Exhibit 177
(Part 1)

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Standard Specification
HIGH-STRENGTH LOW-ALLOY COLUMBIUM-VANADIUM STEELS OF STRUCTURAL QUALITY[1]

This Standard is issued under the fixed designation A 572; 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.

This specification has been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1

This specification covers four grades of high-strength low-alloy structural steel shapes, plates, sheet piling, and bars. Grades 42 and 50 are intended for riveted, bolted, or welded construction of bridges, buildings, and other structures. Grades 60 and 65 are intended for riveted or bolted construction of bridges, and for riveted, bolted, or welded construction in other applications. When the steel is used in welded construction, welding procedure shall be suitable for the steel and the intended service.

1.2

For welded bridge construction notch toughness is an important requirement. For this or other applications where notch-toughness requirements are indicated, they shall be negotiated between the purchaser and the producer.

1.3

The use of columbium, vanadium, and nitrogen, or combinations thereof, within the limitations noted in Section 5, shall be at the option of the producer unless otherwise specified. Where designation of one of these elements or combination of elements is desired, reference is made to Supplementary Requirement SI in which these elements and their common combinations are listed as to type. When such a designation is desired, both the grade and type must be specified.

1.4

The maximum thicknesses available in the grades and products covered by this specification are shown in Table 1.

Note—The values stated in inch-pound units are to be regarded as the standard.

2. Applicable Documents

2.1 *ASTM Standard:*

A 6 Specification for General Requirements for Rolled Steel Plates, Shapes, Sheet Piling, and Bars for Structural Use[2]

3. General Requirements for Delivery

3.1

Material furnished under this specification shall conform to the applicable requirements of the current edition of Specification A 6.

4. Process

4.1

The steel shall be made by one or more of the following processes: open-hearth, basic-oxygen, or electric-furnace.

5. Chemical Requirements

5.1

The heat analysis shall conform to the requirements prescribed in Table 2 and in 5.3.

5.2

The steel shall conform on product analysis to the requirements prescribed in Table 2 and 5.3 subject to the product analysis tolerances in Specification A 6.

5.3

Alloy content shall be in accordance with one of the following types:

[1] This specification is under the jurisdiction of ASTM Committee A-1 on Steel, Stainless Steel and Related Alloys, and is the direct responsibility of Subcommittee A01.02 on Structural Steel.

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[2] *Annual Book of ASTM Standards*, Part 4.

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Elements	Heat Analysis, %
Type 1—Columbium[a]	0.005–0.05[b]

[a] Columbium when added either singly of in combination with vanadium shall be restricted to the following unless killed steel is furnished:

Elements	Heat Analysis, %
Type 2—Vanadium	0.01–0.15
Type 3—Columbium[a] (0.05 max, % plus vanadium[c]	0.02–0.15
Type 4—Nitrogen[d] (with vanadium)	0.015 max
[a] Columbium when added either singly of in combination with vanadium shall be restricted to the following unless killed steel is furnished:	

Grades	Maximum Plate and Bar thicknesses, in. (mm)	Structural Shape Size Groupings (Specification A 6, Table A)
42 and 50	¾ (19)	Groups 1 and 2
60 and 65	½ (13)	Group 1
[b] Product analysis limits = 0.004–0.060 %.		
[c] Product analysis limits = 0.01 to 0.16 when columbium and vanadium are used in combination.		
[d] Nitrogen (0.015 max %) when added as a supplement to vanadium shall be reported, and the minimum ratio of vanadium to nitrogen shall be 4 to 1.		

6. Mechanical Requirements

6.1 *Tensile Properties:*

6.1.1

The material as represented by the test specimens shall conform to the tensile properties given in Table 3.

6.1.2

For material under 5/16 in. (7.5 mm) in thickness of diameter, a deduction from the percentage of elongation in 8 in. (200 mm), specified in Table 3, of 1.25 % shall be made for each decrease of 1/32 in. (0.8 mm) of the specified thickness or diameter below 5/16 in.

SUPPLEMENTARY REQUIREMENTS

The following supplementary requirement shall apply when specified in the order or contract:

S1. Types

S1.1

When a purchaser prefers to designate the specific elements (columbium, vanadium, nitrogen, or combinations thereof), one of the types listed below shall be specified. The type in addition to the grade must be shown on the order.

Type 1	—	Columbium
Type 2	—	Vanadium
Type 3	—	Columbium and vanadium
Type 4	—	Vanadium and nitrogen

S1.2

The composition limits of Section 5 shall apply for any of these types.

Standardized supplementary requirements for use at the option of the purchaser are listed in Specification A 6. Those which are considered suitable for use with this specification are listed below by title.

S14. Bend Test.

S18. Maximum Tensile Strength.

TABLE 1 Maximum Product Thickness

Grade	Yield Point, min		Maximum Thickness or Size			
			Plates and Bars		Structural Shapes Groups[b]	Sheet Piling
	psi	Mpa	in.	mm		
42[a]	42 000	290	6	152.4	all	all
50[a]	50 000	345	2	50.8	all	all

[a] In the above tabulation, Grades 42, 50, and 60 are the yield point levels, most closely approximating a geometric progression pattern between 36 000 psi, min, yield point steels covered by Specification A 36, for Structural Steel[2] and 100 000 psi, min, yield, strength steels covered by Specification A 514, for High-Yield-Strength, Quenched and Tempered Alloy Steel Plate, Suitable for Welding.[2]

[b] See Specification A 6.

Grade	Yield Point, min		Maximum Thickness or Size			
			Plates and Bars		Structural Shapes Groups[b]	Sheet Piling
	psi	Mpa	in.	mm		
60[a]	60 000	415	1¼	31.8	1 and 2	not available
65	65 000	450	1¼	31.8	1	not available

[a] In the above tabulation, Grades 42, 50, and 60 are the yield point levels, most closely approximating a geometric progression pattern between 36 000 psi, min, yield point steels covered by Specification A 36, for Structural Steel[2] and 100 000 psi, min, yield, strength steels covered by Specification A 514, for High-Yield-Strength, Quenched and Tempered Alloy Steel Plate, Suitable for Welding.[2]

[b] See Specification A 6.

TABLE 2 Chemical Requirements[a]

(Heat Analysis)

Diameter Thickness, or Distance Between Parallel Faces, in. (mm)	Grade	Carbon, max, %	Manganese,[b]	Phosphorus, max, %	Sulfur, max, %	Silicon[c]	
						Plates to 1½-in. (38.1-mm) Thick, Shapes to 426 1b/ft (634 kg/m), Sheet Piling, and Bars[d]	Plates Over 1½-in. (38.1 mm) Thick and Shapes Over 426 1b/ft (634 kg/m)
						max, %	range, %
6 (152)	42	0.21	1.35	0.04	0.05	0.40	0.15 −0.40
2 (51)	50	0.23	1.35	0.04	0.05	0.40	0.15 −0.40
1¼ (31.8)	60	0.26	1.35	0.04	0.05	0.40	...
>½−1¼ (12.7−31.8)	65	0.23	1.65	0.04	0.05	0.40	...
≤½ (12.7)[e]	65	0.26	1.35	0.04	0.05	0.40	...

[a] Copper when specified shall have a minimum content of 0.20 % by heat analysis (0.18 % product analysis).

[b] Manganese, minimum by heat analysis of 0.80 % (0.75 % product analysis) shall be required for all plates over ⅜ in. (9.5 mm) in thickness; a minimum of 0.50 % (0.45 % product analysis) shall be required for plates ⅜ in. and less in thickness, and for all other products. The manganese to carbon ratio shall not be less than 2 to 1.

Diameter Thickness, or Distance Between Parallel Faces, in. (mm)	Grade	Carbon, max, %	Manganese,[b]	Phosphorus, max, %	Sulfur, max, %	Silicon[c]	
						Plates to l½-in. (38.1-mm) Thick, Shapes to 426 1b/ft (634 kg/m), Sheet Piling, and Bars[d]	Plates Over 1½-in. (38.1 mm) Thick and Shapes Over 426 1b/ft (634 kg/m)
						max, %	range, %

[c] Silicon content in excess of 0.40 % by heat analysis must be negotiated.							
[d] Bars over 1½ in. (38.1 mm) in diameter, thickness, or distance between parallel faces, shall be made by a killed steel practice.							
[e] An alternative chemical requirement with a maximum carbon of 0.21 % and a maximum manganese of 1.65 % is permissible with the balance of the elements as shown in Table 2.							

TABLE 3 Tensile Requirements[a]

Grade	Yield Point, min		Tensile Strength, min		Minimum Elongation,[a, c, d] %	
	psi	Mpa	psi	Mpa	in 8 in. or 200 mm	in 2 in. or 50 mm
42	42 000	290	60 000	415	20	24
50	50 000	345	65 000	450	18	21
60	60 000	415	75 000	520	16	18
65	65 000	450	80 000	550	15	17

[a] For plates wider than 24 in. (610 mm), the test specimen is taken in the transverse direction. See 11.2 of Specification A 6.

[b] Elongation not required to be determined for floor plate.

[c] For wide flange shapes over 426 1b/ft elongation in 2 in. (50 mm) of 19 % minimum applies.

[d] For plates wider than 24 in. (610 mm), the elongation requirement is reduced two percentage 0 points for Grades 42 and 50, and three percentage points for Grades 60 and 65.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, Pa. 19103, which will schedule a further hearing regarding your comments. Failing satisfaction there, you may appeal to the ASTM Board of Directors.

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Designation: C 518 – 91

Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus[1]

This standard is issued under the fixed designation C 518; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue that has been adopted by the Department of Defense.

INTRODUCTION

This test method presents a comprehensive assemblage of information and requirements on the complex problem of the measurement of the thermal transmission properties of insulations using a heat flow meter apparatus. For convenience, it is organized in four major parts:

General Considerations (Sections 1 through 6)

Apparatus and Calibration (Sections 7 through 9)

Test Procedures and Report (Sections 10 through 14)

Annexes (A1 and A2)

This test method shall be used in conjunction with Practice C 1045.

Many advances have been made in thermal insulation technology, both in measurement techniques and in improved understanding of the principles of heat flow through such materials. These advances have prompted revisions in the conceptual approaches to the measurement of the thermal transmission properties, see References (**1**), (**2**), (**3**), and (**4**).[2] All users of this test method should be aware of these concepts.

1. Scope

1.1

This test method covers the measurement of steady state thermal transmission through flat slab specimens using a heat flow meter apparatus.

1.2

This is a comparative (or secondary) method of measurement since specimens of known thermal transmission properties must be used to calibrate the apparatus. Properties of the calibration specimens must be traceable to an absolute measurement method, and should be obtained from or traceable to a recognized national standards laboratory.

1.3

The test method is applicable to the measurement of thermal transmission through a wide range of specimen properties and environmental conditions. The method has been used at ambient conditions of 10 to 40°C with thicknesses up to approximately 250 mm, and with plate temperatures from—195°C to 540°C at 25 mm thickness (Refs. (**5**, **6**)).

1.4

This test method may be used to characterize material properties which may or may not be representative of actual conditions of use. Other test methods such as Test Methods C 236 or C 976 should be used if needed.

1.5

To meet the requirements of this test method the thermal resistance of the sample must be greater than 0.10. $K \cdot m^2/W$ in all directions.

1.6

It is not practical in a test method of this type to try to establish details of construction and procedures to cover all contingencies that might offer difficulties to a person without pertinent technical knowledge. Thus users of this test method shall have sufficient knowledge to satisfactorily fulfill their needs. For example, knowledge of heat transfer principles, low level electrical measurements, and general test procedures is required.

1.7

Standardization of this test method is not intended to restrict in any way the future development of improved or new methods or procedures by research workers.

1.8

Since the design of a heat meter apparatus is not a simple matter, a procedure for qualifying an apparatus is given in 7.8.

1.9

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

[1] This test method is under the jurisdiction of ASTM Committee C-16 on Thermal Insulation and is the direct responsibility of Subcommittee C16.30 on Thermal Measurement.

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[2] The boldface numbers in parentheses refer to the list of references at the end of this test method.

[3] *Annual Book of ASTM Standards*, Vol 04.06.

2. Referenced Documents

2.1

ASTM Standards:

C 167	Test Methods for Thickness and Density of Blanket or Batt Thermal Insulations[3]
C 168	Terminology Relating to Thermal Insulating Materials[3]
C 177	Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties By Means of the Guarded-Hot-Plate Apparatus[3]
C 236	Test Method for Steady-State Thermal Performance of Building Assemblies by Means of a Guarded Hot Box[3]
C 519	Test Method for Density of Fibrous Loose-Fill Building Insulations[3]
C 687	Practice for Determination of the Thermal Resistance of Loose-Fill Building Insulation[3]
C 976	Test Method for Thermal Performance of Building Assemblies by Means of a Calibrated Hot Box[3]
C 1045	Practice for Calculating Thermal Transmission Properties from Steady-State Heat Flux Measurements[3]
C 1132	Practice for Calibration of the Heat Flow Meter Apparatus[3]
E 230	Temperature-Electromotive Force (EMF) Tables for Standardized Thermopcouples[4]
E 691	Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[5]

3. Terminology

3.1

Definitions—The terms and symbols are as defined in Definitions C 168 with exceptions included as appropriate.

