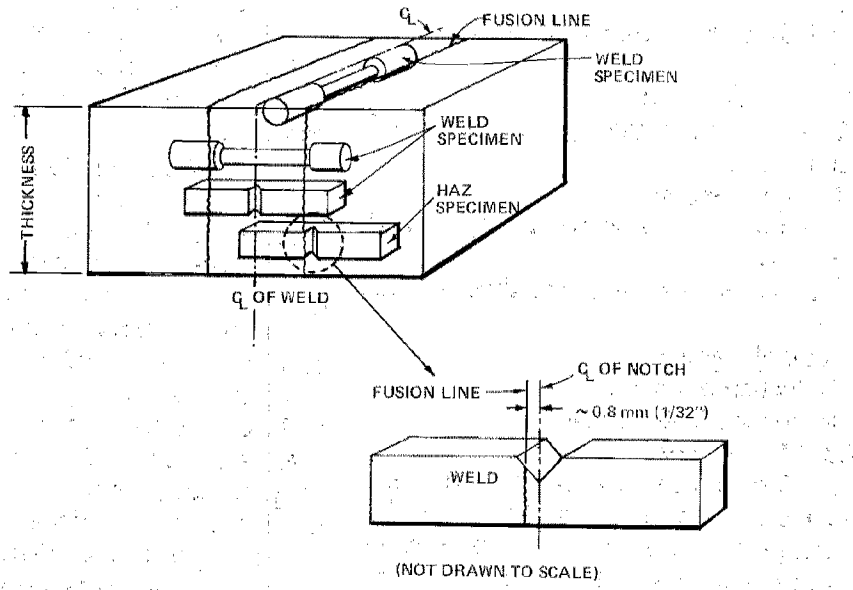


FIG. 1 Location of Test Specimens Within Weld and Heat-Affected-Zone (HAZ) Test Material.

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 E23: Standard Test Methods for Notched Bar Impact Testing of Metallic Materials

CFR Section(s): 46 CFR 56.50-105(a)(1)(ii)

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: E 23 – 82

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-

[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.

Current edition approved March 5, 1982. Published July 1982. Originally published as E 23 – 33 T. Last previous edition E 23 – 81.

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:

$$l = 0.2484p^2, \text{ to determine } l \text{ in metres}$$

$$l = 0.815p^2, \text{ to determine } l \text{ in feet}$$

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

[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:

[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

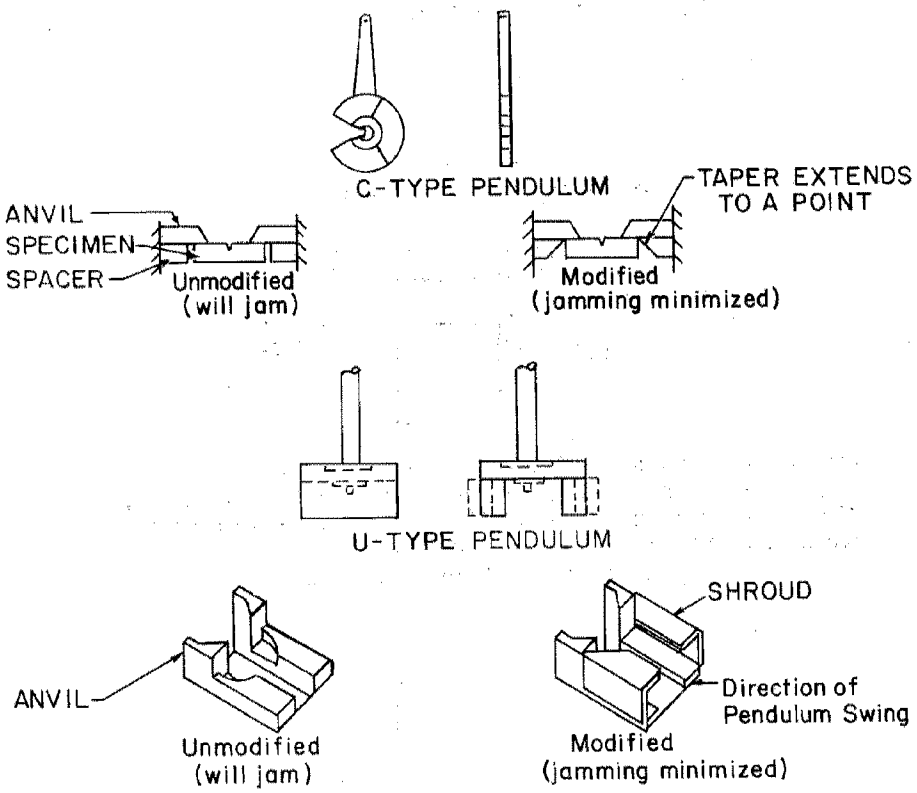
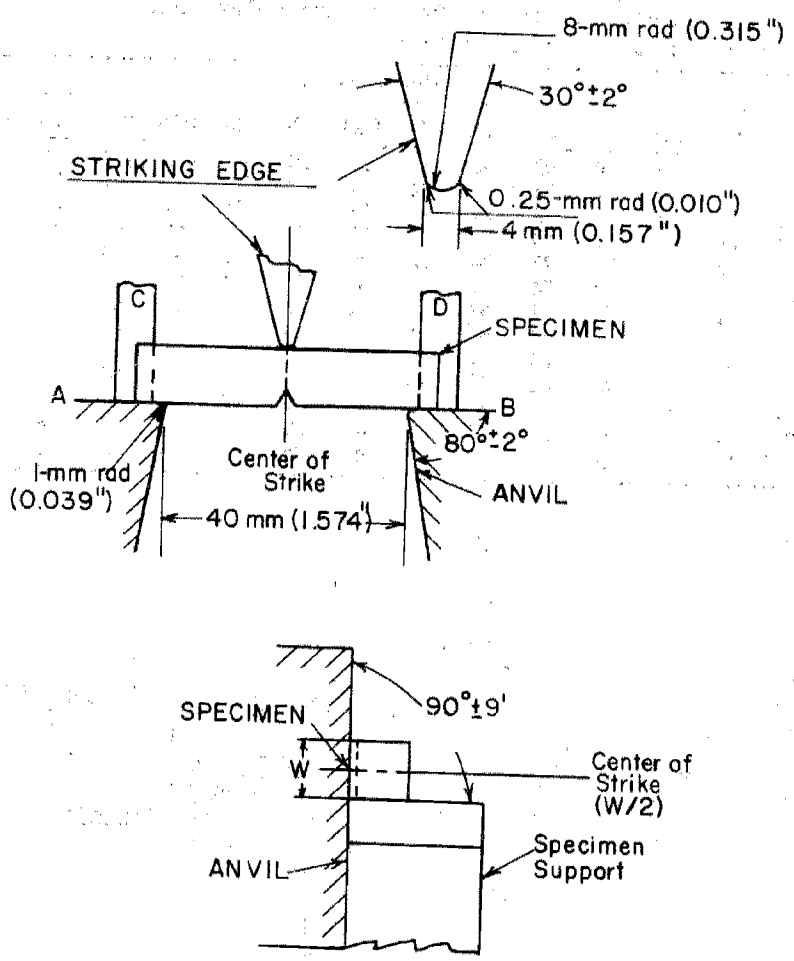


