ASTM Certified Testing Results

Prepared and Tested by:

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Harris Testing Labs Inc.

Prepared for:

MEGATROL INC
FLUID TRANSFER / LUBRICATION / COMBUSTION

Product: **FE^{26}** Premier Metal Conditioner

Date: February 11, 2008

Index

Testing Results Metal Conditioner

Harris Testing Laboratories, Inc.

CERTIFICATE OF ANALYSIS

CLIENT:
Megatrol Inc.
9469 S 500 W
Sandy, Utah 84070

PRODUCT: **FE^{26}** Premier
MARKS: NONE
DATE RECEIVED: 02/11/2008
LAB NO: HH0410-1202-P
SUBMITTED BY: Jon Rose

			SPECIFICATIONS	
METHOD	**TEST**	**RESULT**	**MIN.**	**MAX.**
D-2893	**Oxidation @ 95 ºC & 312 hours**	**XXX**	**XXX**	**XXX**
D-445	**Viscosity, cSt @ 100 ºC**	**XXX**	**XXX**	**XXX**
	Sample as received	**8.07**	**XXX**	**XXX**
	Oxidized sample	**11.51**	**XXX**	**XXX**
	Viscosity cSt @ 100 ºC Increase %	**42.1**	**XXX**	**XXX**
D-91	**Precipitation Number**	**XXX**	**XXX**	**XXX**
	Sample as received	**0.01**	**XXX**	**XXX**
	Oxidized sample	**0.50**	**XXX**	**XXX**

Comments:

Amos Mwangi

Date issued:
02/11/2008

CHEMIST

Standard Test Method for Precipitation Number of Lubricating Oils[1,2]

This standard is issued under the fixed designation D 91; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of the precipitation number of steam cylinder stocks and black oils, and can be used for other lubricating oils.

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

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

2. Referenced Documents

2.1 *ASTM Standards:*

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[3]

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *ASTM precipitation number, of lubricating oils, n*—the number of millilitres of precipitate formed when 10 mL of lubricating oil are mixed with 90 mL of ASTM precipitation naphtha, and centrifuged under the conditions of the test.

4. Significance and Use

4.1 Fully refined petroleum oils normally contain no naphtha insoluble material. Semirefined or black oils frequently contain some naphtha insoluble material (sometimes referred to as *asphaltenes*). This test measures the amount of naphtha insoluble material in the oil. This quantity is reported as the precipitation number.

5. Apparatus

5.1 *Centrifuge Tube*, cone-shaped, conforming to the dimensions given in Fig. 1, and made of thoroughly annealed glass. The graduations, numbered as shown in Fig. 1, shall be clear and distinct, and the mouth shall be constructed in a shape suitable for closure with a cork. Scale-error tolerances and smallest graduations between various calibration marks are given in Table 1 and apply to calibrations made with air-free water at 20°C.

5.2 *Centrifuge*, meeting all the safety requirements for normal use and capable of whirling two or more filled centrifuge tubes at a speed which can be controlled to give a relative centrifugal force (rcf) between 600 and 700 at the tip of the tubes. The revolving head, trunnion rings, and trunnion cups, including the rubber cushion, shall be soundly constructed to withstand the maximum centrifugal force capable of being delivered by the power source. The trunnion cups and cushions shall firmly support the tubes when the centrifuge is in motion. The centrifuge shall be enclosed by a metal shield or case strong enough to eliminate danger if any breakage occurs. Calculate the speed of the rotating head by means of the following equation:

$$\text{rpm} = 1337\sqrt{\text{rcf}/d} \tag{1}$$

where:

rcf = relative centrifugal force, and

d = diameter of swing, in mm, measured between tips of opposite tubes when in rotating position.

Table 2 shows the relationship between diameter swing, rcf, and revolutions per minute.

6. Reagent

6.1 *Hexanes, Reagent Grade*. (**Warning—**Extremely flammable, harmful if inhaled.)

NOTE 1—Precipitation naphtha is sometimes referred to or sold by other names, such as petroleum naphtha, petroleum ether, ligroine, petroleum benzin, and industrial naphtha. One should confirm that it meets the requirements shown in 6.1.

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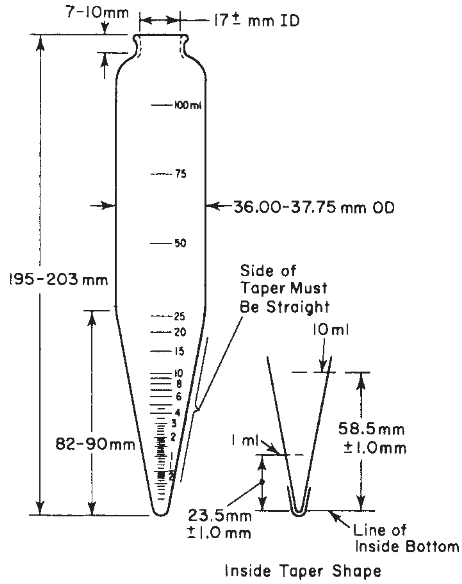
[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.06 on Analysis of Lubricants.

Current edition approved June 10, 2002. Published August 2002. Originally published as D 91–21T. Last previous edition D 91–97.

[2] This test method has been adopted for use by government agencies to replace Method 3101 of Federal Test Method Standard No. 791b.

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



NOTE—For volumetric tolerances see Table 1.

FIG. 1 ASTM Cone-Shaped Centrifuge Tube

TABLE 1 Calibration Tolerances for 200 mm Centrifuge Tube

Range, mL	Subdivision, mL	Volume Tolerance, mL
0 to 0.1	0.05	±0.02
Above 0.1 to 0.3	0.05	±0.03
Above 0.3 to 0.5	0.05	±0.05
Above 0.5 to 1.0	0.10	±0.05
Above 1.0 to 2.0	0.10	±0.10
Above 2.0 to 3.0	0.20	±0.10
Above 3.0 to 5.0	0.5	±0.20
Above 5.0 to 10	1.0	±0.50
Above 10 to 25	5.0	±1.00
Above 25 to 100	25	±1.00

TABLE 2 Rotation Speeds for Centrifuges of Various Diameters

Diameter of Swing, mm[A]	Rpm at 600 rcf	Rpm at 700 rcf
483	1490	1610
508	1450	1570
533	1420	1530
559	1390	1500

[A] Measured in millimetres between tips of opposite tubes when in rotating position.

7. Sampling

7.1 For sampling techniques, see Practices D 4057 or Practice D 4177.

8. Procedure

8.1 Add 10 ± 1 mL of the oil to be tested in each of two clean, dry centrifuge tubes at room temperature. Fill each tube to the 100-mL mark with hexanes and close tightly with a softened cork (not a rubber stopper). Then invert each tube at least 20 times, allowing the liquid to drain thoroughly from the tapered tip of the tube each time. Place the tubes in a water bath at 32 to 35°C for 5 ± 1 min. Momentarily remove the corks to relieve any pressure, and invert each tube again at least 20 times exactly as before. The success of this method depends to a large degree upon having a thoroughly homogeneous mixture which will drain quickly and completely from the tapered tip when the tube is inverted.

8.2 Balance the two centrifuge tubes or pairs of tubes with their respective trunnion cups and place them on opposite sides of the centrifuge head. Then whirl them for 10 min at a rate sufficient to produce a relative centrifugal force (rcf) between 600 and 700 at the tips of the whirling tubes (see 5.2). Repeat this operation until the volume of sediment in each tube remains constant for three consecutive readings. In general, not more than four whirlings will be required for oils having a low precipitation number.

9. Calculation and Report

9.1 Read the volume of the solid sediment at the bottom of each centrifuge tube, estimating to 0.1 mL or closer if possible. If the two readings differ by not more than 0.1 mL, report the mean of the two as the *ASTM Precipitation Number*. If the two readings differ by more than 0.1 mL, make two more determinations and report the average of the four determinations.

10. Precision and Bias

10.1 The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

10.2 *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:

Precipitation Number	Repeatability
0.00 to 1.20	10 % of Mean

10.3 *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:

Precipitation Number	Reproducibility
0.00 to 1.20	30 % of Mean

10.4 *Bias*—This procedure is empirical, precipitation number is defined solely by this procedure, therefore, no statement of bias can be made.

11. Keywords

11.1 asphaltenes; lubricating oils; precipitation number

Designation: D 445 – 04

An American National Standard
British Standard 2000: Part 71:1990

Designation: 71/1/97

Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)[1]

This standard is issued under the fixed designation D 445; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This test method specifies a procedure for the determination of the kinematic viscosity, ν, of liquid petroleum products, both transparent and opaque, by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity, η, can be obtained by multiplying the kinematic viscosity, ν, by the density, ρ, of the liquid.