3.2

Symbols and Units—The symbols used in this test method have the following significance:

λ	=	thermal conductivity, W/m · K,
C	=	thermal conductance, W/m^2 · K,
R	=	thermal resistance, m^2 · K/W,
q	=	

		heat flux, W/m^2
Q	=	heat flow, W,
A	=	area measured on a selected isothermal surface, m^2,
L	=	thickness of specimen measured along a path normal to isothermal surfaces, m,
T_m	=	mean temperature, K
ΔT	=	temperature difference, K
ρ	=	density of the material as tested, kg/m^3,
S	=	sensitivity of heat flow transducer (W/m^2)/V,
E	=	heat flow transducer output, V,
T_h	=	temperature of hot plate surface, K, and
T_c	=	temperature of cold plate surface, K.

4. Summary of Test Method

4.1

The heat flow meter-apparatus establishes steady state unidirectional heat flux through a test specimen between two parallel plates at constant but different temperatures. By appropriate calibration of the heat flux transducer(s) with calibration standards and by measurement of the plate temperatures and plate separation, Fourier's law of heat conduction is used to calculate thermal conductivity, thermal resistance, or resistivity.

4.2

The accurate use of the test method is limited by the capability of the apparatus to reproduce unidirectional constant heat flux density in the specimens, and by the precision in the measurement of temperature, thickness, EMF produced by heat flux transducer, etc.

5. Significance and Use

5.1

This test method provides a rapid means of determining thermal properties with high precision and as such is useful in research and quality control work.

5.2

The thermal transmission properties of specimens of a given material or product may: *(1)* vary due to variability of the composition of the material; (2) be affected by moisture or other conditions; (3) change with time; *(4)* change with mean, temperature and temperature difference, and *(5)* depend upon the prior thermal history. It must be recognized, therefore, that the selection of typical values of thermal transmission properties representative of a material in a particular application should be based on a consideration of these factors and will not necessarily apply without modification to all service conditions. As an example, this test method provides that the thermal properties shall be obtained on specimens that do not contain any free moisture although in service such conditions may not be realized. Even more basic is the dependence of the thermal properties on variables such as mean temperature and temperature difference. These dependencies should be measured or the test made at conditions typical of use.

5.3

Special care must be taken in the measurement procedure for specimens exhibiting appreciable inhomogeneities, anisotropies, rigidity, or especially high or low resistance to heat flow (see Practice C 1045). Special considerations are necessary when the measurements are conducted at either high or low temperatures, in a vacuum or in hazardous gases.

5.4

The determination of the accuracy of the method for any given test is a function of the apparatus design, of the related instrumentation and of the type of specimens under test (see Section 9), but this method is capable of determining thermal transmission properties within ±2 % of those determined by Test Method C 177 when the ambient temperature is near the mean temperature of the test (T (ambient) = T (mean) ±1°C), and in the range of 10 to 40°C.

5.4.1

Where certification of measurement by this test method is required, the apparatus shall have the capabilities required in 7.8 and one of the following procedures must be followed.

5.4.1.1.

The testing laboratory apparatus shall be calibrated within 24 h before or after the certification test using calibration standards that have been issued or whose values have been established by a recognized National Standards Laboratory not more than five years prior to the certification date. The average of two calibrations shall be used as the calibration factor and the specimen(s) certified with this average value. When the change in calibration factor is greater than one percent, the standard specimen shall be retested and a new average calculated. If the change in calibration factor is still greater than 1 % the apparatus must be calibrated using the procedure in Section 8.

5.4.1.2

Where both the short and long term stabilities of the apparatus have been proven to be better than 1 % of the reading, see 8.4, the apparatus may be calibrated at less frequent intervals, not exceeding 30 days. The specimens so tested cannot be certified until after the calibration following the test and then only if the change in calibration, factor from

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

[5] *Annual Book of ASTM Standards;* Vol 14.02.

164   the previous calibration test is less than 1 %. When the change in calibration is greater than one percent, test results from this interval shall be considered void and the tests repeated in accordance with 5.4.1.1.

5.4.2

The precision (repeatability) of measurements made by the heat flow meter apparatus calibrated as in 5.4.1 are normally much better than ±1 % of the mean value. This precision is required to identify changes in calibration and is desirable in quality control applications.

6. Limitations to Test Method

6.1

Limitations Due to Apparatus:

6.1.1

The apparatus shall not be used at temperatures, thicknesses, or resistances, other than those within the range of the calibration, unless it can be shown that there is no difference in accuracy.

6.1.2

The apparatus must be capable of maintaining at least a 10°C temperature difference across the specimen for the duration of the test, unless a smaller ΔT is a requirement of a particular test.

6.2

Limitations Due to the Specimens:

6.2.1

The specimens under test may also limit the use of the test method and these limitations are outlined in Practice C 1045.

7. Apparatus

7.1

The construction details given in this section should be understood by the user of this test method. While it is mandatory that these details be carefully followed when constructing an apparatus, it behooves the user to verify that the equipment was built as specified. Serious errors of measurement may result from this oversight.

7.2

General:

7.2.1

The general features of a heat flow meter apparatus with the specimen or the specimens installed are discussed in Annex Al. A heat flow meter apparatus consists of two isothermal plate assemblies, one, or more heat flux transducers, equipment to measure and control temperature, thickness, and the output of the heat flux transducers and equipment to control the environmental conditions when needed. Each configuration will yield equivalent results if used within the limitations stated in this test method. There are distinct advantages for each method in practice and these are discussed in Annex Al.

7.2.2

The working surfaces of the plates and the heat flux transducer (that is, the surfaces making contact with the specimens) shall be painted or otherwise treated to have a total hemispherical emittance of greater than 0.84 at operating temperatures (Note 1).

Note 1—Hard anodizing of aluminum produces a surface with a total hemispherical emittance of approximately 0.85. Several paints are available which when applied as directed, produce a total hemispherical emittance of approximately 0.86.

7.3

Plate Assemblies, Hot and Cold—The two plate assemblies provide isothermal surfaces in contact with either side of the test specimen. The assemblies consist of heat source or heat sink, a high conductivity surface,

means to measure surface temperature, and means of support. A heat flux transducer may be attached to one, both or neither plate assembly, depending upon the design. In all cases the area defined by the dimensions of the heat flux transducer is called the metering area and the remainder of the plate is the guard area.

7.3.1

The portion of each plate assembly in contact with the heat flux transducer, or if a heat flux transducer is not mounted on the plate assembly, the portion in contact with the sample, shall consist of a high conductivity material, usually metal, known as the isothermal plate. The isothermal plate shall be supported so as to minimize temperature gradients across its surface. For a 2% total uncertainty, deviations from isothermal conditions over the plate surface of no more than ±0.5 K shall be allowed.

7.3.2

A means shall be provided to maintain the temperature of the isothermal plates at the desired level. Examples are fluid baths, electrical heaters and combinations of these (see Refs (**7, 8, 9**)).

7.3.3

If a heat flux transducer is mounted at the midplane of the specimen then means shall be provided to determine the average temperature of the transducer in order to apply temperature corrections to the calibration, except when test temperatures are equal to those used in calibration, in which case no correction is required.

7.3.4

Means shall be provided to measure the temperature of the surfaces of the heat flux transducer or plates in contact with the specimen.

7.3.5

The surface plates shall be sufficiently rigid to maintain flatness and parallelism to within one-tenth the accuracy required of the final result. For measurements on non-rigid materials and an allowable total uncertainty of ±2 %, a flatness and parallelism of 0.2 % of the maximum linear dimension of the heating unit is thus required (Note 2), One recommended way to check this is to generate a L-map over the metering area. This is discussed fully in Ref **(10).**

Note 2—The planeness of the surface can be checked with a straightedge, of a length greater than the width or diameter of the unit, held against the surface and viewed with a light behind the straightedge. Departures as small as 25 μm are readily visible, and larger departures can be measured using shimstock or thin paper.

7.3.5.1

The parallelism of the plates must be maintained for several reasons. In most cases it is the plate separation which is measured in order to determine specimen thickness. Furthermore the plate parallelism is important in maintaining consistent surface contact with specimens in repeat testing (such as calibration), and is required to maintain a uniform temperature difference across the specimen(s).

7.3.6

The rigidity, flatness, and parallelism of the plates may impede the testing of rigid specimens where it is not possible to obtain good surface contact. In such cases the use of a thin sheet of suitable homogeneous material may be interposed between the specimen and the plate surfaces. This thin sheet should have a low thermal resistance relative to the specimen. The resistance of the thin sheet should be determined using a Test

Method C 177 apparatus. The resistance of the composite sandwich (sheet-rigid specimen-sheet) is then determined and the value of the sheet resistance (determined in a separate test) subtracted from the total resistance measured.

7.4

Temperature Measuring and Control Systems:

7.4.1

Any proven device possessing adequate accuracy may be used for measurement and control of the temperatures in the apparatus.

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7.4.2

Surface temperatures are often measured by permanently mounting temperature sensors, such as thermocouples, RTD's or thermistors, in grooves or flush with the working surfaces on both plates.

7.4.3

Many existing systems utilize a minimum of two temperature sensors to determine the average temperature of each isothermal surface. However, to determine the actual surface temperature variations under typical operating conditions, the temperature must be sampled at considerably more than two points. This is especially true when the specimen surface temperature itself must be measured or when the specimen is highly inhomogeneous. Therefore, no strict specification is given as to the number of temperature sensors that must be used for each surface. However, the user must report the uncertainty of the temperature measurement, including the component, due to temperature non-uniformity across the surface. This determination is probably best done with a multijunction thermocouple in ad-hoc experiments and not part of every measurement. (See Ref (**10**).) In some cases where temperature mapping of the plate surfaces has indicated high uniformity under all conditions of use, one thermal sensor per surface has been satisfactorily used.

7.4.3.1

Special precautions should be taken to ensure that the temperature sensors are thermally anchored to the surface to be measured and that the temperature gradients along the wires leading to the sensors are minimized. If sensors on opposing surfaces are connected differentially, they must be electrically insulated from the plates with a resistance of $1\text{M}\Omega$ or greater (**11,12**).

7.4.3.2

Thermocouples mounted in the surfaces of the plates should normally be made of wire not larger than 0.51 mm in diameter (No. 24 B & S gage). Thermocouples placed against or set into the surfaces of the specimens should be made of wire not larger than 0.25 mm in diameter (No. 30 B & S gage). For information concerning sensitivity and accuracy of thermocouples in the cryogenic temperature range, and installation, see Refs (**6**) and (**13**).

7.4.3.3

Temperature sensors should be calibrated to an accuracy equivalent to that for thermocouples conforming to Tables E 230. The temperature measuring system shall have an equivalent accuracy. The precision of the temperature measuring system may need to be better than this to detect the effect of drift on the results as discussed in 7.9. Tables E 230 lists both "standard" and "special" limits on the accuracy of thermocouples. The

accuracy required by a heat flow meter apparatus can best be determined by carrying out an error analysis (see Section 9), and then calibrating the temperature sensors to the degree required.

7.5

Heat Flux Transducer—The heat flux transducer is a device including both the metering and guard areas which measures heat flux by sensing the temperature difference across a core of appropriate thermal resistance and thermal stability. The metering area usually consists of a number of differentially connected temperature sensors placed on each face of the core and surface sheets to protect the assembly. The guard area is usually constructed of the same material as the core. A properly designed transducer will have a sensitivity that is essentially independent of the specimens' thermal properties (**14, 15**).

7.5.1

Types of Heat Flux Transducers—The type of heat flux transducer described in this test method is called a gradient type. This type consists of a slab of material, the "core," across which the temperature gradient is measured, normally with a thermopile.

7.5.2

Temperature Difference Detector—The temperature difference across the core material shall be measured with a sensitive stable temperature detector. The main transducer surfaces are assumed to be isothermal, so that heat flow will be normal to them. Precautions must be taken to limit the effect of heat flow through the leads on the output of the temperature difference detector. Often the heat flux transducer is also instrumented to measure one of the surface temperatures of the specimen(s).

7.5.3

Surface Sheets—To prevent damage to the temperature difference detector that will affect its calibration, both surfaces shall be covered with a layer of material as thin as is compatible with protection from thermal shunting of the temperature difference detector wires. The working surfaces of the heat flux transducer shall be smoothly finished to conform to the desired geometric shape to within the limits of 7.3.

7.6

Plate Separation, Specimen Thickness—A means shall be provided to determine the average separation between the heating and cooling plate surfaces during operation. Rigid specimens generally act as the spacers themselves and plate separation is determined by their thickness at operating temperature. In this case a small constant force is generally applied to hold the plates against the specimen. It is unlikely that a pressure greater than 2.5 kPa (approximately 50 lbf/ft^2) will be required. For easily compressible specimens, small stops interposed between the corners of the hot and cold plates, or some other positive means shall be used to limit the compression of the specimens, (Note 3). Provision must be made for checking the linearity of any thickness measuring system.