FIG. 1 Typical Pendulums and Anvils for Charpy Machines, Shown with Modifications to Minimize Jamming



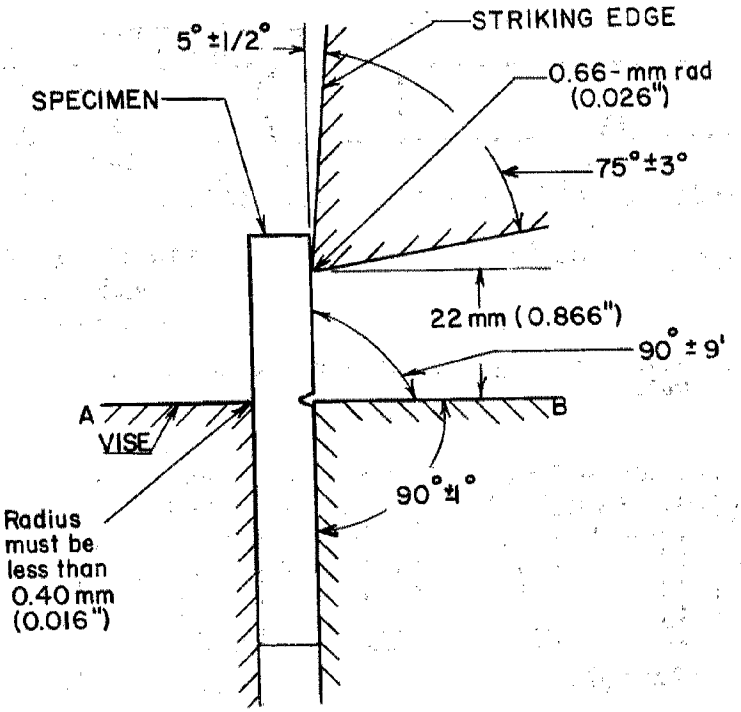
All dimensional tolerances shall be ±0.05 mm (0.002 in.) unless otherwise specified.

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



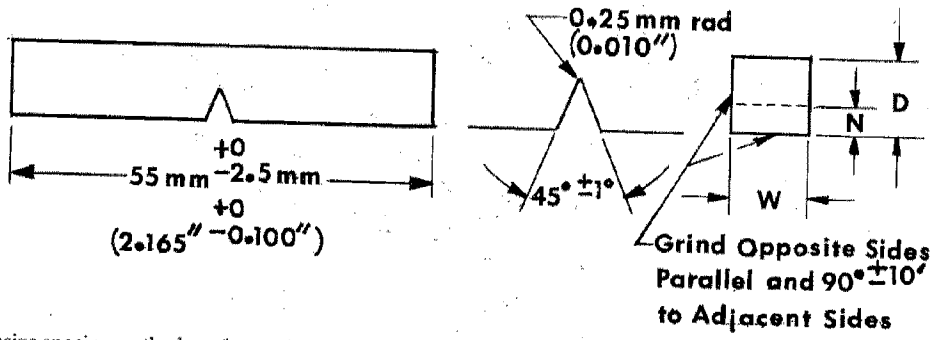
All dimensional tolerances shall be ±0.05 mm (0.002 in.) unless otherwise specified.