NOTE 1—For the measurement of the kinematic viscosity and viscosity of bitumens, see also Test Methods D 2170 and D 2171.

1.2 The result obtained from this test method is dependent upon the behavior of the sample and is intended for application to liquids for which primarily the shear stress and shear rates are proportional (Newtonian flow behavior). If, however, the viscosity varies significantly with the rate of shear, different results may be obtained from viscometers of different capillary diameters. The procedure and precision values for residual fuel oils, which under some conditions exhibit non-Newtonian behavior, have been included.

1.3 The range of kinematic viscosities covered by this test method is from 0.2 to 300 000 mm^2/s (see Table A1.1) at all temperatures (see 6.3 and 6.4). The precision has only been determined for those materials, kinematic viscosity ranges and temperatures as shown in the footnotes to the precision section.

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

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

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers
D 1193 Specification for Reagent Water
D 1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer
D 1480 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer
D 1481 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer
D 2162 Test Method for Basic Calibration of Master Viscometers and Viscosity Oil Standards
D 2170 Test Method for Kinematic Viscosity of Asphalts (Bitumens)
D 2171 Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer
D 6074 Guide for Characterizing Hydrocarbon Lubricant Base Oils
D 6617 Practice for Laboratory Bias Detection Using Single Test Result from Standard Material
E 1 Specification for ASTM Liquid-in-Glass Thermometers
E 77 Test Method for Inspection and Verification of Thermometers

2.2 *ISO Standards:*[3]

ISO 3104 Petroleum Products—Transparent and Opaque Liquids—Determination of Kinematic Viscosity and Calculation of Dynamic Viscosity
ISO 3105 Glass Capillary Kinematic Viscometers—Specification and Operating Instructions

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved May 1, 2004. Published June 2004. Originally approved in 1937. Last previous edition approved in 2003 as D 445–03.

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

[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 American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

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

ISO 3696 Water for Analytical Laboratory Use—Specification and Test Methods
ISO 5725 Accuracy (trueness and precision) of measurement methods and results.
ISO 9000 Quality Management and Quality Assurance Standards—Guidelines for Selection and Use
ISO 17025 General Requirements for the Competence of Testing and Calibration Laboratories

2.3 *NIST Standards:*[4]
NIST Technical Note 1297, Guideline for Evaluating and Expressing the Uncertainty of NIST Measurement Results
NIST GMP 11
NIST Special Publication 819

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *automated viscometer*, *n*—apparatus which, in part or in whole, has mechanized one or more of the procedural steps indicated in 11 or 12 without changing the principle or technique of the basic manual apparatus. The essential elements of the apparatus in respect to dimensions, design and operational characteristics are not changed. The measured result from the apparatus does not require correction to bring it into correlation with the basic manual apparatus. The precision of the apparatus shall be of statistical equivalence to, or better (has less variability) than the manual apparatus.

3.1.1.1 *Discussion*—Automated viscometers have the capability to mimic some operation of the test method while reducing or removing the need for manual intervention or interpretation. Apparatus which determine kinematic viscosity by physical techniques that are different than those used in this test method are not considered to be Automated Viscometers.

3.1.2 *density*, *n*—the mass per unit volume of a substance at a given temperature.

3.1.3 *dynamic viscosity*, *n*—the ratio between the applied shear stress and rate of shear of a liquid.

3.1.3.1 *Discussion*—It is sometimes called the coefficient of dynamic viscosity or, simply, viscosity. Thus dynamic viscosity is a measure of the resistance to flow or deformation of a liquid.

3.1.3.2 *Discussion*—The term dynamic viscosity can also be used in a different context to denote a frequency-dependent quantity in which shear stress and shear rate have a sinusodial time dependence.

3.1.4 *kinematic viscosity*, *n*—the resistance to flow of a fluid under gravity.

3.1.4.1 *Discussion*—For gravity flow under a given hydrostatic head, the pressure head of a liquid is proportional to its density, ρ. For any particular viscometer, the time of flow of a fixed volume of fluid is directly proportional to its kinematic viscosity, ν, where $\nu = \eta/\rho$, and η is the dynamic viscosity coefficient.

4. Summary of Test Method

4.1 The time is measured for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled and known temperature. The kinematic viscosity (determined value) is the product of the measured flow time and the calibration constant of the viscometer. Two such determinations are needed from which to calculate a kinematic viscosity result that is the average of two acceptable determined values.

5. Significance and Use

5.1 Many petroleum products, and some non-petroleum materials, are used as lubricants, and the correct operation of the equipment depends upon the appropriate viscosity of the liquid being used. In addition, the viscosity of many petroleum fuels is important for the estimation of optimum storage, handling, and operational conditions. Thus, the accurate determination of viscosity is essential to many product specifications.

6. Apparatus

6.1 *Viscometers*—Use only calibrated viscometers of the glass capillary type, capable of being used to determine kinematic viscosity within the limits of the precision given in the precision section.

6.1.1 Viscometers listed in Table A1.1, whose specifications meet those given in Specifications D 446 and in ISO 3105 meet these requirements. It is not intended to restrict this test method to the use of only those viscometers listed in Table A1.1. Annex A1 gives further guidance.

6.1.2 *Automated Viscometers*—Automated apparatus may be used as long as they mimic the physical conditions, operations or processes of the manual apparatus they replace. Any viscometer, temperature measuring device, temperature control, temperature controlled bath or timing device incorporated in the automated apparatus shall conform to the specification for these components as stated in 6 of this test method. The automated apparatus shall be capable of determining kinematic viscosity of a certified viscosity reference standard within the limits stated in 9.2.1 and Section 17.

6.2 *Viscometer Holders*—Use viscometer holders to enable all viscometers which have the upper meniscus directly above the lower meniscus to be suspended vertically within 1° in all directions. Those viscometers whose upper meniscus is offset from directly above the lower meniscus shall be suspended vertically within 0.3° in all directions (see Specifications D 446 and ISO 3105).

6.2.1 Viscometers shall be mounted in the constant temperature bath in the same manner as when calibrated and stated on the certificate of calibration. See Specifications D 446, see Operating Instructions in Annexes A1–A3. For those viscometers which have Tube L (see Specifications D 446) held vertical, vertical alignment shall be confirmed by using (*1*) a holder ensured to hold Tube L vertical, or (*2*) a bubble level mounted on a rod designed to fit into Tube L, or (*3*) a plumb line suspended from the center of Tube L, or (*4*) other internal means of support provided in the constant temperature bath.

6.3 *Temperature-Controlled Bath*—Use a transparent liquid bath of sufficient depth such, that at no time during the measurement of flow time, any portion of the sample in the

[4] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 3460, Gaithersburg, MD 20899-3460.

viscometer is less than 20 mm below the surface of the bath liquid or less than 20 mm above the bottom of the bath.

6.3.1 *Temperature Control*—For each series of flow time measurements, the temperature control of the bath liquid shall be such that within the range from 15 to 100°C, the temperature of the bath medium does not vary by more than ±0.02°C of the selected temperature over the length of the viscometer, or between the position of each viscometer, or at the location of the thermometer. For temperatures outside this range, the deviation from the desired temperature must not exceed ±0.05°C.

6.4 *Temperature Measuring Device in the Range from 0 to 100°C*—Use either calibrated liquid-in-glass thermometers (Annex A2) of an accuracy after correction of ±0.02°C or better, or any other thermometric device of equal or better accuracy.

6.4.1 If calibrated liquid-in-glass thermometers are used, the use of two thermometers is recommended. The two thermometers shall agree within 0.04°C.

6.4.2 Outside the range from 0 to 100°C, use either calibrated liquid-in-glass thermometers of an accuracy after correction of ±0.05°C or better, or any other thermometric device of equal or better accuracy. When two temperature measuring devices are used in the same bath, they shall agree within ±0.1°C.

6.4.3 When using liquid-in-glass thermometers, such as those in Table A2.1, use a magnifying device to read the thermometer to the nearest ⅕ division (for example, 0.01°C or 0.02°F) to ensure that the required test temperature and temperature control capabilities are met (see 10.1). It is recommended that thermometer readings (and any corrections supplied on the certificates of calibrations for the thermometers) be recorded on a periodic basis to demonstrate compliance with the test method requirements. This information can be quite useful, especially when investigating issues or causes relating to testing accuracy and precision.