Note 3—Because of the changes of specimen thickness possible as a result of temperature or compression by the plates, it is recommended that specimen thickness be measured in the apparatus, at the existing test temperature and compression conditions whenever possible.

7.7

Edge Insulation—Heat loss from the outer edges of the heat flow meter apparatus and specimens shall be restricted by edge insulation or by governing the surrounding air temperature or by both methods. The three (different configurations differ in their sensitivity to edge heat losses as is discussed in Annex Al (**2, 4, 16**).

7.7.1

For all three configurations, the sensitivity to edge heat losses is strongly related to the sensitivity of the transducer to temperature differences along its main surfaces and therefore only experimental checks while changing environmental conditions can confirm, for each operating condition, the magnitude of the effect of edge heat losses On measured heat flux. This error should be smaller than 0.5 %. An approximate guideline is to maintain power lost through the edges below 5 % of the power flowing through the specimen.

7.8

Measuring System Requirements for Certification—When the apparatus is used for certification testing, the Measuring system shall have the following capabilities:

7.8.1

The accuracy of the measurement of the temperature difference across the specimens shall be within +0.5 %. The accuracy of the measurement of the output from the

166   heat flux transducer shall be within ±0.6 %.

7.8.2

A voltage sensitivity of better than 0.2 % of the minimum output (from the transducer) to be measured.

7.8.3

Sufficient linearity so that it contributes less than 0. 2 % error at all outputs.

7.8.4

Sufficient input impedance so that it contributes less than 0.1 % error for all readings. One megohm has been found adequate for many apparatuses.

7.8.5

Sufficient stability so that it contributes less than 0.2 % error during the period between calibrations, or 30 days, whichever is greater.

7.8.6

Adequate noise immunity so that less than 0.2 % rms noise occurs in the readings.

7.8.7

Where direct readout equipment is used, adequate provision shall be made for calibration of the electronic circuitry, independent of the remainder of the apparatus, and shall contribute no more than 0.2 % error, for each variable.

7.9

Proven Performance—Any heat flow meter apparatus that is new or has been modified must be tested for the following characteristics and corrections shall be made where a change of greater than one percent occurs in

the transducer output due to the effects of 7.9.1 to 7.9 over the range of operation and are recommended for changes of 0.3 % over the range of operation.

7.9.1

Zero Offset—If there is any output from the transducer for zero heat flux, this may be due to:

7.9.1.1

Electrical contact resistance on a transducer with low output: improve connections to eliminate the problem as this type of output may be temperature dependent.

7.9.1.2

Also check grounding circuits because such a signal may be due to AC pickup in the leads from the transducer. If after checking 7.9.1.1 and 7.9.1.2 there is still a zero off-set, it may be possible to correct for this by assuring that the calibration curve of output versus heat flux is linear over the range of operating conditions.

7.9.2

Sensitivity to warm or cold plate temperature non-uniformity. Check for temperature non-uniformity under all operating conditions.

7.9.3

Drift in the transducer due to material aging or delamination. This can be used to determine the required calibration frequency.

7.9.4

Temperature coefficient of the transducer sensitivity, as this depends on the type of temperature detectors used in the temperature difference detector (thermocouple materials used in the thermopile) and the type of material used for the transducer core. If it is found that the sensitivity is temperature dependent, a curve of sensitivity versus temperature shall be developed and used to correct measurement data.

7.9.5

Heat flow down the leads. One possible way to check for this is to move one's hand across the surface of the transducer between the metering area to the point where the leads exit the plate assembly, while observing the transducer output. In a well designed plate or transducer assembly there should be no observable output from the transducer except in the metering area.

7.9.6

Effect of the thermal conductivity of the specimen on the sensitivity of the transducer. A "thermal shorting" effect between elements caused by low thermal resistance between the sensors of the thermopile or a funneling of heat through the sensors can change the sensitivity of the meter. This can best be tested by running specimens with known widely different thermal conductivities.

7.9.7

Effect of loading pressure on the transducer sensitivity, if the transducer core is flexible.

7.9.8

The test results obtained by this method can only be assured if the limitations of the apparatus are known. To establish the limitations, one must prove the performance by comparing the results with materials of similar thermal properties previously tested on a guarded hot plate apparatus as those to be evaluated.

7.9.8.1

A single point of reference may lead to serious errors. It is best to select a range of transfer standards having known thermal transmission properties which cover the range of values to be tested, in both resistance and thickness.

7.9.8.2

If the apparatus is to be used at thicknesses greater than that of the available reference materials, a series of calibration measurements must be performed to insure that the equipment does not introduce additional errors which may be due to lateral heat losses or gains brought about by insufficient guarding (**4, 17**).

7.10

Environmental Control—In many applications, it is desirable to control the environment surrounding the test specimen to reduce edge heat losses and it is especially important if the mean test temperature must be below the ambient temperature, in order to avoid condensation on the cold plate.

7.10.1

A cabinet or enclosure surrounding the heat flow meter assembly to maintain the ambient temperature at the mean temperature of the specimen may also be used as a means to maintain the dew point temperature at least 5 K lower than the temperature of the cold plates, that will prevent condensation and moisture pickup by the specimen. This is required when conducting tests at mean temperatures substantially lower than the laboratory air temperature.

7.10.2

Any environmental control system employed in conjunction with a heat flow meter apparatus shall be capable of maintaining its set point condition within ±1°C in temperature, and ±2 % in humidity.

8. Calibration

8.1

The calibration of a heat flow meter apparatus is a very critical operation. Since lateral heat losses or gains of heat are not automatically controlled or eliminated, but only lessened by increasing the size of the guard area and edge insulation, there is no guarantee that the heat losses or gains are negligible under all testing conditions. To ensure that the equipment is performing properly with specimens of different thermal resistances, the apparatus must be calibrated with materials having similar characteristics as the materials to be evaluated.

8.2

See Practice C 1132.

9. Error Analysis

9.1

A complete error analysis is complex. However, it is mandated for compliance with this test method. The performance of this analysis involves consideration of:

9.1.1

Estimates of errors in each individual measurement procedure and propagation of these errors to the final result,

9.1.2

Measurements to determine apparatus sensitivity to intentional deviations from normal operations, and

9.1.3

Measurements on reference materials and participation in round-robin programs.

9.1.4

For a more complete discussion of this error analysis the reader is directed to Test Method C 177, and Ref (**10**).

9.2

To illustrate a procedure of error analysis estimation, consider the operational definition of thermal conductivity:

$$\lambda = Q \cdot L/A \cdot \Delta T$$

The uncertainties in Q, A, L, and ΔT ($\delta Q, \delta A, \delta L$, and $\delta \Delta T$) can be used to form the uncertainty $S\lambda$ by the usual error propagation formula where the total uncertainty is calculated from the square root of the sums of the squares of the individual standard deviations.

$$S\lambda^2 = (\delta Q/Q)^2 + (\delta A/A)^2 + (\delta L/L)^2 + (\delta \Delta T/\Delta T)^2$$

This equation assumes that the errors in Q, A, etc., are random and independent of each other since other covariance terms are omitted. This is valid here if different instrumentation is used for measurements on each of the variables.

9.2.1

An alternative approach, which generally leads to a larger calculated uncertainty, is to sum the upper limits of the individual uncertainties (**10**).

9.2.2

Care must be taken to properly evaluate all of the uncertainties in the variables Q, A, etc. For example, the obvious sources of error in Q are those causing extraneous transverse heat flow along leads and deviations from one dimensional heat flow. However, an often neglected but important quasi-heat leak is that caused by a temperature drift of the transducer itself, or in the heater, or both.

9.2.2.1

This can readily be estimated from the heat capacity of the transducer assembly and the drift detection limit of the measurement system. The error in ΔT, $\delta\Delta T$, can be caused by calibration errors and measurement errors but also by incorrect placement, incorrect, thermal anchoring, and disturbances introduced by the thermocouple itself.

9.3

Numerous experiments can be performed to determine, the sensitivity of the measurement results to deviations from normal operating conditions. This sensitivity combined with the probable deviation under normal operating conditions can be used to estimate the error from this source. As one example, the effect of imperfect guard balance control can be determined by purposely offsetting the guard (if this is possible) by a sufficient δT in both directions and measuring the, differences in the measured output.

9.3.1

Care should be taken to not use such large offsets that nonlinear effects occur in the specimens. These results combined with the probable value of the offset during normal operation yield the error due to imperfect guard balance.

9.4

Finally, measurements should be performed on accepted reference materials, if available, to qualify the apparatus. Care should be taken here to ensure that the reference materials have characteristics similar to the specimens to be measured and that the uncertainties of the standards themselves are known.

9.5

The total estimated imprecision can be listed in a table of errors such as shown in Table 1. This table is shown as an example only and does not represent any one particular heat flow meter apparatus since the errors will be specific to each apparatus.

10. Test Procedures

10.1

Foreword on Testing Procedures—The simplicity of this test procedure may lead one to overlook very important factors which may affect the results. To ensure accurate measurement, the operator must be fully instructed in the operation of the equipment. Furthermore, the equipment must-be properly calibrated with reference materials having similar heat transfer characteristics. It is also necessary that the specimen be properly prepared for the evaluation.

10.2

Sampling and Preparation of Specimens:

10.2.1

Test Specimens—One or two-piece specimens may be used, depending on the configuration selected for the test. Where two pieces are used, they shall be selected from the same sample to be essentially identical in constitution, thickness, and density. For loose fill materials, the method specified in the material specification or in Practice C 687 shall be used to produce a specimen or specimens of the desired density.

10.2.2

Selection of Specimens—The specimen or specimens shall be of such size as to cover the heat flux transducer, cold plate and warm plate surfaces and shall either be of the actual thickness to be applied, or of sufficient thickness to give a true average representation of the material to be tested.

10.3

Specimen Conditioning—Details of the specimen selection and conditioning are preferably specified in the material specification. Where such specifications are not given, the specimen preparation shall be conducted in accordance with the requirement that materials shall not be exposed to temperatures that will change the specimens in an irreversible manner. Typically the material specifications call for sample conditioning at 22°C (72°F) and 50 % R.H. for a

TABLE 1 Estimates of Random and Systematic Errors at Room Temperature

Variable	Absolute Variations		Percent Variations	
	Random	Systematic	Random	Systematic
Mean Temperature, T	0.02 K	0.2 K	0.007	0.07
ΔT	0.01 K	0.2 K	0.04	0.40
ΔL	0	0.1 mm	0	0.40
Transducer area	0	50 mm^2	0	0.10
Heat flux				
drift	1 mW	1 mW	0.2	0.2
lateral	1 mW	2 mW	0.2	0.4
calibration	0	2 mW	0	0.2
Temperature				
drift	0.05 K	0.05 K	0.01	0.01
calibration	0	0.01 K	0	0.4
λ	0.2 mW/m-K	0.3 mW/m-K	0.4	0.6

168   period of time until less than a 1 % weight change is observed.

10.4

Measurements on Specimens:

10.4.1

Blanket and Batt Type Materials—When specified, the test thickness of blankets and batt type materials shall be determined before testing in accordance with Test Methods C 167, provided that good contact is maintained between the specimen and the isothermal plates.

10.5

Limitations on Specimen Thickness:

10.5.1

General—The combined thickness of the specimen or specimens, the heat flux transducer and any damping material, which in total equals the distance between the cold and hot plates, must be restricted in order to limit the effect of edge losses on the measurement of heat flux. The edge losses are affected by the edge insulation and the ambient temperature, so the requirements on both the these parameters must be met as well.

10.5.2

Maximum Spacing Between Hot and Cold Plates—The maximum allowable distance between the hot and cold plates during the test, is related to the dimensions of the heat flux transducer, the heat metering area, the width of the non-metering plate, the construction of the heat meter apparatus, and the properties of the specimen. No suitable theoretical analysis is available to predict the maximum allowable thickness of specimens. It is possible to use the results of an analysis for a similarly sized guarded hot plate as a guide (**8, 18**).

11. Procedure of Measurement

11.1

Temperature Difference and Gradient—For any test, make the temperature difference across the specimen not less than 10 K. For specimens that are expected to have a large thermal resistance, a larger temperature difference in the specimen is recommended. The actual temperature difference or gradient is best specified in the material specifications or by agreement of the parties concerned.

11.2

Edge Insulation—Wrap the edges of the specimens with the required thermal insulation to reduce edge heat losses to an acceptable level (see 7.7).