NOTE 1—The clamping surfaces of A and B shall be flat and parallel within 0.025 mm (0.001 in.).

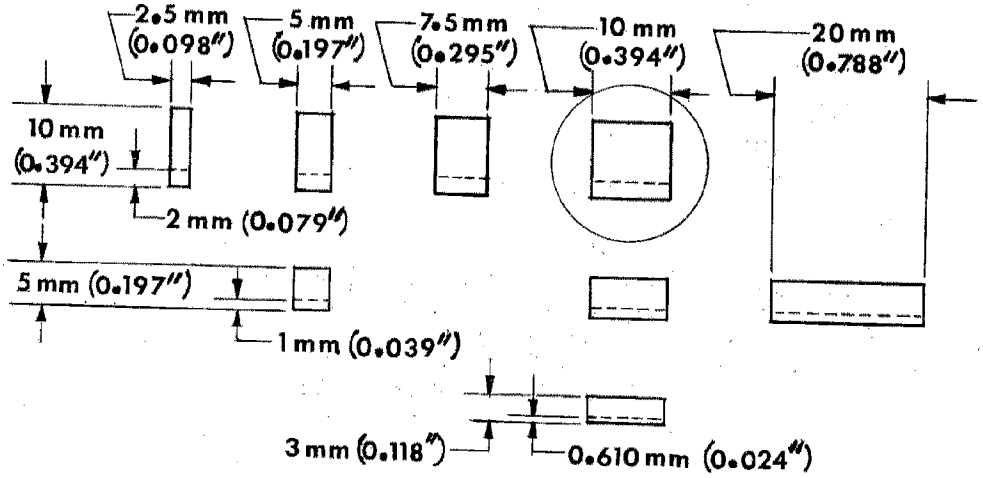
NOTE 2—Finish on unmarked parts shall be 2 μm (63 μin.).

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

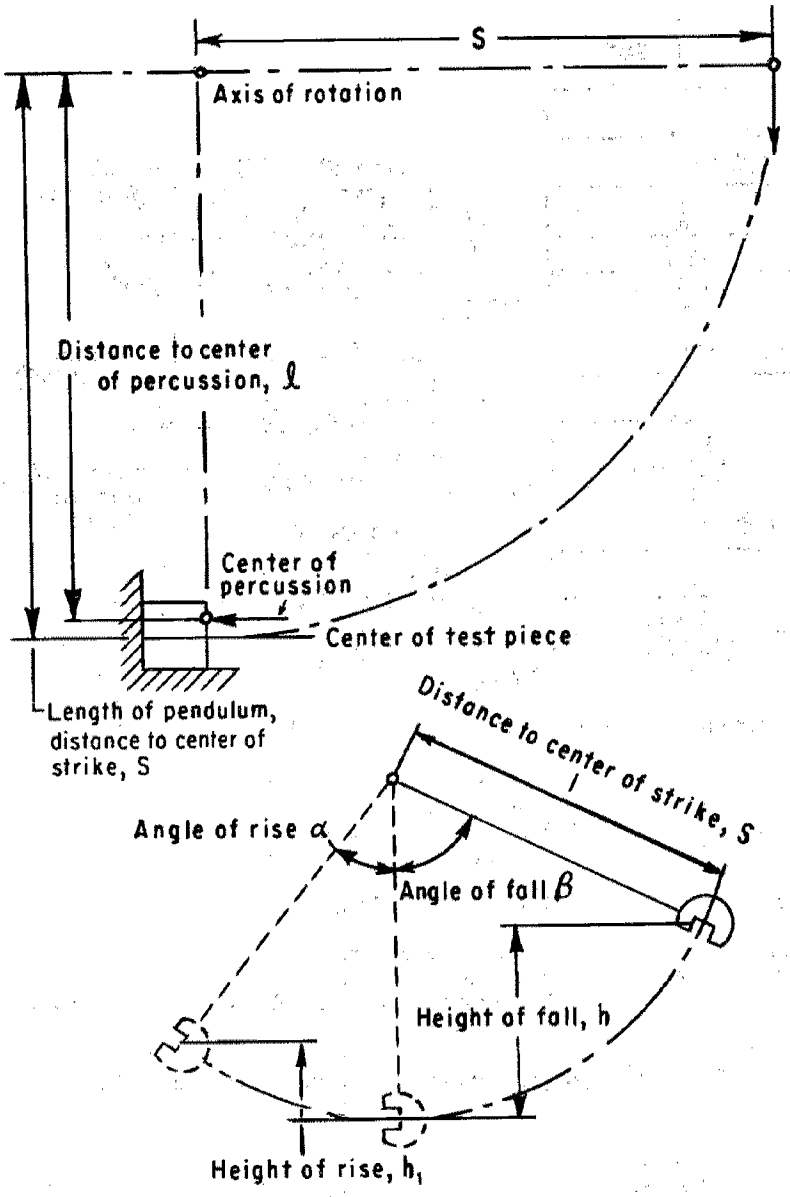
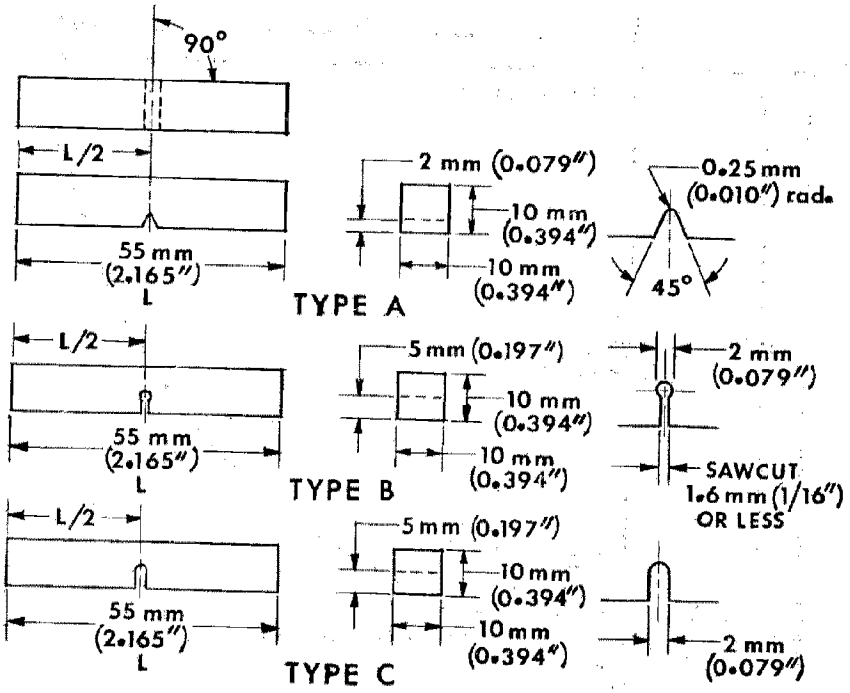