6.5 *Timing Device*—Use any timing device that is capable of taking readings with a discrimination of 0.1 s or better and has an accuracy within ±0.07 % (see Annex A3) of the reading when tested over the minimum and maximum intervals of expected flow times.

6.5.1 Electrical timing devices may be used if the current frequency is controlled to an accuracy of 0.05 % or better. Alternating currents, as provided by some public power systems, are intermittently rather than continuously controlled. When used to actuate electrical timing devices, such control can cause large errors in kinematic viscosity flow time measurements.

7. Reagents and Materials

7.1 *Chromic Acid Cleaning Solution*, or a nonchromium-containing, strongly oxidizing acid cleaning solution. (**Warning—**Chromic acid is a health hazard. It is toxic, a recognized carcinogen, highly corrosive, and potentially hazardous in contact with organic materials. If used, wear a full face-shield and full-length protective clothing including suitable gloves. Avoid breathing vapor. Dispose of used chromic acid carefully as it remains hazardous. Nonchromium-containing, strongly oxidizing acid cleaning solutions are also highly corrosive and potentially hazardous in contact with organic materials, but do not contain chromium which has special disposal problems.)

7.2 *Sample Solvent*, completely miscible with the sample. Filter before use.

7.2.1 For most samples a volatile petroleum spirit or naphtha is suitable. For residual fuels, a prewash with an aromatic solvent such as toluene or xylene may be necessary to remove asphaltenic material.

7.3 *Drying Solvent*, a volatile solvent miscible with the sample solvent (see 7.2) and water (see 7.4). Filter before use.

7.3.1 Acetone is suitable. (**Warning—**Extremely flammable.)

7.4 *Water*, deionized or distilled and conforming to Specification D 1193 or Grade 3 of ISO 3696. Filter before use.

8. Certified Viscosity Reference Standards

8.1 Certified viscosity reference standards shall be certified by a laboratory that has been shown to meet the requirements of ISO 17025 by independent assessment. Viscosity standards shall be traceable to master viscometer procedures described in Test Method D 2162.

8.2 The uncertainty of the certified viscosity reference standard shall be stated for each certified value (k = 2, 95% confidence). See ISO 5725 or NIST 1297.

9. Calibration and Verification

9.1 *Viscometers*—Use only calibrated viscometers, thermometers, and timers as described in Section 6.

9.2 *Certified Viscosity Reference Standards*[5] *(*Table A1.2*)*—These are for use as confirmatory checks on the procedure in the laboratory.

9.2.1 If the determined kinematic viscosity does not agree within the acceptable tolerance band, as calculated from Annex A4, of the certified value, recheck each step in the procedure, including thermometer and viscometer calibration, to locate the source of error. Annex A1 gives details of standards available.

NOTE 2—In previous issues of Test Method D 445, limits of ±0.35% of the certified value have been used. The data to support the limit of ±0.35% cannot be verified. Annex A4 provides instructions on how to determine the tolerance band. The tolerance band combines both the uncertainty of the certified viscosity reference standard as well as the uncertainty of the laboratory using the certified viscosity reference standard.

9.2.1.1 As an alternative to the calculation in Annex A4, the approximate tolerance bands in Table 1 may be used.

9.2.2 The most common sources of error are caused by particles of dust lodged in the capillary bore and temperature measurement errors. It must be appreciated that a correct result obtained on a standard oil does not preclude the possibility of a counterbalancing combination of the possible sources of error.

9.3 The calibration constant, C, is dependent upon the gravitational acceleration at the place of calibration and this

[5] The ASTM Viscosity Oil Standards are available in 1-pt (0.47 L) containers. Purchase orders should be addressed to the Cannon Instrument Co., P.O. Box 16, State College, PA 16804. Shipment will be made as specified or by best means.

TABLE 1 Approximate Tolerance Bands

NOTE—The tolerance bands were determined using Practice D 6617. The calculation is documented in Research Report D02–1490.

Viscosity of Reference Material, mm^2/s	Tolerance Band
< 10	±0.30%
10 to 100	±0.32%
100 to 1000	±0.36%
1000 to 10 000	±0.42%
10 000 to 100 000	±0.54%
> 100 000	±0.73%

must, therefore, be supplied by the standardization laboratory together with the instrument constant. Where the acceleration of gravity, *g*, differs by more that 0.1 %, correct the calibration constant as follows:

$$C_2 = (g_2/g_1) \times C_1 \quad (1)$$

where the subscripts 1 and 2 indicate, respectively, the standardization laboratory and the testing laboratory.

10. General Procedure for Kinematic Viscosity

10.1 Adjust and maintain the viscometer bath at the required test temperature within the limits given in 6.3.1 taking account of the conditions given in Annex A2 and of the corrections supplied on the certificates of calibration for the thermometers.

10.1.1 Thermometers shall be held in an upright position under the same conditions of immersion as when calibrated.

10.1.2 In order to obtain the most reliable temperature measurement, it is recommended that two thermometers with valid calibration certificates be used (see 6.4).

10.1.3 They should be viewed with a lens assembly giving approximately five times magnification and be arranged to eliminate parallax errors.

10.2 Select a clean, dry, calibrated viscometer having a range covering the estimated kinematic viscosity (that is, a wide capillary for a very viscous liquid and a narrower capillary for a more fluid liquid). The flow time shall not be less than 200 s or the longer time noted in Specifications D 446.

10.2.1 The specific details of operation vary for the different types of viscometers listed in Table A1.1. The operating instructions for the different types of viscometers are given in Specifications D 446.

10.2.2 When the test temperature is below the dew point, fill the viscometer in the normal manner as required in 11.1. To ensure that moisture does not condense or freeze on the walls of the capillary, draw the test portion into the working capillary and timing bulb, place rubber stoppers into the tubes to hold the test portion in place, and insert the viscometer into the bath. After insertion, allow the viscometer to reach bath temperature, and the remove the stoppers. When performing manual viscosity determinations, do not use those viscometers which cannot be removed from the constant temperature bath for charging the sample portion.

10.2.2.1 The use of loosely packed drying tubes affixed to the open ends of the viscometer is permitted, but not required. If used, the drying tubes shall fit the design of the viscometer and not restrict the flow of the sample by pressures created in the instrument.

10.2.3 Viscometers used for silicone fluids, fluorocarbons, and other liquids which are difficult to remove by the use of a cleaning agent, shall be reserved for the exclusive use of those fluids except during their calibration. Subject such viscometers to calibration checks at frequent intervals. The solvent washings from these viscometers shall not be used for the cleaning of other viscometers.

11. Procedure for Transparent Liquids

11.1 Charge the viscometer in the manner dictated by the design of the instrument, this operation being in conformity with that employed when the instrument was calibrated. If the sample is thought or known to contain fibers or solid particles, filter through a 75 µm screen, either prior to or during charging (see Specifications D 446).

NOTE 3—To minimize the potential of particles passing through the filter from aggregating, it is recommended that the time lapse between filtering and charging be kept to a minimum.

11.1.1 In general, the viscometers used for transparent liquids are of the type listed in Table A1.1, A and B.

11.1.2 With certain products which exhibit *gel-like* behavior, exercise care that flow time measurements are made at sufficiently high temperatures for such materials to flow freely, so that similar kinematic viscosity results are obtained in viscometers of different capillary diameters.

11.1.3 Allow the charged viscometer to remain in the bath long enough to reach the test temperature. Where one bath is used to accommodate several viscometers, never add or withdraw, or clean a viscometer while any other viscometer is in use for measuring a flow time.

11.1.4 Because this time will vary for different instruments, for different temperatures, and for different kinematic viscosities, establish a safe equilibrium time by trial.

11.1.4.1 Thirty minutes should be sufficient except for the highest kinematic viscosities.

11.1.5 Where the design of the viscometer requires it, adjust the volume of the sample to the mark after the sample has reached temperature equilibrium.

11.2 Use suction (if the sample contains no volatile constituents) or pressure to adjust the head level of the test sample to a position in the capillary arm of the instrument about 7 mm above the first timing mark, unless any other value is stated in the operating instructions for the viscometer. With the sample flowing freely, measure, in seconds to within 0.1 s, the time required for the meniscus to pass from the first to the second timing mark. If this flow time is less than the specified minimum (see 10.2), select a viscometer with a capillary of smaller diameter and repeat the operation.