11.3

Settling Time and Measurement Interval—Check for thermal equilibrium by observing and if possible recording, the mean temperature and the emf output of the heat flux transducer, the mean temperature of the specimens, and the temperature drop across the specimen. Make observations at time intervals of at least 10 minutes unless experience has shown that a shorter time interval may be used until five successive observations yield values of thermal conductance which fall with one-half percent of their mean value. For high density specimens ($\rho > 40$ Kg/m^3) or for low conductance specimens (C < 0.05 W/K.m^2) the time between readings may have to be increased to 30 minutes or longer (**19**). If the five readings show a unidirectionally increasing or decreasing trend the data is suspect and additional sets of readings shall be taken.

12. Calculation

12.1

Density and Change in Mass—When required, calculate the density of the dry specimen as tested, ρ, the loss of mass due to conditioning of the material, and the mass regain of the specimen during test.

12.1.1

Density of Batt and Blanket Specimens—It has been found that in characterizing the thermal performance of low density materials it is important to measure the mass of the specimens in contact with the heat flux transducer metering area. For these materials, the area of the specimen directly measured must be cut out and its mass determined after testing. This procedure is desirable for all specimens.

12.2

Thermal Properties for One Specimen—When only one specimen is used, calculate the thermal conductance of the specimens as follows:

$$C = S \cdot E/\Delta T$$

and where applicable, calculate the thermal conductivity, as follows:

$$\lambda = S \cdot E \cdot (L/\Delta T)$$

12.3

Thermal Properties for Two Specimens—When two specimens are used, calculate the total thermal conductance, C, as follows:

$$C = S \cdot E/(\Delta T_1 + \Delta T_2)$$

The λ factor, that is, the average thermal conductivity of the specimen is calculated as follows:

$$\lambda_{ave} = \frac{S \cdot E}{2} \frac{L_1 + L_2}{\Delta T_1 + \Delta T_2}$$

where the subscripts refer to the two specimens.

12.4

Other derived thermal transmission properties may be calculated but only under the provisions given in Practice C 1045.

12.5

Thermal Properties for Two Transducers—All pertinent specifications of 12.2 apply on this configuration, provided S - E will be replaced by *(S′ - E′ + S″ · E″)/2*, where the superscripts ′ and ″ refer to the first and second heat flux transducer, respectively.

13. Precision and Bias

13.1

The precision and bias of this test method cannot be stated quantitatively for all apparatus variations and test conditions. However, it is mandated that for compliance with this test method, any result must be accompanied with its uncertainty. Further details on doing this are given in Section 9.

13.2

It should be noted that the only statistically valid procedure to determine uncertainty is to force all sources of systematic bias to be exhibited as random errors and then uncertainty can be directly estimated from imprecision. However, this is impractical since it involves variation of all measurement variables (Global set) and cost constraints make this impossible. For example, this implies the repetition of an experiment with differently calibrated thermocouples, different sizes and configurations of the apparatus, different operators, different environment, differently calibrated instrumentation, etc. For any one given apparatus a careful error analysis as outlined in Section 9 will, in most cases, show up any major difficulties, which may need correcting in order to improve the measurement accuracy of the heat flow meter apparatus.

13.3

A series of three round robins was conducted between 1976 and 1983, as reported by Hust and Pelanne (**21**), and employed low density fiberglass specimens from 2.54 to 10.2 cm thick with densities ranging from 10 to 33 kg/m^3. A total of twelve laboratories was involved in these studies. The

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imprecision, at the two standard deviation level, was found to vary from 1.92 % to 3.54 % between 2.54 cm and 10.2 cm.

13.4

Inter-laboratory testing, earned out between nine, laboratories under the National Voluntary Laboratory Accreditation Program is currently showing an imprecision of 2.12 % at the two standard deviation level based on testing of similar but not identical specimens.

13.5

A round robin, conducted in 1987, was reported by Adams and Hust with eleven participating laboratories testing a fiberglass blanket and several types of loose-fill insulations (**22**). The blanket insulation had an imprecision of 3.7 % at the two standard deviation level. The loose-fill imprecision was found to be > 10 % for different materials at the two standard deviation level. It has been suggested that the principal cause for the significant differences observed was the various sample preparation techniques used by the various laboratories.

14. Report

14.1

The report of the results of each test shall include the following information with all data to be reported in both SI and inch-pound units unless specified otherwise:

14.1.1

Name and any other pertinent identification of the material including a physical description.

14.1.2

Description of the specimen and its relationship to the sample, including a brief history of the specimen, if known.

14.1.3

Thickness of the specimen as received and as tested. In a two piece specimen configuration this is the thickness of one piece only.

14.1.4

Method and environment used for conditioning, if used.

14.1.5

Density of the conditioned specimen as tested, kg/m^3.

14.1.6

Mass loss of the specimen during conditioning and testing in percentage of conditioned mass, if measured.

14.1.7

Mass regain of the specimen during test; in percentage of conditioned mass, if measured.

14.1.8

Average temperature gradient in the specimen during test as computed from the temperatures of the hot and cold surfaces, K/m.

14.1.9

Mean temperature of the test, K or °C.

14.1.10

Heat flux per unit area through the specimen, W/m^2.

14.1.11

Thermal conductance, $W/m^2 \cdot K$.

14.1.12

Orientation and position of the heat meter apparatus during test (vertical, horizontal, etc.) and whether the meter was against the hot or cold surface of the specimen and whether the edges of the specimen(s) were sealed or open to the ambient.

14.1.13

Duration of the measurement portion of the test, min or h.

14.1.14

For loose-fill materials report the sample preparation followed.

14.1.15

Date of the test, the date of the last heat meter calibration, and the type or types of materials used.

14.1.16

For certification testing only, the specimens used in calibration must be identified as to the type, thermal resistance date of specimen certification, source of certification expiration date of calibration and the certification test number.

14.1.17

Statement of Compliance or, where circumstances or requirements preclude complete compliance with the procedures of the test, agreed exceptions. A suggested wording is: "This test conformed with all requirements of ASTM C 518 - _____ with the exception of (a complete list of exceptions follows)."

14.1.18

For direct reading apparatus, the results of the calibration of electronic circuitry and equipment, or a statement of compliance including date, and a statement of compliance on linearity requirements.

14.1.19

Estimated or calculated uncertainty in reported values.

15. Keywords

15.1

thermal insulation; thermal resistance; thermal conductivity, test method heat flux; instrument verification; test procedures; error analysis; heat flow meter apparatus

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ANNEXES

(Mandatory Information)

TABLE A1.1 Limits of Error for Thermocouples							
Note 1—Limits of error in this table apply to new thermocouple wire, normally in the size range 0.25 to 3 mm in diameter (No. 30 to No. 8 Awg) and used to temperatures not exceeding the recommended limits of Table 2. If used at higher temperatures these limits of error may not apply.							
Note 2—Limits of error apply to new wire as delivered to the user *and do not allow lor calibration drift during use*. The magnitude of such changes depends on such factors as wire size, temperature, time of exposure, and environment.							
Note 2—Note 3—Where limits of error are given in percent, the percentage applies to the temperature being measured when expressed in degrees Celsius. To determine the limit of error in degrees Fahrenheit multiply the limit of error in degrees Celsius by 9/5.							
Thermo-couple Type	Temperature Range		Limits of Error-Reference Junction 0°C (32°F)				
			Standard		Special		
	°C	°F	°C (whichever is greater)	°F	°C (whichever is greater	°F
T	0 to 350	32 to 700	±1 or ±0.75 %	Note 3	±0.5 or 0.4 %	Note 3
J	0 to 750	32 to 1400	±2.2 or ±0.75 %		±1.1 or 0.4%	
E	0 to 900	32 to 1600	±1.7 or ±0.5%		±1 or ±0.4 %	
K	0 to 1250	32 to 2300	±2.2 or ±0.75 %		±1.1 or ±0.4 %	
R or S	0 to 1450	32 to 2700	±1.5 or ±0.25%		±0.6 or ±0.1%	
B	870 to 1700	1600 to 3100	±0.5 %		—	
T[A]	-200 to 0	-328 to 32	±1 or ±1.5 %		*B*	
E[A]	-200 to 0	-328 to 32	±1.7 or ±1 %		*B*	
K[A]	-200 to 0	-328 to 32	±2.2 or ±2 %		*B*	

[A] Thermocouples and thermocouple materials are normally supplied to meet the limits of error specified in the table for temperatures above 0°C. The same materials, however, may not fall within the sub-zero limits of error given in the second section of the table. If materials are required to meet the sub-zero limits, the purchase order must so state. Selection of materials usually will be required.

[B] Little information is available to justify establishing special limits of error for sub-zero temperatures. Limited experience suggests the following limits for Types E and T thermocouples:
Type E -200 to 0°C ±1°C or ±0.5 % (whichever Is greater)
Type T -200 to 0°C ±0.5°C or ±0.8 % (whichever is greater)
These limits are given only as a guide for discussion between purchaser and supplier.
   Due to the characteristics of the materials, sub-zero limits of error for Type J thermocouples and special sub-zero limits for Type K thermocouples are not listed.

A.2 Response of Heat Flux Transducers

A2.1

Configurations—The general features of a heat flow meter apparatus with the specimen or the specimens installed are shown in Fig. A2.1. A heat flow meter apparatus consists of two isothermal plate assemblies, one or more heat flux transducers, equipment to measure and control temperature, thickness and the output of the heat flux transducers and equipment to control the environmental conditions when needed. Configuration a is called single transducer, one face; the heat flux transducer may be placed against either plate. The Configuration b is called two transducer, both faces. The Configuration c is called single transducer, midplane; in this case, the specimens should be halves of the same specimen (20). Each configuration will yield equivalent results if used within the limitations stated in this method. For a particular heat flux transducer, the test configuration that has the fastest response, that is shortest settling time, is best determined by experiment. Four specimens of different materials, such as urethane, ceramic fiber board, a rubber, and a high conductivity, low-thermal capacity material, each of which is conditioned to the mean test-temperature, should be tested in each configuration. A study of these will allow either the selection of the proper configuration for each type of material, or the selection of a reasonable configuration for all types.

A2.1.1

In order to predict settling times for all types of specimens, each of the above specimens must be retested after being conditioned to temperatures both below and above the mean temperature of the test.

A2.2

Time Responses of Heat Flux Transducers:

A2.2.1

High Thermal Resistance Transducer—A transducer with a high thermal resistance is generally used in Configuration a. When the specimens are preconditioned to the mean temperature of the test and when the plates are capable of both heating and cooling the specimen, the time response of a high resistance transducer will be more rapid than a comparable guarded hot plate apparatus. However, if the heat flux transducer has appreciable mass the response will not be rapid.

A2.2.1.1

The temperature drop across a high thermal resistance transducer may be greater than desirable, that is, one percent of the total, when used in Configuration c, and thus its use in this configuration is not recommended.

A2.2.1.2

Configuration b can be used to obtain very rapid response times if both plates are capable of heating and cooling and if the outputs of both transducers are summed, (15). Configuration b is always faster than Configuration a with the same transducers and specimens and can be Comparable to Configuration c if a low thermal resistance transducer is employed.

A2.2.2

Low Thermal Resistance Transducers—The low-thermal resistance, gradient-type heat flux transducer is better suited to the Configuration c. The temperature drop across the low-resistance transducer is small enough so that the two specimens can be considered as halves of a single specimen. When the specimens are first conditioned to the mean temperature of the test and when the specimens are identical, the response is sufficiently rapid to be used for quality control work.

TABLE A1.2a Conversion Factors (Thermochemical)

Note—Conversion factors for thermal resistivity and thermal conductance can be found by using these tables in the reverse direction.

Thermal Conductivity[A]

	$W \cdot m^{-1} \cdot K^{-1}$([B])	$W \cdot cm^{-1} \cdot K^{-1}$	$cal \cdot s^{-1} \cdot cm^{-1} \cdot K^{-1}$	$kg\text{-}cal \cdot h^{-1} \cdot m^{-1} \cdot K^{-1}$	$Btu \cdot h^{-1} \cdot ft^{-1} \cdot °F^{-1}$	$Btu \cdot in. \cdot h^{-1} \cdot ft^{-2} \cdot °F^{-1}$
$1\ W \cdot m^{-1} \cdot K^{-1} =$	1.000	1.000×10^{-2}	2.390×10^{-3}	0.8604	0.5782	6.938
$1\ W \cdot cm^{-1} \cdot K^{-1} =$	100.0	1.000	0.2390	86.04	57.82	693.8
$1\ cal \cdot s^{-1} \cdot cm^{-1} \cdot K^{-1} =$	418.4	4.184	1.000	360.0	241.9	2903
$1\ kg\text{-}cal \cdot h^{-1} \cdot m^{-1} \cdot K^{-1} =$	1.162	1.162×10^{-2}	2.778×10^{-3}	1.000	0.6720	8.064
$1\ Btu \cdot h^{-1} \cdot ft^{-1} \cdot °F^{-1} =$	1.730	1.730×10^{-2}	4.134×10^{-3}	1.488	1.000	12.00
$1\ Btu \cdot in. \cdot h^{-1} \cdot ft^{-2} \cdot °F^{-1} =$	0.1441	1.441×10^{-3}	3.445×10^{-4}	0.1240	8.333×10^{-2}	1.000

Thermal Resistance[A]

	$K \cdot m^2 \cdot W^{-1}$([B])	$K \cdot cm^2 \cdot W^{-1}$	$K \cdot cm^2 \cdot s \cdot cal^{-1}$	$K \cdot m^2 \cdot kg\text{-}cal^{-1}$	$°F \cdot ft^2 \cdot h \cdot Btu^{-1}$	
$1\ K \cdot m^2 \cdot W^{-1} =$	1.000	1.000×10^4	4.184×10^4	1.162	5.674	
$1\ K \cdot cm^2 \cdot W^{-1} =$	1.000×10^{-4}	1.000	4.184	1.162×10^{-4}	5.674×10^{-4}	
$1\ K \cdot cm^2 \cdot s \cdot cal^{-1} =$	2.390×10^{-5}	0.2390	1.000	2.778×10^{-5}	1.356×10^{-4}	
$1\ K \cdot m^2 \cdot h \cdot kg\text{-}cal^{-1} =$	0.8604	8.604×10^3	3.600×10^4	1.000	4.882	
$1\ °F \cdot ft^2 \cdot h \cdot Btu^{-1} =$	0.1762	1.762×10^3	7.373×10^3	0.2048	1.000	

[A] Units are given in terms of (1) the absolute joule per second or watt, (2) the calorie (thermochemical) = 4.184 J, or the British thermal unit (thermochemical) = 1054.35

[B] This is the SI (International System of Units) unit.