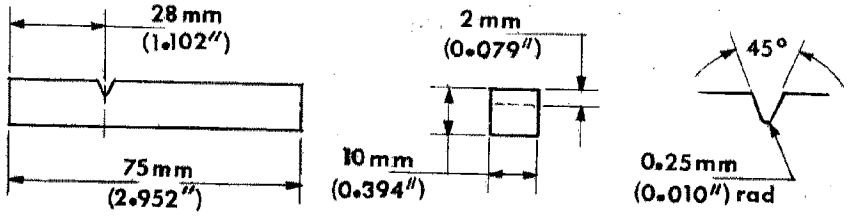
FIG. 5 Dimensions for Calculations



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

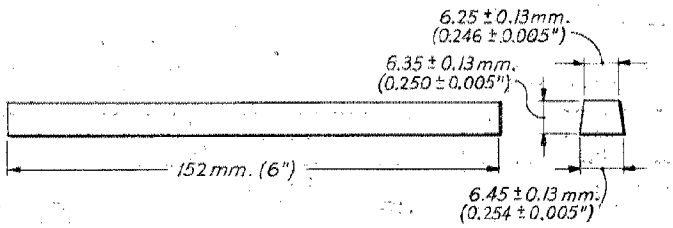
FIG. 6 Charpy (Simple-Beam) Impact Test Specimens, Types A, B, and C



NOTE—Permissible variations shall be as follows:

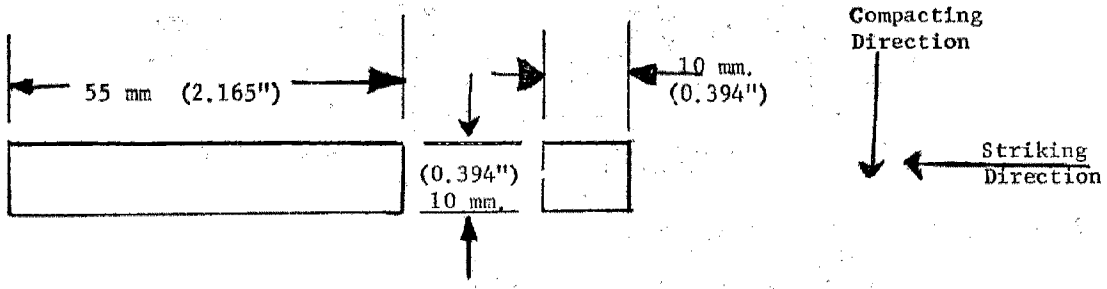
Notch length to edge	90 ±2°
Cross-section dimensions	±0.025 mm (±0.001 in.)
Length of specimen	+0, −2.5 mm (±0, −0.100 in.)
Angle of notch	±1°
Radius of notch	±0.025 mm (±0.001 in.)
Notch depth	±0.025 mm (±0.001 in.)
Adjacent sides shall be at	90° ± 10 min
Finish requirements	2 μm (63 μin.) on notched 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.