11.2.1 Repeat the procedure described in 11.2 to make a second measurement of flow time. Record both measurements.

11.2.2 From the two measurements of flow time, calculate two determined values of kinematic viscosity.

11.2.3 If the two determined values of kinematic viscosity calculated from the flow time measurements agree within the stated determinability figure (see 17.1.1) for the product, use the average of these determined values to calculate the kinematic viscosity result to be reported. Record the result. If not, repeat the measurements of flow times after a thorough cleaning and drying of the viscometers and filtering (where required, see 11.1) of the sample until the calculated kinematic viscosity determinations agree with the stated determinability.

11.2.4 If the material or temperature, or both, is not listed in 17.1.1, for temperatures between 15 and 100ºC, use as an estimate of the determinability 0.20% and 0.35% for temperatures outside this range.

12. Procedure for Opaque Liquids

12.1 For steam-refined cylinder oils and black lubricating oils, proceed to 12.3 ensuring a thoroughly representative sample is used. The kinematic viscosity of residual fuel oils and similar waxy products can be affected by the previous thermal history and the following procedure described in 12.1.1-12.2.2 shall be followed to minimize this.

12.1.1 In general, the viscometers used for opaque liquids are of the reverse-flow type listed in Table A1.1, C.

12.1.2 Heat in the original container, in an oven, at 60 ± 2°C for 1 h.

12.1.3 Thoroughly stir the sample with a suitable rod of sufficient length to reach the bottom of the container. Continue stirring until there is no sludge or wax adhering to the rod.

12.1.4 Recap the container tightly and shake vigorously for 1 min to complete the mixing.

12.1.4.1 With samples of a very waxy nature or oils of high kinematic viscosity, it may be necessary to increase the heating temperature above 60°C to achieve proper mixing. The sample should be sufficiently fluid for ease of stirring and shaking.

12.2 Immediately after completing 12.1.4, pour sufficient sample to fill two viscometers into a 100-mL glass flask and loosely stopper.

12.2.1 Immerse the flask in a bath of boiling water for 30 min. (**Warning—**Exercise care as vigorous boil-over can occur when opaque liquids which contain high levels of water are heated to high temperatures.)

12.2.2 Remove the flask from the bath, stopper tightly, and shake for 60 s.

12.3 Two determinations of the kinematic viscosity of the test material are required. For those viscometers that require a complete cleaning after each flow time measurement, two viscometers may be used. A single viscometer in which an immediate, repeat flow time measurement can be made without cleaning may also be used for the two measurements of flow time and calculation of kinematic viscosity. Charge two viscometers in the manner dictated by the design of the instrument. For example, for the cross-arm or the BS U-tube viscometers for opaque liquids, filter the sample through a 75-µm filter into two viscometers previously placed in the bath. For samples subjected to heat treatment, use a preheated filter to prevent the sample coagulating during the filtration.

12.3.1 Viscometers which are charged before being inserted into the bath may need to be preheated in an oven prior to charging the sample. This is to ensure that the sample will not be cooled below test temperature.

12.3.2 After 10 min, adjust the volume of the sample (where the design of the viscometer requires) to coincide with the filling marks as in the viscometer specifications (see Specifications D 446).

12.3.3 Allow the charged viscometers enough time to reach the test temperature (see 12.3.1). Where one bath is used to accommodate several viscometers, never add or withdraw, or clean a viscometer while any other viscometer is in use for measuring flow time.

12.4 With the sample flowing freely, measure in seconds to within 0.1 s, the time required for the advancing ring of contact to pass from the first timing mark to the second. Record the measurement.

12.4.1 In the case of samples requiring heat treatment described in 12.1 through 12.2.1, complete the measurements of flow time within 1 h of completing 12.2.2. Record the measured flow times.

12.5 Calculate kinematic viscosity, ν, in mm^2/s, from each measured flow time. Regard these as two determined values of kinematic viscosity.

12.5.1 For residual fuel oils, if the two determined values of kinematic viscosity agree within the stated determinability figure (see 17.1.1), use the average of these determined values to calculate the kinematic viscosity result to be reported. Record the result. If the calculated kinematic viscosities do not agree, repeat the measurements of flow times after thorough cleaning and drying of the viscometers and filtering of the sample. If the material or temperature, or both, is not listed in 17.1.1, for temperatures between 15 and 100°C use as an estimate of the determinability 1.0 %, and 1.5 % for temperatures outside this range; it must be realized that these materials can be non-Newtonian, and can contain solids which can come out of solution as the flow time is being measured.

13. Cleaning of Viscometer

13.1 Between successive determinations of kinematic viscosity, clean the viscometer thoroughly by several rinsings with the sample solvent, followed by the drying solvent (see 7.3). Dry the tube by passing a slow stream of filtered dry air through the viscometer for 2 min or until the last trace of solvent is removed.

13.2 Periodically clean the viscometer with the cleaning solution (**Warning**—see 7.1), for several hours to remove residual traces of organic deposits, rinse thoroughly with water (7.4) and drying solvent (see 7.3), and dry with filtered dry air or a vacuum line. Remove any inorganic deposits by hydrochloric acid treatment before the use of cleaning acid, particularly if the presence of barium salts is suspected. (**Warning—**It is essential that alkaline cleaning solutions are not used as changes in the viscometer calibration can occur.)

14. Calculation

14.1 Calculate each of the determined kinematic viscosity values, ν_1 and ν_2, from the measured flow times, t_1 and t_2, and the viscometer constant, C, by means of the following equation:

$$\nu_{1,2} = C \cdot t_{1,2} \quad (2)$$

where:

$\nu_{1,2}$ = determined kinematic viscosity values for ν_1 and ν_2, respectively, mm^2/s,

C = calibration constant of the viscometer, mm^2/s^2, and

$t_{1,2}$ = measured flow times for t_1 and t_2, respectively, s.
Calculate the kinematic viscosity result, ν, as an average of ν_1 and ν_2 (see 11.2.3 and 12.5.1).

14.2 Calculate the dynamic viscosity, η, from the calculated kinematic viscosity, ν, and the density, ρ, by means of the following equation:

$$\eta = \nu \times \rho \times 10^{-3} \quad (3)$$

where:

η = dynamic viscosity, mPa·s,
ρ = density, kg/m^3, at the same temperature used for the determination of the kinematic viscosity, and
ν = kinematic viscosity, mm^2/s.

14.2.1 The density of the sample can be determined at the test temperature of the kinematic viscosity determination by an appropriate method such as Test Methods D 1217, D 1480, or D 1481.

15. Expression of Results

15.1 Report the test results for the kinematic or dynamic viscosity, or both, to four significant figures, together with the test temperature.

16. Report

16.1 Report the following information:

16.1.1 Type and identification of the product tested,

16.1.2 Reference to this test method or a corresponding international standard,

16.1.3 Result of the test (see Section 15),

16.1.4 Any deviation, by agreement or otherwise, from the procedure specified,

16.1.5 Date of the test, and

16.1.6 Name and address of the test laboratory.

17. Precision

17.1 *Comparison of Determined Values*:

17.1.1 *Determinability (d)*—The difference between successive determined values obtained by the same operator in the same laboratory using the same apparatus for a series of operations leading to a single result, would in the long run, in the normal and correct operation of this test method, exceed the values indicated only in one case in twenty:

Base oils at 40 and 100°C[6]	0.0020 y	(0.20 %)
Formulated oils at 40 and 100°C[7]	0.0013 y	(0.13 %)
Formulated oils at 150°C[8]	0.015 y	(1.5 %)
Petroleum wax at 100°C[9]	0.0080 y	(0.80 %)
Residual fuel oils at 80 and 100°C[10]	0.011 (y + 8)	
Residual fuel oils at 50°C[10]	0.017 y	(1.7 %)
Additives at 100°C[11]	$0.00106\ y^{1.1}$	
Gas oils at 40°C[12]	0.0013 (y+1)	
Jet fuels at –20°C[13]	0.0018 y	(0.18 %)

where: *y* is the average of determined values being compared.

17.2 *Comparison of Results*:

17.2.1 *Repeatability (r)*—The difference between successive results obtained by the same operator in the same laboratory 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 indicated only in one case in twenty:

Base oils at 40 and 100°C[6]	0.0011 x	(0.11 %)
Formulated oils at 40 and 100°C[7]	0.0026 x	(0.26 %)
Formulated oils at 150°C[8]	0.0056 x	(0.56 %)
Petroleum wax at 100°C[9]	$0.0141\ x^{1.2}$	
Residual fuel oils at 80 and 100°C[10]	0.013 (x + 8)	
Residual oils at 50°C[10]	0.015 x	(1.5 %)
Additives at 100°C[11]	$0.00192\ x^{1.1}$	
Gas oils at 40°C[12]	0.0043 (x+1)	
Jet fuels at –20°C[13]	0.007 x	(0.7 %)

where: *x* is the average of results being compared.