TABLE A1.2b Conversion Factors (International Table)

Note—Conversion factors for thermal resistivity and thermal conductance can be found by using these tables in the reverse direction.

Thermal Conductivity[A]

	$W \cdot m^{-1} \cdot K^{-1}$([B])	$W \cdot cm^{-1} \cdot K^{-1}$	$cal \cdot s^{-1} \cdot cm^{-1} \cdot K^{-1}$	$kg\text{-}cal \cdot h^{-1} \cdot m^{-1} \cdot K^{-1}$	$Btu \cdot h^{-1} \cdot ft^{-1} \cdot °F^{-1}$	$Btu \cdot in. \cdot h^{-1} \cdot ft^{-2} \cdot °F^{-1}$
$1\ W \cdot m^{-1} \cdot K^{-1} =$	1.000	1.000×10^{-2}	2.388×10^{-3}	0.8598	0.5778	6.933
$1\ W \cdot cm^{-1} \cdot K^{-1} =$	100.0	1.000	0.2388	85.98	57.78	693.3
$1\ cal \cdot s^{-1} \cdot cm^{-1} \cdot K^{-1} =$	418.7	4.187	1.000	360.0	241.9	2903
$1\ kg\text{-}cal \cdot h^{-1} \cdot m^{-1} \cdot K^{-1} =$	1.163	1.163×10^{-2}	2.778×10^{-3}	1.000	0.6720	8.064

[A] Units are given in terms of (1) the absolute joule per second or watt, (2) the calorie (International Table) = 4.1868 J, or the British thermal unit (International Table) = 1055.06 J. These tables are meant for use in Canada and Great Britain.

[B] This is the SI (International System of Units) unit.

TABLE A1.2b Conversion Factors (International Table)

1 Btu · h^{-1} · ft^{-1} · °F^{-1} =	1.731	1.731 × 10^{-2}	4.134 × 10^{-3}	1.488	1.000	12.00
1Btu · in. · h^{-1} · ft^{-2} · °F^{-1} =	0.1442	1.442 × 10^{-3}	3.445 × 10^{-4}	0.1240	8.333 × 10^{-2}	1.000

Thermal Resistance[A]

	K · m^2 · W^{-1}(^{B})	K · cm^2 · W^{-1}	K · cm^2 · s · cal^{-1}	K · m^2 · kg-cal^{-1}	°F · ft^2 · h · Btu^{-1}	
1 K · m^2 · W^{-1} =	1.000	1.000 × 10^4	4.187 × 10^4	1.163	5.678	
1 K · cm^2 · W^{-1} =	1.000 × 10^{-4}	1.000	4.187	1.163 × 10^{-4}	5.678 × 10^{-4}	
1 K · cm^2 · s · cal^{-1} =	2.388 × 10^{-5}	0.2388	1.000	2.778 × 10^{-5}	1.356 × 10^{-4}	
1 K · m^2 · h · kg-cal^{-1} =	0.8598	8.598 × 10^3	3.600 × 10^4	1.000	4.882	
1 °F · ft^2 · h · Btu^{-1} =	0.1761	1.761 × 10^3	7.373 × 10^3	0.2048	1.000	

[A] Units are given in terms of (1) the absolute joule per second or watt, (2) the calorie (International Table) = 4.1868 J, or the British thermal unit (International Table) = 1055.06 J. These tables are meant for use in Canada and Great Britain.

[B] This is the SI (International System of Units) unit.

A2.2.2.1

Where half thicknesses of the normal specimen can be used, it can be more rapid than Configuration a, especially when each of the specimens is first conditioned to the mean temperature at which it will operate in the test. The use of the low-resistance transducer in Configuration a is ideal if Plate 1 can provide both heating and cooling, but unless the temperature of Plate 1 is extremely well controlled, a damping layer will be required between the transducer and the plate. The advantage of the fast response time is then partially lost, but the response time is likely rapid enough in any event. The transducer can be mounted permanently in this configuration.

A2.3

Sensitivity of Configuratipnp to Edge Losses:

A2.3.1

The Configuration a is, similar to the guarded hot plate apparatus, regarding edge heat losses through the specimen. The edge heat losses in the transducer may be much more significant than those in the guarded hot plate apparatus because they may produce errors due to the temperature non-uniformity on the side in contact with the specimen.

A2.3.2

The Configuration b is the most insensitive to edge conditions if the average of the readings of the two meters is assumed to be the measured heat flux per unit area through the specimen. If the plates were perfectly uniform in temperature, if the two transducers were exactly equal in the layout of the thermopile junctions, and if the specimen had thermal conductivity independent of temperature, this configuration would be insensitive to edge conditions. However, even under these ideal conditions the use of Configuration b does not eliminate edge losses but only reduces the apparatus sensitivity to variations in the boundary temperatures. Also, the settling time is short if the average of the two readings is used.

A2.3.3

      Configuration c is very sensitive to edge heat losses on the heat flux transducer as the power that flows through the edges is supplied, not by a heavy nearly isothermal metal plate, but by the specimens, so that their surface temperatures may not be uniform. If the transducer is sensitive to temperature differences along its main surfaces, edge heat

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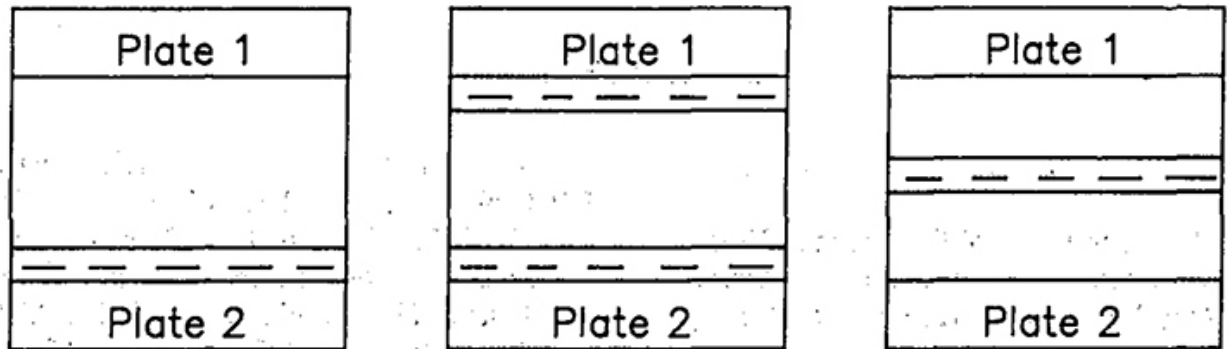
FIG. A2.1 Typical Heat Flow Meter Apparatus Configurations
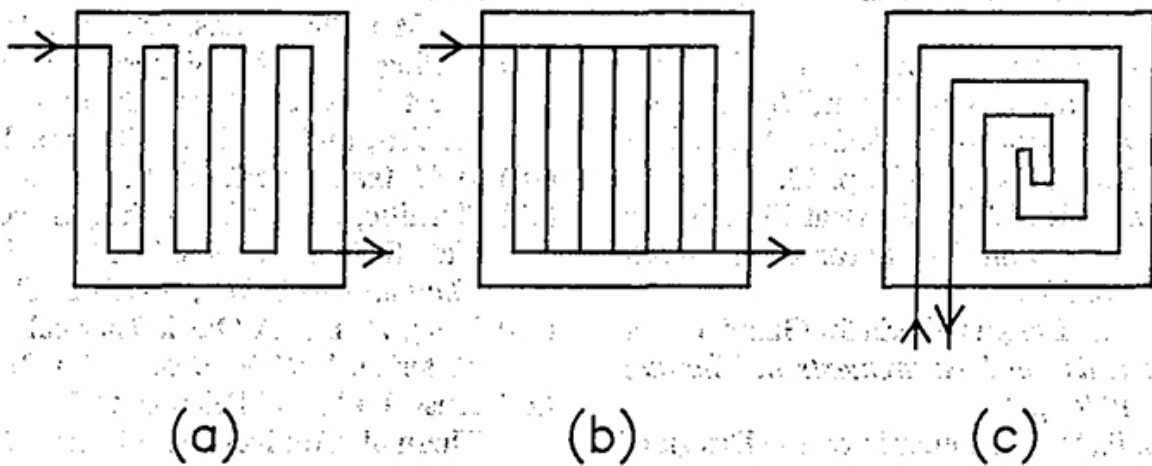
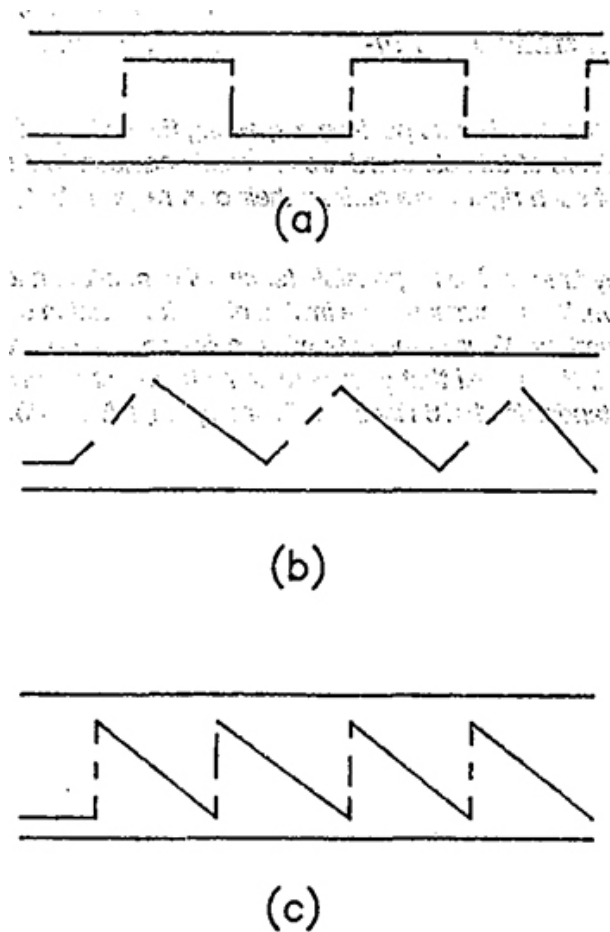FIG. A2.2 Some Layouts of the Liquid Paths in Heating or Cooling Plates

FIG. A2.3 Schematic Designs of Transducers

losses may now create serious errors. Edge heat losses within the specimens are similar to those in the guarded hot plate when the surrounding temperature is that of the hot or cold plate.

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An American National Standard

Designation: D 86 – 07

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 (∈) 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 B3P 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
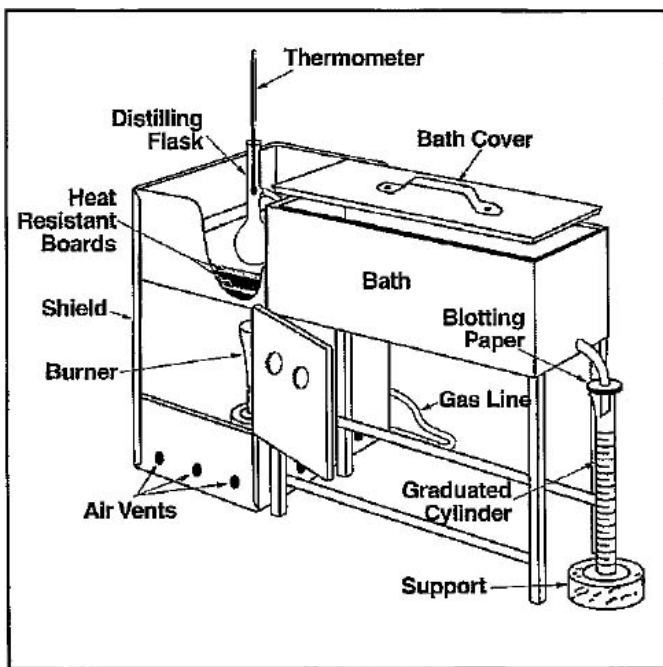
FIG. 1 Apparatus Assembly Using Gas Burner

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.