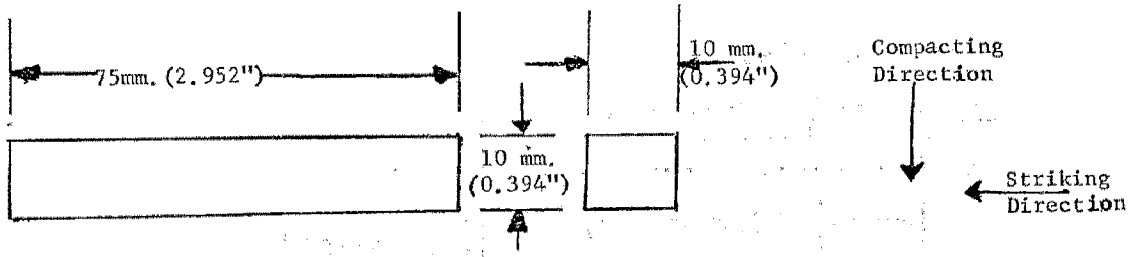
FIG. 8 Simple Beam Impact Test Bar for Die Castings Alloys



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.)

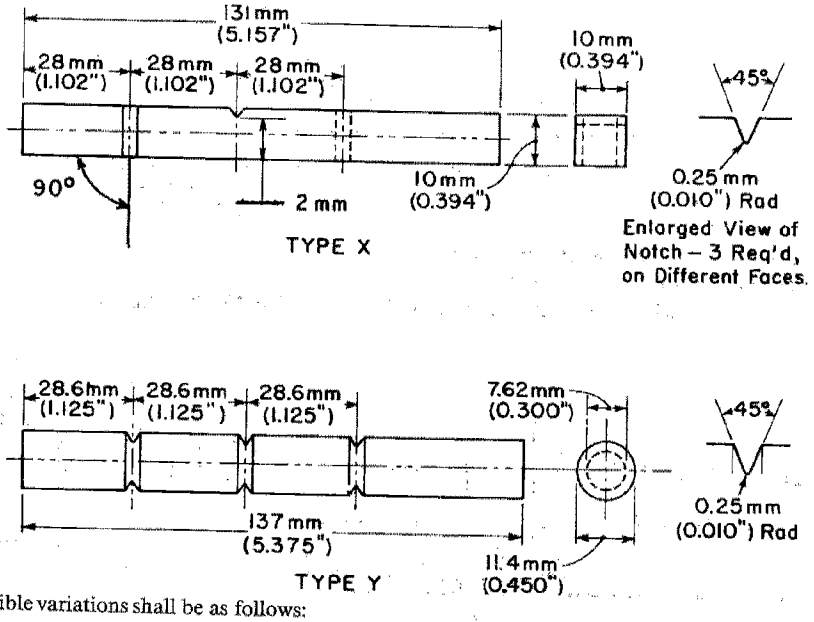
FIG. 9 Charpy (Simple Beam) Impact Test Specimens for Metal Powder Structural Parts



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.)

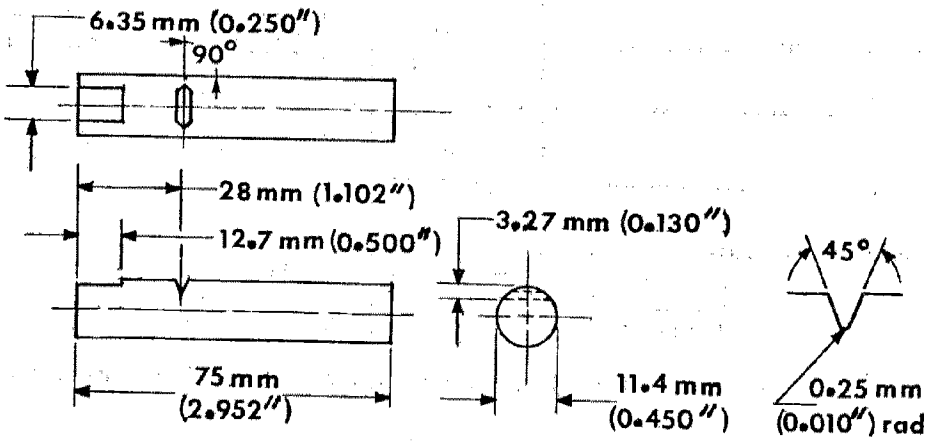
FIG. 10 Izod (Cantilever-Beam) Impact Test Specimen for Metal Powder Structural Parts