17.3 *Repeatability (r)*—The difference between successive results obtained by the same operator in the same laboratory 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 indicated only in one case in twenty:

Base oils at 40 and 100°C[6]	0.0011 x	(0.11 %)
Formulated oils at 40 and 100°C[7]	0.0026 x	(0.26 %)
Formulated oils at 150°C[8]	0.0056 x	(0.56 %)
Petroleum wax at 100°C[9]	$0.0141\ x^{1.2}$	
Residual fuel oils at 80 and 100°C[10]	0.013 (x + 8)	
Residual oils at 50°C[10]	0.015 x	(1.5 %)
Additives at 100°C[11]	$0.00192\ x^{1.1}$	
Gas oils at 40°C[12]	0.0043 (x+1)	
Jet fuels at –20°C[13]	0.007 x	(0.7 %)

where: *x* is the average of results being compared.

17.4 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators working in different laboratories on nominally identical test material would, in the long run, in the normal and correct

[6] These precision values were obtained by statistical examination of interlaboratory results from six mineral oils (base oils without additive package) in the range from 8 to 1005 mm^2/s at 40°C and from 2 to 43 mm^2/s at 100°C, and were first published in 1989. Precision data available from ASTM Headquarters. Request RR:D02-1331 and RR:D02-1132. See Guide D 6074.

[7] These precision values were obtained by statistical examination of interlaboratory results from seven fully formulated engine oils in the range from 36 to 340 mm^2/s at 40°C and from 6 to 25 mm^2/s at 100°C, and were first published in 1991. Precision data available from ASTM Headquarters. Request RR:D02-1332. See Guide D 6071.

[8] These precision values were obtained by statistical examination of interlaboratory results for eight fully formulated engine oils in the range from 7 to 19 mm^2/s at 150°C, and first published in 1991. Precision data available from ASTM Headquarters. Request RR:D02-1333. See Guide D 6074.

[9] These precision values were obtained by statistical examination of interlaboratory results from five petroleum waxes in the range from 3 to 16 mm^2/s at 100°C, and were first published in 1988. Precision data available from ASTM Headquarters. Request RR:D02-1334.

[10] These precision values were obtained by statistical examination of interlaboratory results from fourteen residual fuel oils in the range from 30 to 1300 mm^2/s at 50°C and from 5 to 170 mm^2/s at 80 and 100°C, and were first published in 1984. Precision data available from ASTM Headquarters. Request RR:D02-1198.

[11] These precision values were obtained by statistical examination of interlaboratory results from eight additives in the range from 145 to 1500 mm^2/s at 100°C and were first available in 1997. Precision data available from ASTM Headquarters. Request RR:D02-1421.

[12] These precision values were obtained by statistical examination of interlaboratory results from eight gas oils in the range from 1 to 13 mm^2/s at 40°C and were first available in 1997. Precision data available from ASTM Headquarters. Request RR:D02-1422.

[13] These precision values were obtained by statistical examination of interlaboratory results from nine jet fuels in the range from 4.3 to 5.6 mm^2/s at– 20°C and were first available in 1997. Precision data available from ASTM Headquarters. Request RR:D02-1420.

operation of this test method, exceed the values indicated below only in one case in twenty.

Base oils at 40 and 100°C[6]	0.0065 x	(0.65 %)
Formulated oils at 40 and 100°C[7]	0.0076 x	(0.76 %)
Formulated oils at 150°C[8]	0.018 x	(1.8 %)
Petroleum wax at 100°C[9]	$0.0366\ x^{1.2}$	
Residual fuel oils at 80 and 100°C[10]	0.04 (x + 8)	
Residual oils at 50°C[10]	0.074 x	(7.4 %)
Additives at 100°C[11]	$0.00862\ x^{1.1}$	
Gas oils at 40°C[12]	0.0082 (x+1)	
Jet fuels at –20°C[13]	0.019 x	(1.9 %)

where: x is the average of results being compared.

17.5 The precision for used oils has not been determined but is expected to be poorer than that for formulated oils. Because of the extreme variability of such used oils, it is not anticipated that the precision of used oils will be determined.

17.6 The precision for specific automated viscometers has not been determined. However, an analysis has been made of a large data set including both automated and manual viscometers over the temperature range of 40 to 100°C. The reproducibility of automated viscometer data is not statistically significantly different than the reproducibility of manual viscometer data. It is also shown that there is no bias of the automated data in comparison to the manual data.[14]

18. Keywords

18.1 dynamic viscosity; kinematic viscosity; viscometer; viscosity

[14] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1498.

ANNEXES

(Mandatory Information)

A1. VISCOMETER TYPES AND CERTIFIED VISCOSITY REFERENCE STANDARDS

A1.1 Viscometer Types

A1.1.1 Table A1.1 lists capillary viscometers commonly in use for viscosity determinations on petroleum products. For specifications, operating instructions, and calibration, refer to specifications in Specifications D 446.

A1.1.2 Table A1.2 lists certified viscosity reference standards.

TABLE A1.1 Viscometer Types

Viscometer Identification	Kinematic Viscosity Range,[A] mm^2/s
A. Ostwald Types for Transparent Liquids	
Cannon-Fenske routine[B]	0.5 to 20 000
Zeitfuchs	0.6 to 3 000
BS/U-tube[B]	0.9 to 10 000
BS/U/M miniature	0.2 to 100
SIL[B]	0.6 to 10 000
Cannon-Manning semi-micro	0.4 to 20 000
Pinkevitch[B]	0.6 to 17 000
B. Suspended-level Types for Transparent Liquids	
BS/IP/SL[B]	3.5 to 100 000
BS/IP/SL(S)[B]	1.05 to 10 000
BS/IP/MSL	0.6 to 3 000
Ubbelohde[B]	0.3 to 100 000
FitzSimons	0.6 to 1 200
Atlantic[B]	0.75 to 5 000
Cannon-Ubbelohde(A), Cannon Ubbelohde dilution[B](B)	0.5 to 100 000
Cannon-Ubbelohde semi-micro	0.4 to 20 000
C. Reverse-flow Types for Transparent and Opaque Liquids	
Cannon-Fenske opaque	0.4 to 20 000
Zeitfuchs cross-arm	0.6 to 100 000
BS/IP/RF U-tube reverse-flow	0.6 to 300 000
Lantz-Zeitfuchs type reverse-flow	60 to 100 000

[A] Each range quoted requires a series of viscometers. To avoid the necessity of making a kinetic energy correction, these viscometers are designed for a flow time in excess of 200 s except where noted in Specifications D 446.

[B] In each of these series, the minimum flow time for the viscometers with lowest constants exceeds 200 s.

TABLE A1.2 Certified Viscosity Reference Standards

Designation	Approximate Kinematic Viscosity, mm²/s					
	20°C	25°C	40°C	50°C	80 °C	100°C
S3	4.6	4.0	2.9	...	...	1.2
S6	11	8.9	5.7	...	...	1.8
S20	44	34	18	...	...	3.9
S60	170	120	54	...	...	7.2
S200	640	450	180	...	...	17
S600	2400	1600	520	280	67	32
S2000	8700	5600	1700	...	...	75
S8000	37 000	23 000	6700	...	...	...
S30 000	...	81 000	23 000	11 000	...	...

A2. KINEMATIC VISCOSITY TEST THERMOMETERS

A2.1 Short-Range Specialized Thermometer

A2.1.1 Use a short-range specialized thermometer conforming to the generic specification given in Table A2.1 and Table A2.2 and to one of the designs shown in Fig. A2.1.

A2.1.2 The difference in the designs rests mainly in the position of the ice point scale. In Design A, the ice point is within the scale range, in Design B, the ice point is below the scale range, and in Design C, the ice point is above the scale range.

A2.2 Calibration

A2.2.1 Use liquid-in-glass thermometers with an accuracy after correction of 0.02°C or better, calibrated by a laboratory meeting the requirements of ISO 9000 or ISO 17025, and carrying certificates confirming that the calibration is traceable to a national standard. As an alternative, use thermometric devices such as platinum resistance thermometers, of equal or better accuracy, with the same certification requirements.

A2.2.2 The scale correction of liquid-in-glass thermometers can change during storage and use, and therefore regular re-calibration is required. This is most conveniently achieved in a working laboratory by means of a re-calibration of the ice point, and all of the main scale corrections altered for the change seen in the ice point.