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 E1 or IP Standard Methods for Analysis and Testing of Petroleum and Related Products 1996—Appendix A, or both, for thermometers ASTM

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FIG. 2 Apparatus Assembly Using Electric Heater
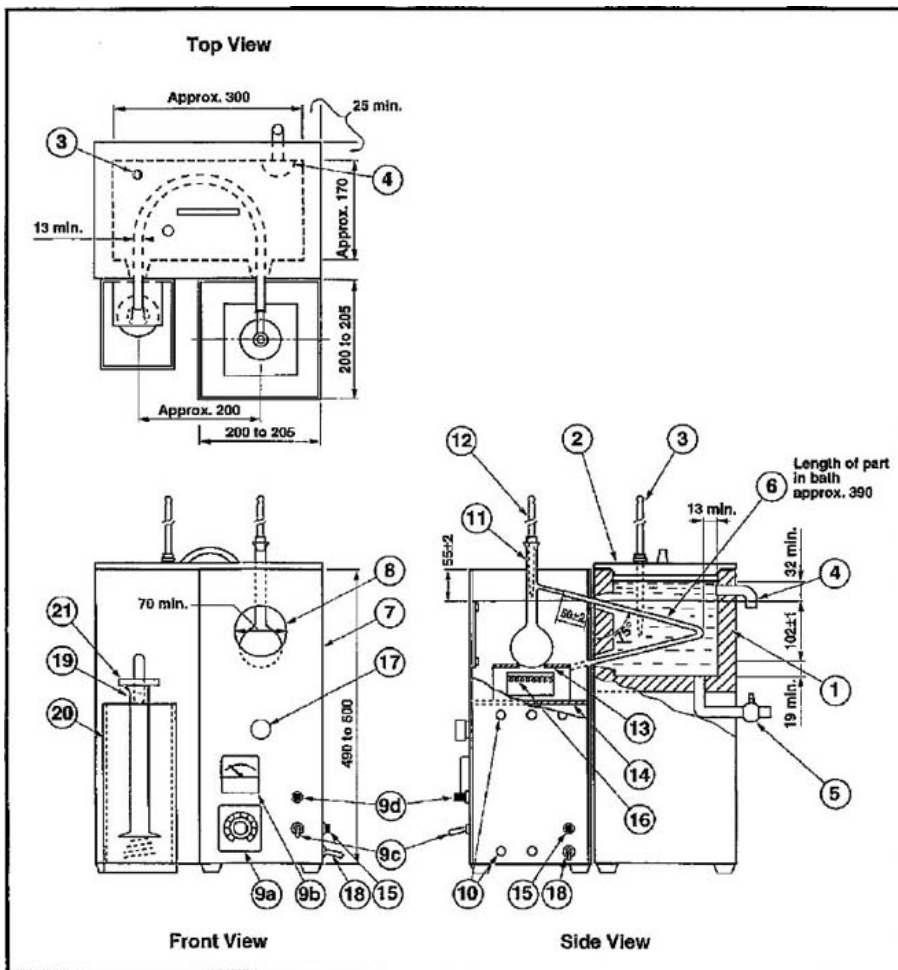
1–Condenser bath
2–Bath cover
3–Bath temperature sensor
4–Bath overflow
5–Bath drain
6–Condenser tube
7–Shield
8–Viewing window
9a–Voltage regulator
9b–Voltmeter or ammeter
9c–Power switch
9d–Power light indicator
10–Vent

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. 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
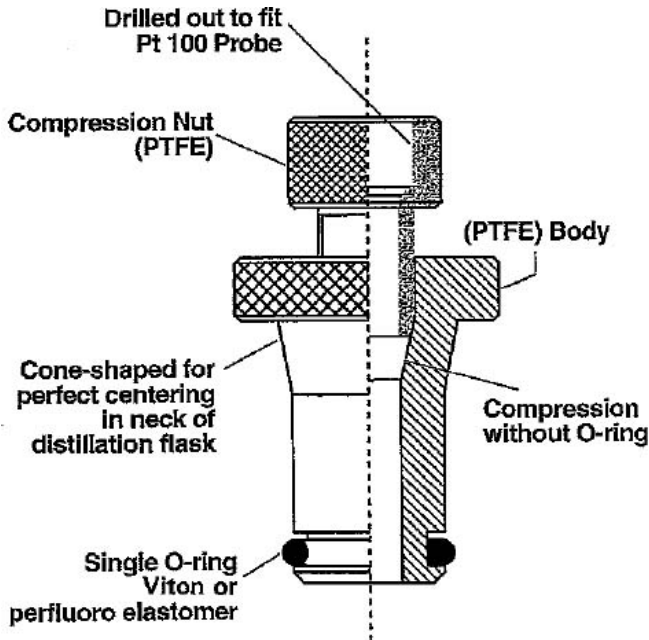
FIG. 4 Example of Centering Device Designs for Straight-Bore Neck Flasks
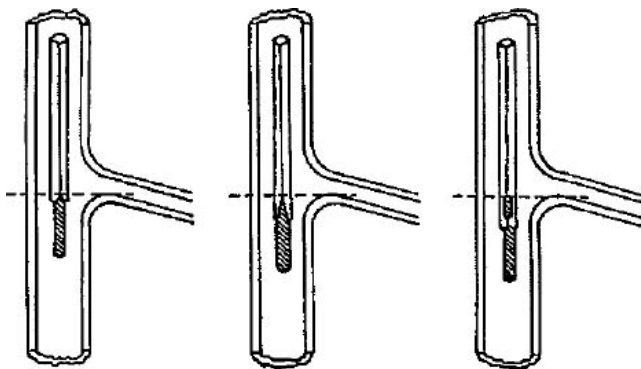
FIG. 5 Position of Thermometer in Distillation Flask

a low IBP may have one or more readings obscured by the centering device. See also 10.14.3.1.

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				

	Group 1	Group 2	Group 3	Group 4
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	°C	<10	<10	Ambient or	Ambient or
conditioning prior to analysis				9 to 21°C above pour point[B]	
	°F	<50	<50	Ambient or	Ambient or
				48 to 70°F above pour point[B]	
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.

[7]

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	109.7
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:D02–1580.					

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, rightly 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 D97, 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).
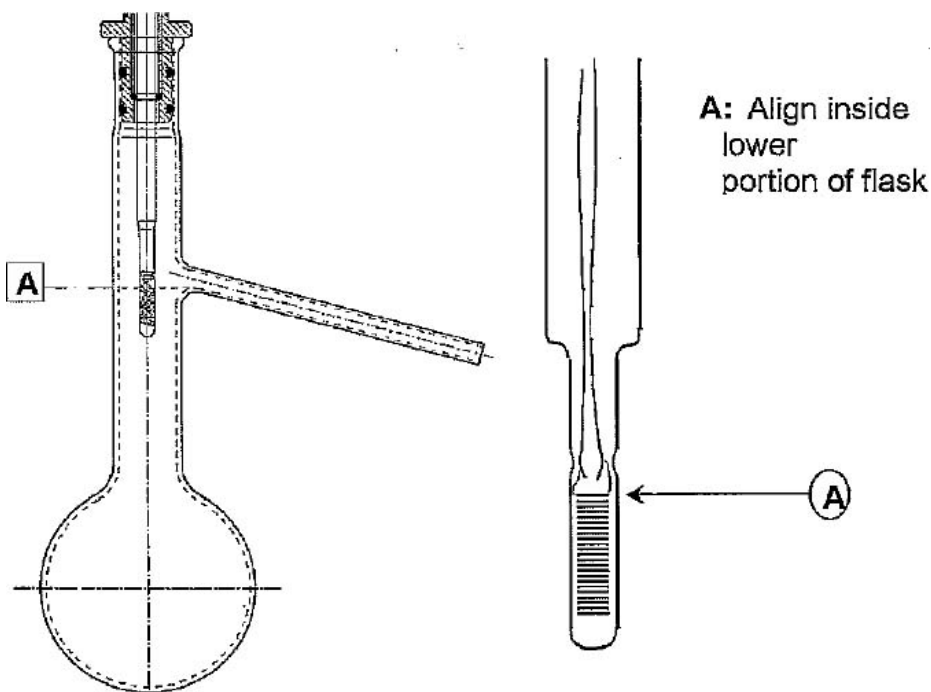

FIG. 6 Example of Recommended Placement of Pt-100 Probe Relative to Distillation Flask Sidearm for Automated D 86 Distillation Instrument

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

		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 to 10 % recovered, min		60–100	60–100		
Uniform average rate of condensation from 5 % recovered to 5 mL in flask, mL/min		4–5	4–5	4–5	4–5
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 oft 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).

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

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

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:

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

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

For Fahrenheit temperatures:

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

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

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

[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.

Temperature Range		Correction[A] per 1.3 kPa (10 mm Hg) Difference in Pressure	
°C	°F	°C	°F
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.			

apparatus being used, use the corrected temperature readings in all further calculations and reporting.

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.

11

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:

$$P_e = P_r + L \quad (9)$$

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:

$$T = T_L + (T_H - T_L)\,(R - R_L)/(R_H - R_L) \qquad (10)$$

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 XI 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.

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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	$1.9+0.B6S_C$	$3.4+0.86S_F$	$3.1+1.74S_C$	$5.6+1-74S_F$	$2.1+0.67S_C$	$3.8+0.67S_F$	$4.4+2.0S_C$	$7.9+2.0S_F$

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

Evaporated Point, %	Manual Repeatability[A]		Manual Reproducibility[A]		Automated Repeatability[A]		Automated Reproducibility[A]	
	°C	°F	°C	°F	°C	°F	°C	°F
10	$1.2+0.B6S_C$	$2.2+0.B6S_F$	$2.0+1.74S_C$	$3.6+1.74S_F$	$1.7+0.67S_C$	$3.0+0.67S_F$	$3.3+2.0S_C$	$6.0+2.0S_F$
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_G 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:

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

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:

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

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:

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

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:*

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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	$0.7+0.36S_C$	$1.3+0.36S_F$	$3.1+0.42S_C$	$5.7+C.42S_F$
% volume at temperature reading	$0.7+0.92/S_C$	$0.7+1,66/S_F$	$1.5+1.78/S_C$	$1.53+3.20/S_F$

[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 die 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 bath 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.

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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%	$1.2 + 1.42S_C$	$2.2 + 1.42S_F$	$3.0 + 2.64S_C$	$5.4 + Z64S_F$
90–95%	$1.1 + 1.08S_C$	$2.0 + 1.08S_F$	$2.0 + 2.53Sc$	$3.6 + 2,53S_F$
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	
			Reproducibility	Repeatability
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

15   contact with the wall of the receiving cylinder at a point 25 to 32 mm below the top of die 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 7*—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.
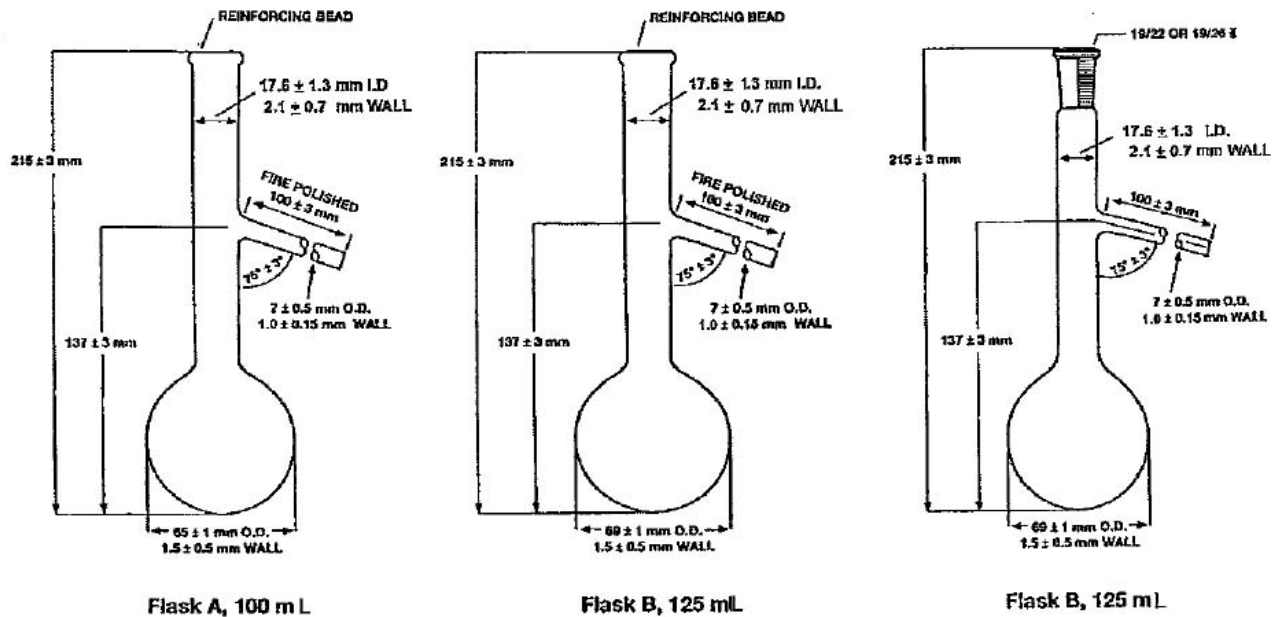
16