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

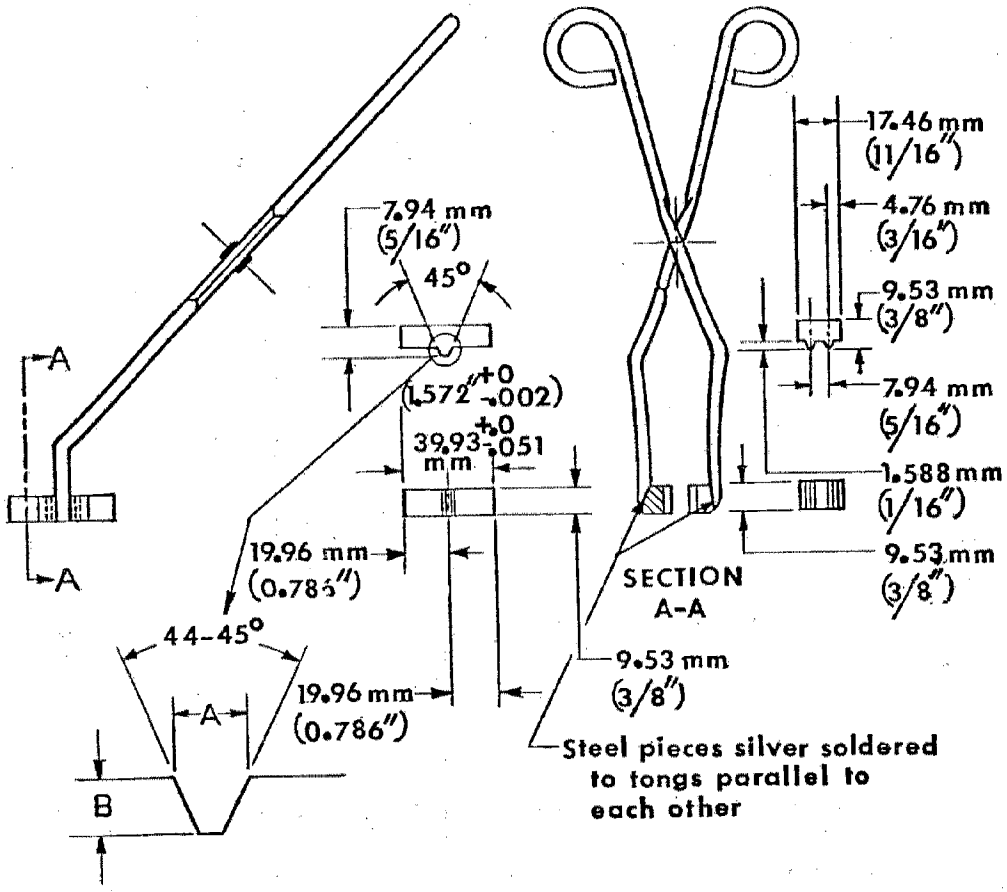


The flat shall be parallel to the longitudinal centerline of the specimen and shall be parallel to the bottom of the notch within 2:1000.

NOTE—Permissible variations shall be as follows:

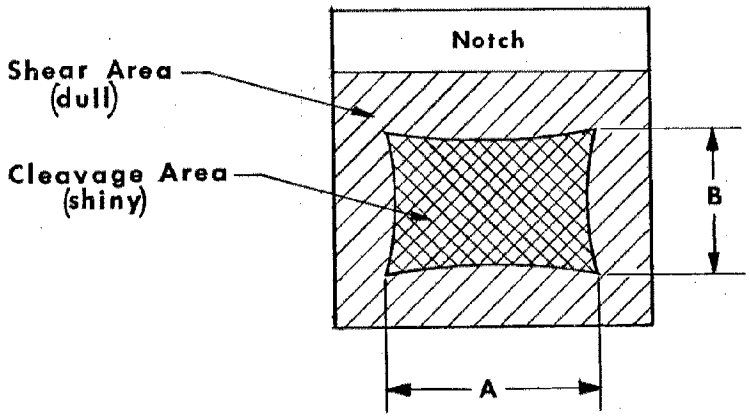
Notch length to longitudinal centerline	±2°
Cross-section dimensions	±0.025 mm (−0.001 in.)
Length of specimen	+0, −2.5 mm (+0 −0.100 in.)
Angle of notch	±1°
Radius of notch	±0.025 mm (±0.001 in.)
Notch depth	±0.025 mm (.130 ±0.001 in.)

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)

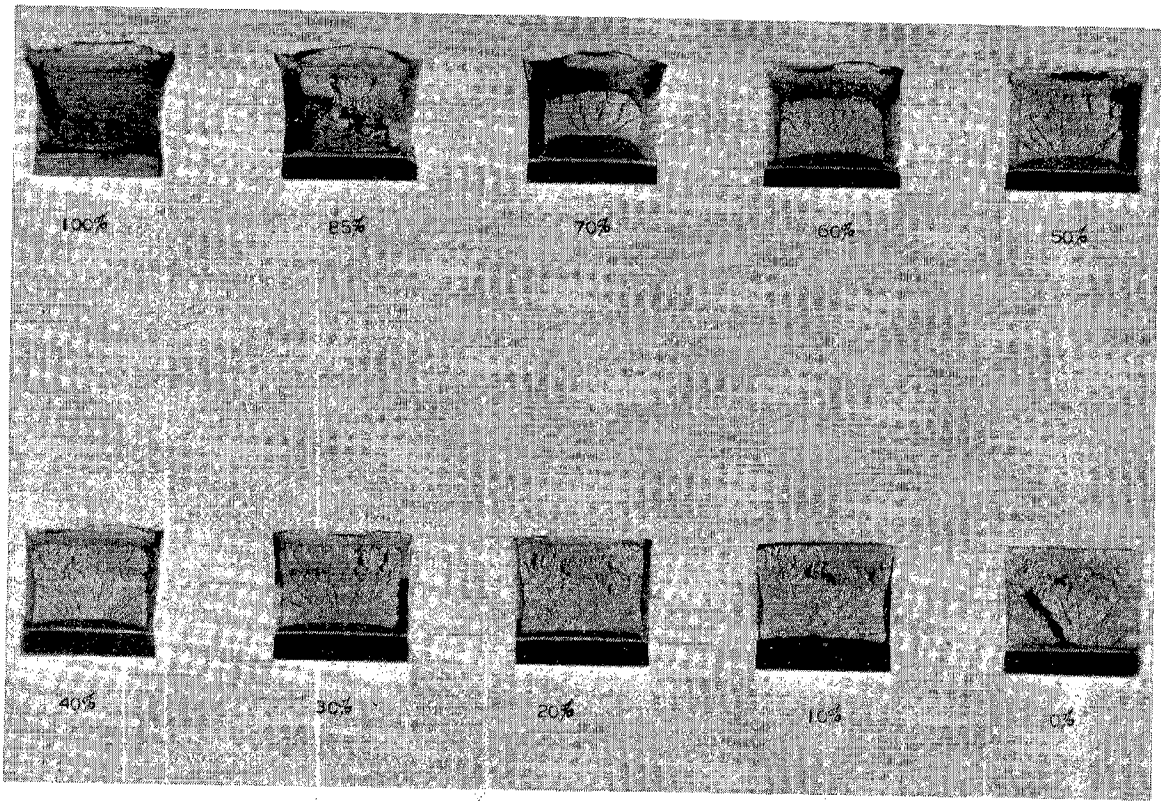
FIG. 13 Centering Tongs for V-Notch Charpy Specimens