TABLE A2.1 General Specification for Thermometers

NOTE—Table A2.2 gives a range of ASTM, IP, and ASTM/IP thermometers that comply with the specification in Table A2.1, together with their designated test temperatures. See Specification E 1 and Test Method E 77.

Immersion		Total
Scale marks:		
Subdivisions	°C	0.05
Long lines at each	°C	0.1 and 0.5
Numbers at each	°C	1
Maximum line width	mm	0.10
Scale error at test temperature, max	°C	0.1
Expansion chamber:		
Permit heating to	°C	105 up to 90, 120 between 90 and 95 130 between 95 and 105, 170 above 105
Total length	mm	300 to 310
Stem outside diameter	mm	6.0 to 8.0
Bulb length	mm	45 to 55
Bulb outside diameter	mm	no greater than stem
Length of scale range	mm	40 to 90

TABLE A2.2 Complying Thermometers

Thermometer No.	Test Temperature		Thermometer No.	Test Temperature	
	°C	°F		°C	°F
ASTM 132C, IP 102C	150		ASTM 128C, F/IP 33C	0	32
ASTM 110C, F/IP 93C	135	275	ASTM 72C, F/IP 67C	−17.8	0
ASTM 121C/IP 32C	98.9, 100	210, 212	ASTM 127C/IP 99C	−20	−4
			ASTM 126C, F/IP 71C	−26.1	−20
ASTM 129C, F/IP 36C	93.3	200	ASTM 73C, F/IP 68C	−40	−40
ASTM 48C, F/IP 90C	82.2	180	ASTM 74C, F/IP 69C	−53.9	−65
IP 100C	80				
ASTM 47C, F/IP 35C	60	140			
ASTM 29C, F/IP 34C	54.4	130			
ASTM 46C F/IP 66C	50	122			
ASTM 120C/IP 92C	40				
ASTM 28C, F/IP 31C	37.8	100			
ASTM 118C, F	30	86			
ASTM 45C, F/IP 30C	25	77			
ASTM 44C, F/IP 29C	20	68			

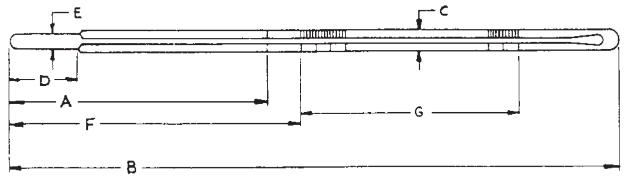
A2.2.2.1 The interval for ice-point recalibration shall be no longer than six months (see NIST GMP 11). For new thermometers, monthly checking for the first six months is recommended. A change of one or more scale divisions in the ice point means that the thermometer may have been overheated or damaged, and it may be out of calibration. Such thermometers shall be removed from service until inspected, or recalibrated, or both. A complete recalibration of the thermometer, while permitted, is not necessary in order to meet the accuracy ascribed to this design thermometer (see NIST Special Publication 819). Any change in ice-point correction shall be added to the other corrections of the original Report of Calibration.

A2.2.2.2 Other thermometric devices, if used, will also require periodic recalibration. Keep records of all recalibration.

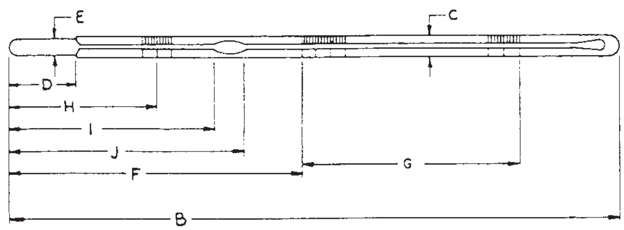
A2.2.3 *Procedure for Ice-point Recalibration of Liquid-in-glass Thermometers*.

A2.2.3.1 Unless otherwise listed on the certificate of calibration, the recalibration of calibrated kinematic viscosity thermometers requires that the ice-point reading shall be taken within 60 min after being at test temperature for not less than 3 min.

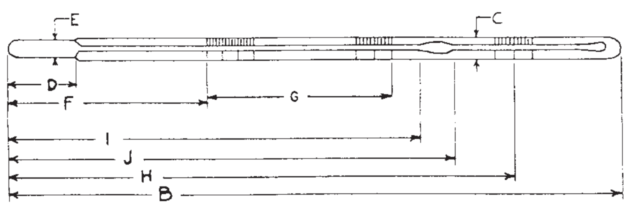
A2.2.3.2 Select clear pieces of ice, preferably made from distilled or pure water. Discard any cloudy or unsound portions. Rinse the ice with distilled water and shave or crush into small pieces, avoiding direct contact with the hands or any chemically unclean objects. Fill the Dewar vessel with the



(a)



(b)



(c)

FIG. A2.1 Thermometer Designs

crushed ice and add sufficient water to form a slush, but not enough to float the ice. As the ice melts, drain off some of the water and add more crushed ice. Insert the thermometer, and pack the ice gently about the stem, to a depth approximately one scale division below the 0°C graduation.

A2.2.3.3 After at least 3 min have elapsed, tap the thermometer gently and repeatedly at right angles to its axis while making observations. Successive readings taken at least 1 min apart shall agree within 0.005°C.

A2.2.3.4 Record the ice-point readings and determine the thermometer correction at this temperature from the mean reading. If the correction is found to be higher or lower than that corresponding to a previous calibration, change the correction at all other temperatures by the same value.

A2.2.3.5 During the procedure, apply the following conditions:

(*1*) The thermometer shall be supported vertically.

(*2*) View the thermometer with an optical aid that gives a magnification of approximately five and also eliminates parallax.

(*3*) Express the ice-point reading to the nearest 0.005°C.

A2.2.4 When in use, immerse the thermometric device to the same depth as when it was fully calibrated. For example, if a liquid-in-glass thermometer was calibrated at the normal total immersion condition, it shall be immersed to the top of the mercury column with the remainder of the stem and the expansion volume at the uppermost end exposed to room temperature and pressure. In practice, this means that the top of the mercury column shall be within a length equivalent to four scale divisions of the surface of the medium whose temperature is being measured.

A2.2.4.1 If this condition cannot be met, then an extra correction may be necessary.

A3. TIMER ACCURACY

A3.1 Regularly check timers for accuracy and maintain records of such checks.

A3.1.1 Time signals as broadcast by the National Institute of Standards and Technology are a convenient and primary standard reference for calibrating timing devices. The following can be used to an accuracy of 0.1 s:

WWV	Fort Collins, CO	2.5, 5, 10, 15, 20 MHz
WWVH	Kauai, HI	2.5, 5, 10, 15, MHz
CHU	Ottawa, Canada	3.33, 7.335, 14.67 MHz

A3.1.2 Radio broadcast of voice and audio on a telephone line at phone 303-499-7111. Additional time services are available from the National Institute of Standards and Technology.

A4. CALCULATION OF ACCEPTABLE TOLERANCE ZONE (BAND) TO DETERMINE CONFORMANCE WITH A CERTIFIED REFERENCE MATERIAL

A4.1 Determine the standard deviation for site uncertainty, σ_{site}, from a laboratory quality control program.

A4.1.1 If the standard deviation for site uncertainty, σ_{site}, is not known, use the value 0.19%.

A4.2 Determine the combined extended uncertainty (CEU) of the accepted reference value (ARV) of the certified reference material (CRM) from the supplier's label or included documentation.

A4.3 Calculate the standard error of the accepted reference value (SEARV) by dividing the CEU by the coverage factor, *k*, listed on the supplier's label or included documentation.

A4.3.1 If the coverage factor, *k*, is not known, use the value 2.

A4.4 Construct the acceptable tolerance zone:

$$TZ = \pm 1.44 \sqrt{\sigma_{site}^2 + SE_{ARV}^2}$$

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D 445–03) that may impact the use of this standard.

(1) Replaced A2.2.2.1.
(2) Added text to 12.3.
(3) Added text to 6.5.

Standard Test Methods for Oxidation Characteristics of Extreme-Pressure Lubrication Oils[1]

This standard is issued under the fixed designation D 2893; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 These test methods (A and B) cover the determination of the oxidation characteristics of extreme-pressure fluid lubricants, gear oils, or mineral oils.

NOTE 1—The changes in the lubricant resulting from these test methods are not always necessarily associated with oxidation of the lubricant. Some changes may be due to thermal degradation.