FIG. A2.1 Flask A, 100 mL, Flask B, 125 mL, and Flask B with Ground Glass Joint, 125 mL
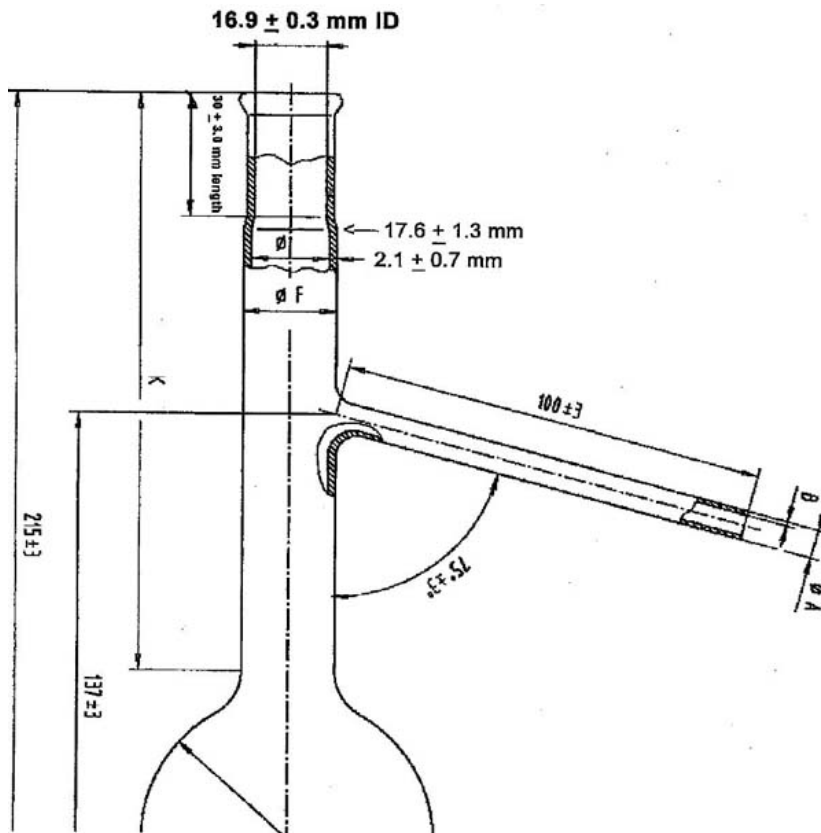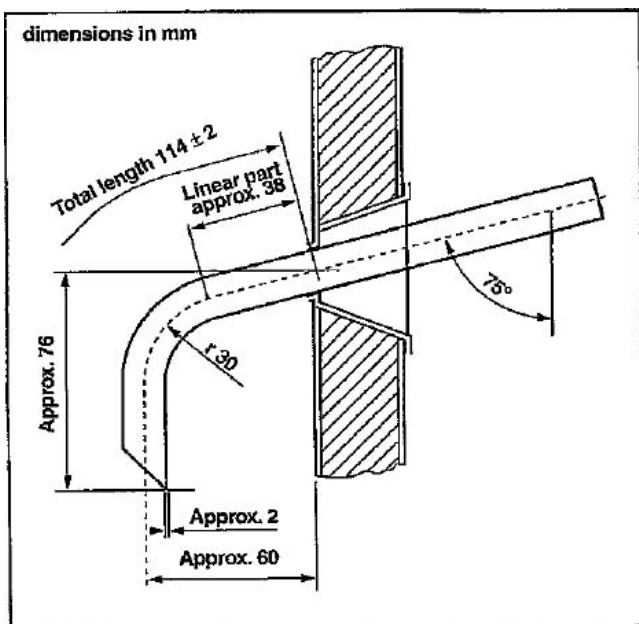
17

FIG. A2.2 Detail of Upper Neck Section

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FIG. A2.3 Lower End of Condenser Tube
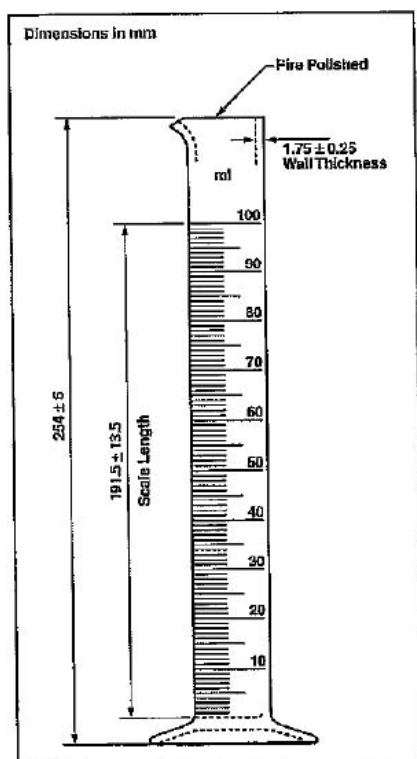
NOTE—1 to 100 mL in 1 mL graduations; tolerance ± 1.0 mL.

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:

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

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

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:

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

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

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.

$$\text{corrected loss} = (0.5 + (4.7 - 0.5)/ \qquad \text{(X1.3)}$$
$$\{1 + (101.3 - 98.6)/8.0\} = 3.6$$

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

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

X1.2 *Temperanire 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:

$$T_{10E}\,(°C) = 33.7 + [(40.3 - 33.7) \qquad \text{(X1.5)}$$
$$(5.3 - 5)/(10 - 5)] = 34.1°C$$
$$T_{10E}(°F) = 92.7 + [(104.5 - 92.7) \qquad \text{(X1.6)}$$
$$(5.3 - 5)/(10 - 5)] = 93.1°F$$

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

$$T_{50E}(°C) = 93.9 + [(108.9 - 93.9) \qquad \text{(X1.7)}$$
$$(45.3 - 40)/(50 - 40)] = 101.9°C$$
$$T_{50E}\,(°F) = 201 + [(228 - 201) \qquad \text{(X1.8)}$$
$$(45.3 - 40)/50 - 40)] = 215.3°F$$

X1.2.3 Temperature reading at 90 % evaporated (85.3 % recovered) (see 11.6,1) are as follows:

$$T_{90E}\,(°C) = 181.6 + [(201.6 - 181.6) \qquad \text{(X1.9)}$$
$$(85.3 - 85)/(90 - 85)\,] = 182.8°C$$

$$T_{90E}\,(°F) = 358.9 + [(394.8 - 358.9) \qquad \text{(XI.10)}$$
$$(85.3 - 85)/(90 - 85)] = 361.0°F$$

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:

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

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				**procedure arithmetical/ graphical**		
	observed 98.6 kPa		**corrected 101.3 kPa**				
%	**740 mm Hg**		**760 mm Hg**		**%**	**T******	
recovered	**°C**	**°F**	**°C**	**°F**	**evaporated**	**°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	46.0	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	2O0.4	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:

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

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

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

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

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

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 (mL) 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.

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Barometric Pressure, kPa																								
from	76.1	80.9	84.5	87.9	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	97.8	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	0.06	0.04	0.02	−0.00	−0.02	−0.06	−0.09	−0.13	−0.17
1	0.63	0.65	0.67	0.69	0.71	0.73	0.75	0.78	0.80	0.82	0.86	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	0.89	0.95	1.01	1.08	1.14	1.20	1.26	1.33	1.40	1.46	1.52	1.67	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	1.16	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	2.89	2.79	2.90	3.00	3.13	3.29	3.48	3.63	3.84
4	1.41	1.66	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	3.08	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	4.38	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	2.49	2.76	3.07	3.38	3.70	4.02	4.31	4.63	4.98	5.28	5.58	5.85	6.12	6.41	6.74	7.06	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	4.96	5.33	5.73	6.17	6.56	6.94	7.28	7.52	7.99	8.41	8.81	9.19	9.60	10.00	10.48	11.08	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	10.56	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	11.58	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	9.53	10.35	11.03	11.68	12.27	12.66	13.51	14.23	14.93	15.59	16.31	17.00	17.84	18.88	19.97	21.19	22.58
18	5.07	5.78	6.50	7.22	7.96	8.71	9.40	10.14	10.94	11.65	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	22.39	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	19.19	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	0.04	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	0.13
0.2	0.05	0.06	0.07	0.08	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	0.29	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	0.26	0.27	0.29	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	0.36	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, mmHg.

from	571	607	634	655	672	686	698	706	716	723	729	734	738	742	746	750	753	756	759	762	765	768	771	774
through	806	633	654	671	685	697	706	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	0.63	0.65	0.67	0.69	0.71	0.73	0.75	0.77	0.80	0.82	0.84	0.86	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	0.89	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	1.48	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	3.28	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	7.38	7.84
7	2.19	2.46	2.72	2.99	3.26	3.54	3.79	4.07	4.36	4.63	4.86	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	4.98	5.27	5.56	5.83	6.09	6.38	6.71	7.02	7.32	7.64	8.00	8.40	8.84	9.33	9.88	10.50
9	2.72	3.06	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	9.46	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	6.31	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	8.87	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	6.83	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	9.08	9.61	10.09	10.57	11.09	11.68	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	11.61	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	6.62	7.51	8.22	8.86	9.56	10.31	10.98	11.63	12.22	12.80	13.45	14.16	14.85	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	13.66	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	6.83	7.59	8.36	9.15	9.88	10.66	11.50	12.25	12.98	13.84	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	5.88	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	0.06	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	0.13
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	0.36	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	0.58	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	0.88	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:

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

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:

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

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

"Percent Evaporated" Report Form   Laboratory:

Date:

Time

Operator:

Ambient temperature (°C)	
Atmospheric pressure (kPa)	
Condenser temperature (°C)	
Temperature of the bath around receiving cylinder (°C)	

Percent Recovered	Corrected Temperature Reading (°C)	Time or mL / min	Percent Evaporated	Temperature Readings at prescribed percent evaporated (°C)
IBP			IBP	
5			5	
10			10	
15			15	
20			20	
25			25	
30			30	
35			35	
40			40	
45			45	
50			50	
55			55	
60			60	
65			65	
70			70	
75			75	
80			80	
85			85	
90			90	
5 ml residue				
95			95	
FBP			FBP	

Percent Recovery	
Percent Residue	
Percent Total Recovery	
Percent Loss	Corrected Loss
Corrected Percent Recovery	Corrected Total Recovery

Comments:

Annotations (right side):
- Ambient temperature at the start of the test
- Ambient barometric pressure at the start of the test
- Volume of condensate observed in the receiving cylinder at any point in the distillation, expressed as a percentage of the charge volume, in connection with simultaneous temperature reading
- Temperature measuring device readings which are corrected to 101,3 kPa barometric pressure
- Sum of the percent recovered and the percent loss
- Temperature measuring device readings at specified percentages evaporated calculated with arithmetical or graphical procedures
- Group 0: 2 to 5 minutes / Group 1, 2 & 3: 5 to 10 minutes / Group 4: 5 to 15 minutes
- Group 1 & 2: 60 to 100 seconds
- Group0: time from first application of heat ro 10% recovered = 3 to 4 minutes / Group 0, 1, 2, 3 & 4: 4 to 5 ml / min uniform average rate from 5% recovered to 5 ml in flask
- Volume of condensate observed in the receiving cylinder when the 5ml conditions are reached
- Volume of condensate observed in the receiving cylinder when the final boiling point is observed
- Maximum percent recovered
- Volume of residue in the flask expressed as a percentage of the charge volume
- Combined Percent Recovery and Percent Residue in the flask
- Time from 5 ml in flask to FBP = < 5 minutes
- 100 minus the Total Recovery
- Percent Recovery corrected for barometric pressure
- Percent Loss corrected for barometric pressure
- Combined Percent Recovery and Percent Residue in the flask corrected for barometric pressure

FIG. X5.2 Percent Evaporated Report Form

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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.

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Designation: D 129 – 95

Designation: 61/99

An American National Standard British Standard 4454

Standard Test Method for Sulfur in Petroleum Products (General Bomb Method)[1]

This standard is issued, under the fixed designation D129; 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 test method has been adopted for use by government agencies to replace Method 5202 of Federal Test Method No. 791b

1. Scope

1.1

This test method covers the determination of sulfur in petroleum products, including lubricating oils containing additives, additive concentrates, and lubricating greases that cannot be burned completely in a wick lamp. The test method is applicable to any petroleum product

sufficiently low in volatility that it can be weighed accurately in an open sample boat and containing at least 0.1 % sulfur.