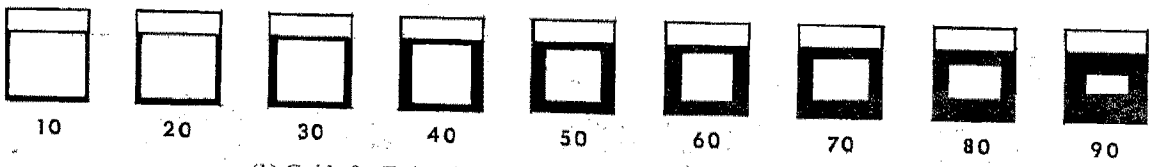
NOTE 1—Measure average dimensions *A* and *B* to the nearest 0.5 mm or 0.02 in.

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

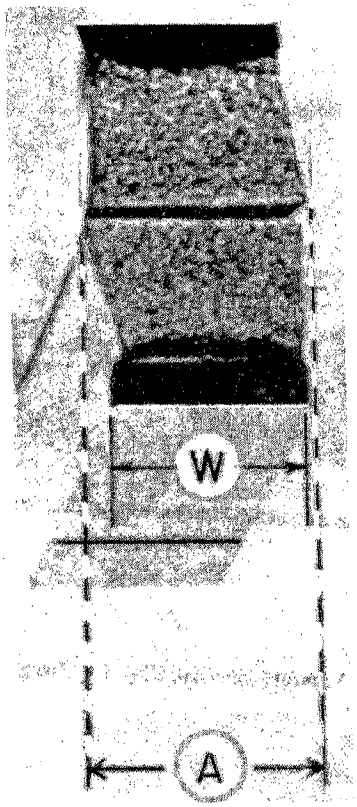


FIG. 16 Halves of Broken Charpy V-Notch Impact Specimen Positioned to Illustrate the Measurement of Lateral Expansion, Dimension *A* and Original Width, Dimension *W*