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.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 91 Test Method for Precipitation Number of Lubricating Oils

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (the Calculation of Dynamic Viscosity)[2]

D 943 Test Method for Oxidation Characteristics of Inhibited Mineral Oils

E 1 Specification for ASTM Liquid-in-Glass Thermometers

3. Summary of Test Method

3.1 The oil sample is subjected to a temperature of 95°C (Test Method A) or 121°C (Test Method B) in the presence of dry air for 312 h.

3.2 The oil is then tested for precipitation number and increase in kinematic viscosity.

4. Significance and Use

4.1 These test methods have been widely used to measure the oxidation stability of extreme pressure lubricating fluids, gear oils, and mineral oils.

5. Apparatus

5.1 *Heating Bath or Block*, thermostatically controlled, capable of maintaining the oil sample in the test tube at a temperature of 95 ± 0.2°C (Test Method A), or 121 ± 1.0°C (Test Method B) and large enough to hold the desired number of oxidation cells immersed in the heating bath or block to a depth of approximately 350 mm. The liquid heating bath shall be fitted with a suitable stirring device to provide a uniform temperature throughout the bath.

5.2 *Test Tubes*, of borosilicate glass, 41 ± 0.5 mm inside diameter and 600 mm in length are required, each fitted with a slotted cork (Note 2) stopper into which shall be inserted a glass air delivery tube of 4 to 5 mm of inside diameter. The length of the air delivery tube shall be such that one end reaches to within 6 mm of the bottom of the tube and the other end projects 60 to 80 mm from the cork stopper.

NOTE 2—New corks should be used for each run.

5.3 *Flowmeter*, one to each test tube, capable of measuring an air flow of 10 L/h with an accuracy of ±0.5 L/h.

5.4 *Thermometer*—ASTM Solvent Distillation Thermometer having a range from 76 to 126°C and conforming to the requirement for Thermometer 40C as prescribed in Specification E 1. Alternatively, calibrated thermocouples may be used.

5.5 *Air Supply*—Oil-free, dried air at constant pressure shall be supplied to each flowmeter.

5.6 *Air Dryer*—Before being supplied to the flowmeters, the air shall be passed through a drying tower packed with indicating grade of anhydrous calcium sulfate or equivalent. The quantity of dessicant should be sufficient to last for the entire test.

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[1] These test methods are under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.09 on Oxidation.

Current edition approved May 1, 2004. Published May 2004. Originally approved in 1970. Last previous edition approved in 2003 as D 2893–03.

[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.

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

6. Preparation of Apparatus

6.1 *Cleaning of Oxidation Cells*—Clean glassware with a suitable cleaning solution. (**Warning**—Causes severe burns. A recognized carcinogen. Strong oxidizer, contact with other material may cause fire. Hygroscropic.)

NOTE 3—While other suitable cleaning solutions are now available, the round robin used glassware cleaned with chromic acid. Other cleaning solutions such as NoChromix and Micro Clean have been found suitable. In a referee situation, glassware shall be cleaned by a cleaning solution satisfactory to all parties involved.

7. Procedure

7.1 Adjust the heating bath to a temperature high enough to maintain the oil in the desired number of oxidation cells at the required temperature of 95 ± 0.2°C (Test Method A) or 121 ± 1.0°C (Test Method B). Determine the viscosity at 100°C by Test Method D 445/IP 71 and the precipitation number by Test Method D 91, on each sample.

7.2 Pour 300 mL of each oil sample into a test tube and immerse the test tube in the heating bath so that the heating medium is at least 50 mm above the level of the oil sample. Place the corks and air delivery tubes in the test tubes making sure that the lower ends of the tubes are within 6 mm of the bottoms of the test tubes.

7.3 Connect the air delivery tubes to the dried air supply through the flowmeters. Adjust the flow of air to 10 ± 0.5 L/h. Check the temperature of the oil samples and the rate of air flow every hour and make necessary adjustments. Once the oil samples have reached the desired temperature of 95 ± 0.2°C (Test Method A) or 121 ± 1.0°C (Test Method B), initiate the start of the test.

NOTE 4—When using multi-cell baths, one way of checking the temperature of the oil samples can be to use a dummy cell in the bath, similar to the way it is used in Test Method D 943.

7.4 Maintain the air flow and bath or block temperature constant, checking them periodically for the duration of the test.

7.5 Remove the test tubes from the bath or block 312 ± 1 h (13 days) after the start of the test. Mix each oil sample thoroughly and test them for viscosity at 100°C by Test Method D 445/IP 71 and precipitation number by Test Method D 91.

8. Calculation

8.1 Calculate the kinematic viscosity increase as follows:

$$\text{Viscosity increase, \%} = [(B - A)/A] \times 100 \quad (1)$$

where:
A = kinematic viscosity on original sample, and
B = kinematic viscosity after oxidation.

9. Report

9.1 On the original sample, and on the oxidized sample at the termination of test, report the precipitation number determined in accordance with Test Method D 91.

9.2 Report the percent increase in viscosity at 100°C as determined in Section 8.

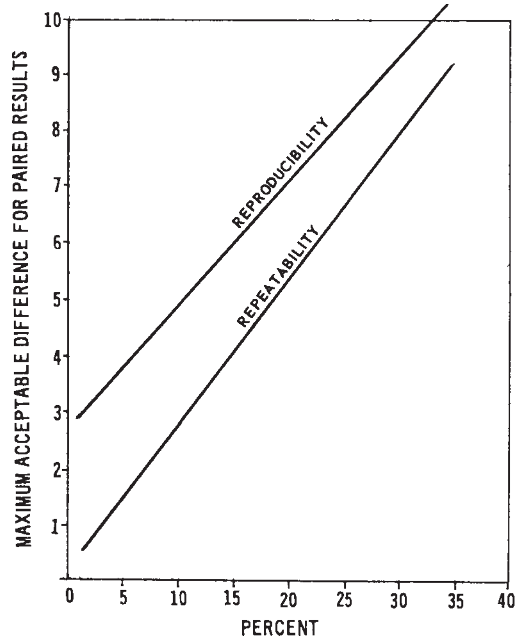


FIG. 1 Precision Data, Viscosity Increase

10. Precision and Bias (Test Method A)[3]

10.1 The precision of this test method is not known to have been obtained in accordance with currently accepted guidelines (for example, in Committee D02 Research Report RR:D02-1007[4]).

10.2 *Viscosity Increase*:

10.2.1 *Repeatability*—Duplicate results by the same operator shall be considered suspect if they differ by more than the maximum acceptable difference for repeatability as shown in Fig. 1.

10.2.2 *Reproducibility*—The results submitted by each of two laboratories shall be considered suspect if they differ by more than the maximum acceptable difference for reproducibility as shown in Fig. 1.

10.3 *Precipitation Number, Increase*:

10.3.1 *Repeatability*—Duplicate results by the same operator shall be considered suspect if they differ by more than the maximum acceptable difference for repeatability as shown in Fig. 2.

10.3.2 *Reproducibility*—The results submitted by each of two laboratories shall be considered suspect if they differ by more than the maximum acceptable difference for reproducibility as shown in Fig. 2.

10.4 *Bias*—The procedure in this test method has no bias because the value of these changes can only be defined in terms of a test method.

11. Precision and Bias (Test Method B)[5]

11.1 *Viscosity Increase*:

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1150.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1007.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1539.

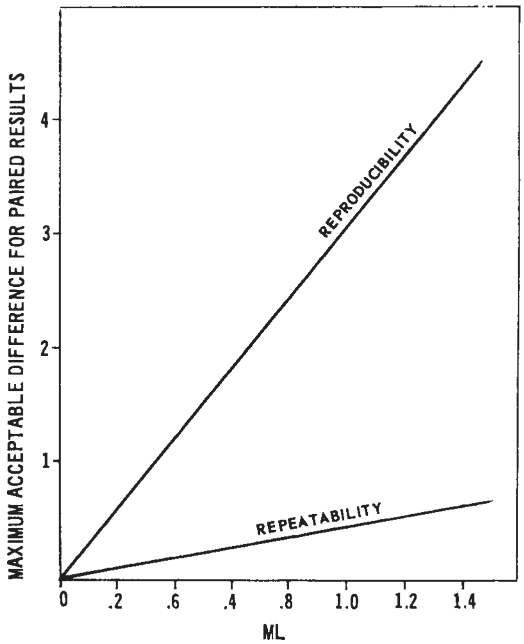


FIG. 2 Precision Data, Precipitation Number Increase

11.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of this test method, exceed the flowing values only in one in twenty:

$$\text{Repeatability} = 0.30X \quad (2)$$

where:
X = the mean value.