Note 1—This test method is not applicable to samples containing elements that give residues, other than barium sulfate, which are insoluble in dilute hydrochloric acid and would interfere in the precipitation step. These interfering elements include iron, aluminum, calcium, silicon, and lead which are sometimes present in greases, lube oil additives, or additive oils. Other acid insoluble materials that interfere are silica, molybdenum disulfide, asbestos, mica, etc. The test method is not applicable to used oils containing wear metals, and lead or silicates from contamination. Samples that are excluded can be analyzed by Test Method D 1552.

1.2

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. See 3.2 for specific precautionary directions incorporated in the test method.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193	Specification for Reagent Water[2]
D 1552	Test Method for Sulfur in Petroleum Products (High-Temperature Method)[3]
E 144	Practice for Safe Use of Oxygen Combustion Bombs[4]

3. Summary of Test Method

3.1

The sample is oxidized by combustion in a bomb containing oxygen under pressure. The sulfur, as sulfate in the bomb washings, is determined gravimetrically as barium sulfate.

3.2

Warning—*Strict adherence to all of the provisions prescribed hereafter ensures against explosive rupture of the bomb, or a blow-out, provided the bomb is of proper design and construction and in good mechanical condition. It is desirable, however, that the bomb be enclosed in a shield of steel plate at least 13 mm thick, or equivalent protection be provided against unforseeable contingencies.*

4. Apparatus and Materials

4.1

Bomb,[5,6] having a capacity of not less than 300 mL, so constructed that it will not leak during the test and that quantitative recovery of the liquids from the bomb may be achieved readily. The inner surface of the bomb may be made of stainless steel or any other material that will not be affected by the combustion process or products. Materials used in the bomb assembly, such as the head gasket and lead-wire insulation, shall be resistant to heat and chemical action, and shall not undergo any reaction that will affect the sulfur content of the liquid in the bomb.

4.2

Sample Cup, platinum, 24 mm in outside diameter at the bottom, 27 mm in outside diameter at the top, 12 mm in height outside, and weighing 10 to 11 g.

4.3

Firing Wire, platinum, No. 26 B & S gage, 0.41 mm (16 thou), 27 SWG, or equivalent.

Note 2—**Caution**: The switch in the ignition circuit shall be of a type which remains open, except when held in closed position by the operator.

4.4

Ignition Circuit, capable of supplying sufficient current to ignite the cotton wicking or nylon thread without melting the wire. The current shall be drawn from a step-down transformer or from a suitable battery.

4.5

Cotton Wicking or Nylon Sewing Thread, white.

[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

Current edition approved Aug. 15, 1995. Published October 1995. Originally published as D 129 – 22. Last previous edition D 129 – 91.

This test method was adopted as a joint ASTM-EP standard in 1964.

In the IP, this test method is under the jurisdiction of the Standardization Committee.

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

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

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

[5] Criteria for judging the acceptability of new and used oxygen combustion bombs are described in Practice E 144.

[6] A bomb conforming to the test specifications in IP Standard IP 12 is suitable.

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5. Reagents and Materials

5.1

Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[7] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2

Purity of Water—Unless otherwise indicated, references to water shall mean water as defined by Type II or III of Specification D 1193.

5.3

Barium Chloride Solution (85 g/litre)—Dissolve 100 g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) in distilled water and dilute to 1 liter.

5.4

Bromine Water (saturated).

5.5

Hydrochloric Acid (sp gr 1.19)—Concentrated hydrochloric acid (HC1).

5.6

Oxygen, free of combustible material and sulfur compounds, available at a pressure of 41 kgf/cm^2 (40 atm).

5.7

Sodium Carbonate Solution (50 g/litre)—Dissolve 135 g of sodium carbonate decahydrate ($Na_2CO_3 \cdot 10H_2O$) or its equivalent weight in distilled water and dilute to 1 litre.

5.8

White Oil, USP, or Liquid Paraffin, BP, *or equivalent.*

6. Procedure

6.1

Preparation of Bomb and Sample—Cut a piece of firing wire 100 mm in length. Coil the middle section (about 20 mm) and attach the free ends to the terminals. Arrange the coil so that it will be above and to one side of the sample cup. Insert between two loops of the coil a wisp of cotton or nylon thread of such length that one end will extend into the sample cup. Place about 5 mL of Na_2CO_3 solution in the bomb (Note 3) and rotate the bomb in such a manner that the interior surface is moistened by the solution. Introduce into the sample cup the quantities of sample and white oil (Note 5 and Note 6) specified in the following table, weighing the sample to the nearest 0.2 mg (when white oil is used, stir the mixture with a short length of quartz rod and allow the rod to remain in the sample cup during the combustion).

Note 3—After repeated use of the bomb for sulfur determinations, a film may be noticed on the inner surface. This dullness can be removed by periodic polishing of the bomb. A satisfactory method for doing this is to rotate the bomb in a lathe at about 300 rpm and polish the inside surface with emery polishing papers Grit No. 2/0, or equivalent paper,[8] coated with a light machine oil to prevent cutting, and then with a paste of grit-free chromic oxide[9] and water. This procedure will remove all but very deep pits and put a high polish on the surface. Before the bomb is used it shall be washed with soap and water to remove oil or paste left from the polishing operation.

Note 4—**Caution:** Do not use more than 1.0 g total of sample and white oil or other low sulfur combustible material or more than 0.8 g if the IP 12 bomb is used.

Sulfur Content percent	Weight of Sample, g	Weight of White Oil, g
5 or under	0.6 to 0.8	0.0
Over 5	0.3 to 0.4	0.3 to 0.4

Note 5—Use of sample weights containing over 20 mg of chlorine may cause corrosion of the bomb. To avoid this, it is recommended that for samples containing over 2 % chlorine, the sample weight be based on the chlorine content as given in the following table:

Chlorine Content percent	Weight of Sample, g	Weight of White Oil, g
2 to 5	0.4	0.4
Over 5 to 10	0.2	0.6
Over 10 to 20	0.1	0.7
Over 20 to 50	0.05	0.7

Note 6—If the sample is not readily miscible with white oil, some other low sulfur combustible diluent may be substituted. However, the combined weight of

sample and nonvolatile diluent shall not exceed 1.0 g or more than 0.8 g if the IP
12 bomb is used.

6.2

Addition of Oxygen—Place the sample cup in position and arrange the cotton wisp or
nylon thread so that the end dips into the sample. Assemble the bomb and tighten the cover
securely. (**Caution**—See Note 7.) Admit oxygen slowly (to avoid blowing the oil from the cup)
until a pressure is reached as indicated in the following table:

Capacity of Bomb, ml	Minimum Gage Pressure,[A] kgf/cm^2(atm)	Maximum Gage Pressure,[A] kgf/cm^2(atm)
300 to 350	39 (38)	41 (40)
350 to 400	36 (35)	38 (37)
400 to 450	31 (30)	33 (32)
450 to 500	28(27)	30(29)
[A] The minimum pressures are specified to provide sufficient oxygen for complete combustion and the *maximum pressures represent a safety requirement.*		

Note 7—**Caution:** Do not add oxygen or ignite the sample if the bomb has
been jarred, dropped, or tilted.

6.3

Combustion—Immerse the bomb in a cold distilled-water bath. Connect the terminals to
the open electrical circuit. Close the circuit to ignite the sample. (**Caution**—See Note 8.)
Remove the bomb from the bath after immersion for at least 10 min. Release the pressure at a
slow, uniform rate such that the operation requires not less than 1 min. Open the bomb and
examine the contents. If traces of unburned oil or sooty deposits are found, discard the
determination and thoroughly clean the bomb before again putting it in use (Note 3).

Note 8—**Caution:** Do not go near the bomb until at least 20 s after firing.

6.4

Collection of Sulfur Solution—Rinse the interior of the bomb, the oil cup, and the inner
surface of the bomb cover with a fine jet of water, and collect the washings in a 600-mL beaker
having a mark to indicate 75 mL. Remove any precipitate in the bomb by means of a rubber
policeman. Wash the base of the terminals until the washings are neutral to the indicator
methyl red. Add 10 mL of saturated bromine water to the washings in the beaker. (The volume
of the washings is normally in excess of 300 mL.) Place the sample cup in a 50-mL beaker. Add
5 mL of saturated bromine water, 2 mL of HC1, and enough water just to cover the cup. Heat
the contents

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

[8] Emery Polishing Paper Grit No. [2]/ocan be purchased from Norton Co., Troy, N. Y.

[9] Chromic oxide may be purchased from J. T. Baker & Co., Phillipsburg, N. J.

109   of the beaker to just below its boiling point for 3 or 4 min and add to the beaker containing the bomb Washings. Wash the sample cup and the 50-mL beaker thoroughly with water. Remove any precipitate in the cup by means of a rubber policeman. Add the washings from the cup and the 50-mL beaker, and the precipitate, if any, to the bomb washings in the 600-mL beaker. Do not filter any of the washings, since filtering would remove any sulfur present as insoluble material.

6.5

Determination of Sulfur—Evaporate the combined washings to 200 mL on a hot plate or other source of heat. Adjust the heat to maintain slow boiling of the solution and add 10 mL of the BaCl $_2$ solution, either in a fine stream or dropwise. Stir the solution during the addition and for 2 min thereafter. Cover the beaker with a fluted watch glass and continue boiling slowly until the solution has evaporated to a volume approximately 75 mL as indicated by a mark on the beaker. Remove the beaker from the, hot plate (or other source of heat) and allow it to cool for 1 hr before filtering. Filter the supernatant liquid through an ashless, quantitative filter paper (Note 9). Wash the precipitate with water, first by decantation and then on the filter, until free from chloride. Transfer the paper and precipitate to a weighed crucible and dry (Note 10) at a low heat until the moisture has evaporated. Char the paper completely without igniting if, and finally ignite at a bright red heat until the residue is white in color. After ignition is complete, allow the crucible to cool at room temperature, and weigh.

Note 9—A weighed porcelain filter crucible (Selas type) of 5 to 9-μm porosity may be used in place of the filter paper. In this case the precipitate is washed free of chloride and then dried to constant weight at 500 ± 25°C.

Note 10—A satisfactory means of drying, charring, and igniting the paper and precipitate is to place the crucible containing the wet filter paper in a cold electric muffle furnace and to turn on the current. Drying, charring, and ignition usually will occur at the desired rate.

6.6

Blank—Make a blank determination whenever new reagents, white oil, or other low-sulfur combustible material are used. When running a blank on white oil, use 0.3 to 0.4 g and follow the normal procedure.

7. Calculation

7.1

Calculate the sulfur content of the sample as follows:

Sulfur, weight percent = $(P - B)13.73/W$ (1)

where:

P	=	grams of $BaSO_4$ obtained from sample,
B	=	grams of $BaSO_4$ obtained from blank, and
W	=	grams of sample used.

8. Report

8.1

Report the results Of the test to the nearest 0.01 %.

9. Precision and Bias[10]

9.1

The precision of this test is not known to have been obtained in accordance with currently accepted guidelines (for example in Committee D-2 Research Report, "Manual on Determining Precision Data for ASTM Methods on Petroleum Products and Lubricants")[11].

9.1.1

Repeatability—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material; would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

9.1.2

Reproducibility—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

Sulfur, weight percent	Repeatability	Reproducibility
0.1 to 0.5	0.04	0.05
0.5 to 1.0	0.06	0.09
1.0 to 1.5	0,08	0.15
1.5, to 2.0	0,12	0.25

| 2.0 to 5.0 | 0.18 | 0.27 |

> Note 11—The precision shown in the above table does not apply to samples containing over 2 % chlorine because an added restriction on the amount of sample which can be ignited is imposed.

> Note 12—This test method has been cooperatively tested only in the range of 0.1 to 5.0% sulfur.

> Note 13—The following information on the precision of this method has been developed by the Institute of Petroleum (London):

a. Results, of duplicate tests should not differ by more than the following amounts:

Repeatability	Reproducibility,
$0.016\,x + 0.06$	$0.037\,x + 0.13$

> where x is the mean of duplicate test results.

b. These precision values were Obtained in 1960 by statistical examination of interlaboratory test results.[12] No limits have been established for additive concentrates.

9.2

> *Bias*—Results obtained in one laboratory by Test Method D 129 on NIST Standard Reference Material Nos. 1620A, 1621C, and 1662B were found to be 0.05 mass % higher than the accepted reference values.

10. Keywords

10.1

> bomb; sulfur

> [10] Supporting data is available from ASTM Headquarters. Request RR:D02-1278.

> [11] *Annual Book of ASTM Standards,* Vol 05.03.

> [12] IP Standards for Petroleum and Its Products, Part I, Appendix E.

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