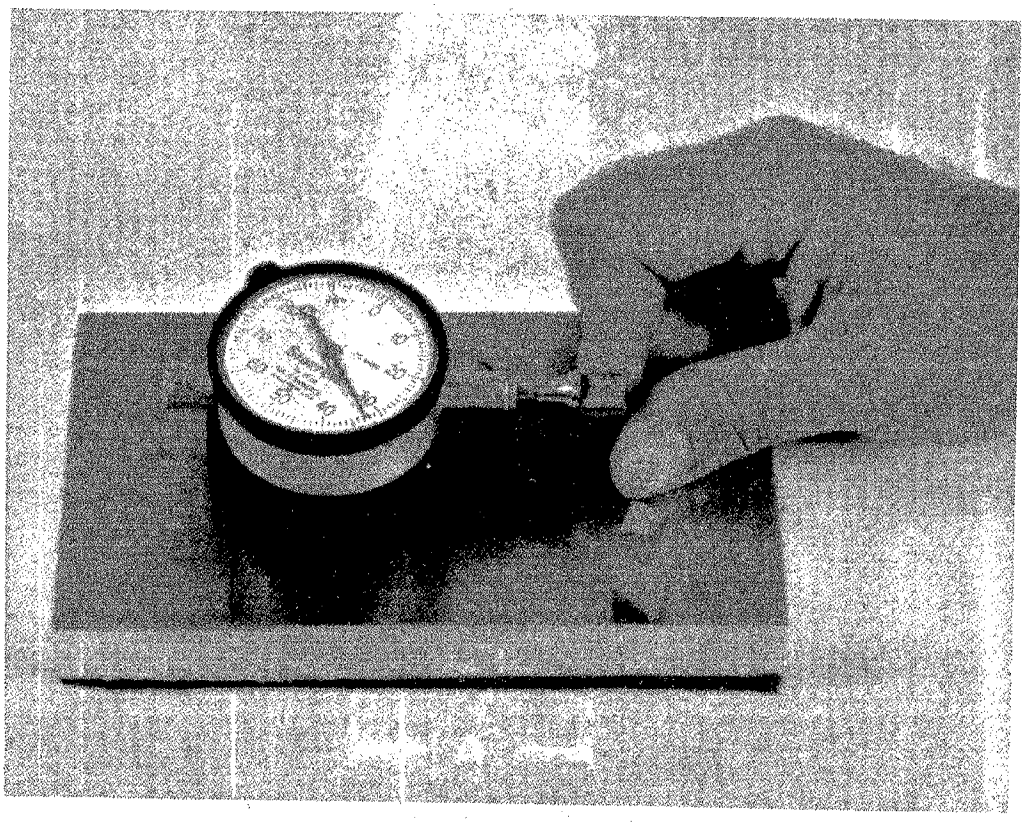


FIG. 17 Lateral Expansion Gage for Charpy Impact Specimens

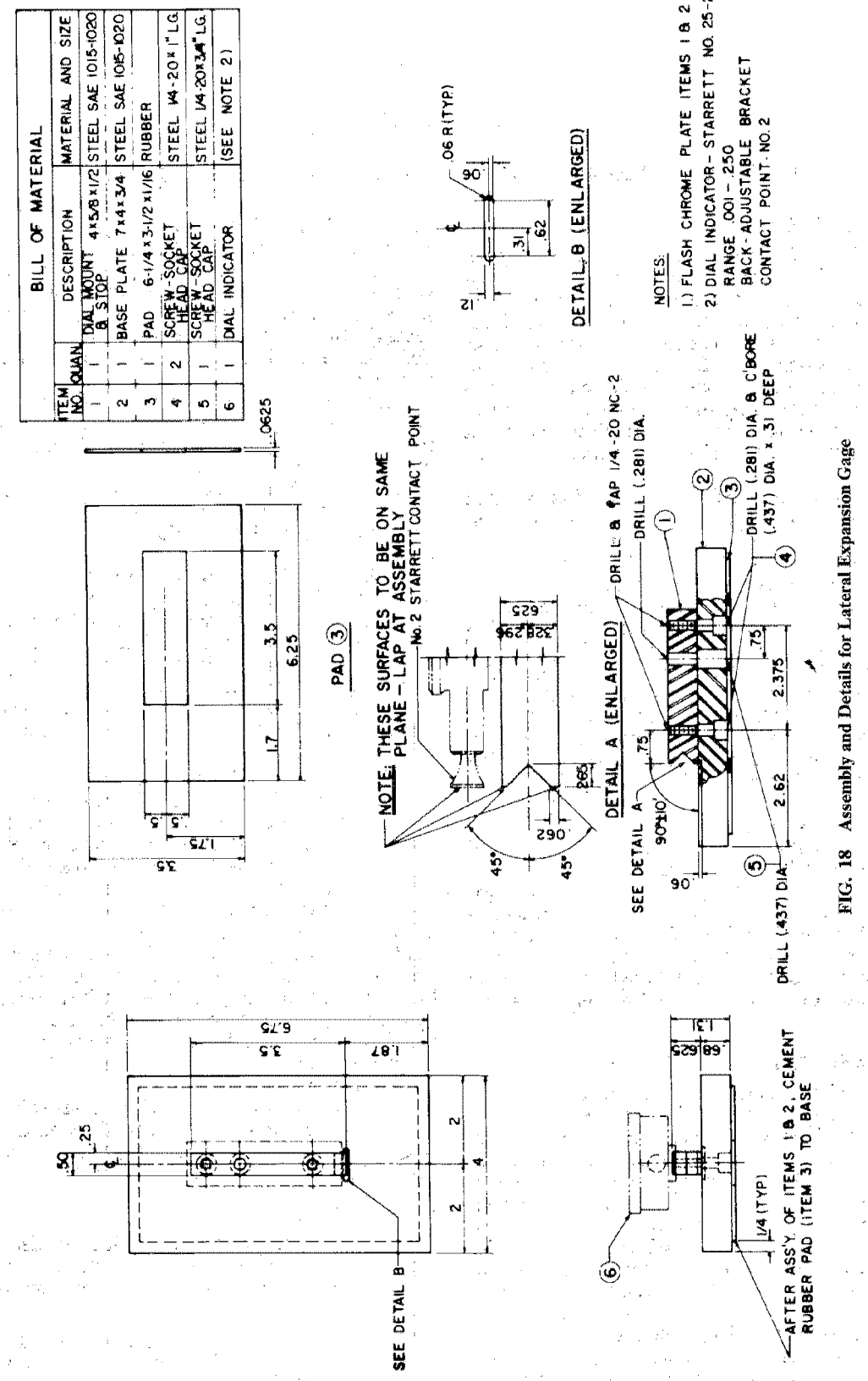


FIG. 18 Assembly and Details for Lateral Expansion Gage

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

[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.).