11.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators in different laboratories on identical material would, in the long run, exceed the following values only in one case in twenty:

$$\text{Reproducibility} = 1.1X \quad (3)$$

where:
X = the mean value.

NOTE 5—This precision statement was prepared with data on six oils tested by six cooperators. The oils covered values of 0-20 % viscosity increase.

11.2 The precision for the precipitation number was not determined.

11.3 *Bias*—The procedure in this test method has no bias, because the value of these changes can only be defined in terms of a test method.

12. Keywords

12.1 extreme pressure gear oils; oxidation testing—petroleum; stability—oxidation; stability—thermal

SUMMARY OF CHANGES

Subcommittee D02.09 has identified the location of selected changes to this standard since the last issue (D 2893–03) that may impact the use of this standard.

(1) Added Test Method D 943 to the Referenced Documents
(2) Clarified that stirring device is used only for liquid baths in 5.1.
(3) Added Note 4.

Harris Testing Laboratories, Inc.

CERTIFICATE OF ANALYSIS

CLIENT:
Megatrol Inc.
9469 S 500 W
Sandy, Ut. 84070

PRODUCT: **FE**[26] Premier
MARKS: NONE
DATE RECEIVED: 02/11/2008
LAB NO: HH0410-1202-P
SUBMITTED BY: Jon Rose

			SPECIFICATIONS	
METHOD	**TEST**	**RESULT**	**MIN.**	**MAX.**
D-92	**Flash Point, Degrees C**	**370**	**XXX**	**XXX**
		XXX	**XXX**	**XXX**
	Flash Point, Degrees F	**698**	**XXX**	**XXX**

Comments:

Amos Mwangi

Date issued:
02/11/2008

CHEMIST

Designation: D 92 – 02b

An American National Standard
American Association State
Highway and Transportation Officials Standard
AASHTO No.: T48
DIN 51 376

Designation: 36/84 (89)

Standard Test Method for Flash and Fire Points by Cleveland Open Cup Tester[1]

This standard is issued under the fixed designation D 92; 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.

INTRODUCTION

This flash point and fire point test method is a dynamic method and depends on definite rates of temperature increases to control the precision of the test method. Its primary use is for viscous materials having flash point of 79°C (175°F) and above. It is also used to determine fire point, which is a temperature above the flash point, at which the test specimen will support combustion for a minimum of 5 s. Do not confuse this test method with Test Method D 4206, which is a sustained burning test, open cup type, at a specific temperature of 49°C (120°F).

Flash point values are a function of the apparatus design, the condition of the apparatus used, and the operational procedure carried out. Flash point can therefore only be defined in terms of a standard test method, and no general valid correlation can be guaranteed between results obtained by different test methods, or with test apparatus different from that specified.

1. Scope *

1.1 This test method describes the determination of the flash point and fire point of petroleum products by a manual Cleveland open cup apparatus or an automated Cleveland open cup apparatus.

NOTE 1—The precisions for fire point were not determined in the current interlaboratory program. Fire point is a parameter that is not commonly specified, although in some cases, knowledge of this flammability temperature may be desired.

1.2 This test method is applicable to all petroleum products with flash points above 79°C (175°F) and below 400°C (752°F) except fuel oils.

NOTE 2—This test method may occasionally be specified for the determination of the fire point of a fuel oil. For the determination of the flash points of fuel oils, use Test Method D 93. Test Method D 93 should also be used when it is desired to determine the possible presence of small, but significant, concentrations of lower flash point substances that may escape detection by Test Method D 92. Test Method D 1310 can be employed if the flash point is known to be below 79°C (175°F).

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

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see 6.4, 7.1, 11.1.3, and 11.2.4.

2. Referenced Documents

2.1 *ASTM Standards:*

D 93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester[2]

D 140 Practice for Sampling Bituminous Materials[3]

D 1310 Test Method for Flash Point and Fire Points of Liquids by Tag Open-Cup Apparatus[4]

D 4057 Practice for Manual Sampling of Petroleum and

[1] This test method is under the joint jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.08 on Volatility. In the IP, this test method is under the jurisdiction of the Standardization Committee. This test method was adopted as a joint ASTM-IP standard in 1965.

Current edition approved Dec. 10, 2002. Published March 2003. Originally approved in 1921. Last previous edition approved in 2002 as D 92–02a.

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

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

[4] *Annual Book of ASTM Standards*, Vol 06.01.

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

Petroleum Products[5]

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[5]

D 4206 Test Method for Sustained Burning of Liquid Mixtures by the Setaflash Tester (Open-Cup)[6]

E 1 Specification for ASTM Thermometers[7]

E 300 Practice for Sampling Industrial Chemicals[8]

2.2 *IP Standard:*[9]

Specifications for IP Standard Thermometers

2.3 *ISO Standards:*[10]

Guide 34 Quality Systems Guidelines for the Production of Reference Materials

Guide 35 Certification of Reference Material—General and Statistical Principles

3. Terminology

3.1 *Definitions:*

3.1.1 *dynamic, adj—in petroleum products,* the condition where the vapor above the test specimen and the test specimen are not in temperature equilibrium at the time that the ignition source is applied.

3.1.1.1 *Discussion*—This is primarily caused by the heating of the test specimen at the constant prescribed rate with the vapor temperature lagging behind the test specimen temperature.

3.1.2 *fire point, n—in petroleum products,* the lowest temperature corrected to a barometric pressure of 101.3 kPa (760 mm Hg), at which application of an ignition source causes the vapors of a test specimen of the sample to ignite and sustain burning for a minimum of 5 s under specified conditions of test.

3.1.3 *flash point, n—in petroleum products,* the lowest temperature corrected to a barometric pressure of 101.3 kPa (760 mm Hg), at which application of an ignition source causes the vapors of a specimen of the sample to ignite under specified conditions of test.

3.1.3.1 *Discussion*—The test specimen is deemed to have flashed when a flame appears and instantaneously propagates itself over the entire surface of the test specimen.

3.1.3.2 *Discussion*—When the ignition source is a test flame, the application of the test flame can cause a blue halo or an enlarged flame prior to the actual flash point. This is not a flash point and shall be ignored.

4. Summary of Test Method

4.1 Approximately 70 mL of test specimen is filled into a test cup. The temperature of the test specimen is increased rapidly at first and then at a slower constant rate as the flash point is approached. At specified intervals a test flame is passed across the cup. The flash point is the lowest liquid temperature at which application of the test flame causes the vapors of the test specimen of the sample to ignite. To determine the fire point, the test is continued until the application of the test flame causes the test specimen to ignite and sustain burning for a minimum of 5 s.

5. Significance and Use

5.1 The flash point is one measure of the tendency of the test specimen to form a flammable mixture with air under controlled laboratory conditions. It is only one of a number of properties that should be considered in assessing the overall flammability hazard of a material.

5.2 Flash point is used in shipping and safety regulations to define flammable and combustible materials. Consult the particular regulation involved for precise definitions of these classifications.

5.3 Flash point can indicate the possible presence of highly volatile and flammable materials in a relatively nonvolatile or nonflammable material. For example, an abnormally low flash point on a test specimen of engine oil can indicate gasoline contamination.

5.4 This test method shall be used to measure and describe the properties of materials, products, or assemblies in response to heat and a test flame under controlled laboratory conditions and shall not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test method may be used as elements of a fire risk assessment that takes into account all of the factors that are pertinent to an assessment of the fire hazard of a particular end use.

5.5 The fire point is one measure of the tendency of the test specimen to support combustion.

6. Apparatus

6.1 *Cleveland Open Cup Apparatus (manual)*—This apparatus consists of the test cup, heating plate, test flame applicator, heater, and supports described in detail in the Annex A1. The assembled manual apparatus, heating plate, and cup are illustrated in Figs. 1-3, respectively. Dimensions are listed with the figures.

6.2 *Cleveland Open Cup Apparatus (automated)*—This apparatus is an automated flash point instrument that shall perform the test in accordance with Section 11 Procedure. The apparatus shall use the test cup with the dimensions as described in Annex A1 and the application of the test flame shall be as described in Annex A1.

6.3 *Temperature Measuring Device*—A thermometer having the range as shown below and conforming to the requirements prescribed in Specification E 1 or in the Specifications for IP Standard Thermometers, or an electronic temperature measuring device, such as a resistance thermometer or thermocouple. The device shall exhibit the same temperature response as the mercury thermometers.

Temperature Range	Thermometer Number ASTM	IP
−6 to +400°C	11C	28C
20 to 760°F	11F	

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

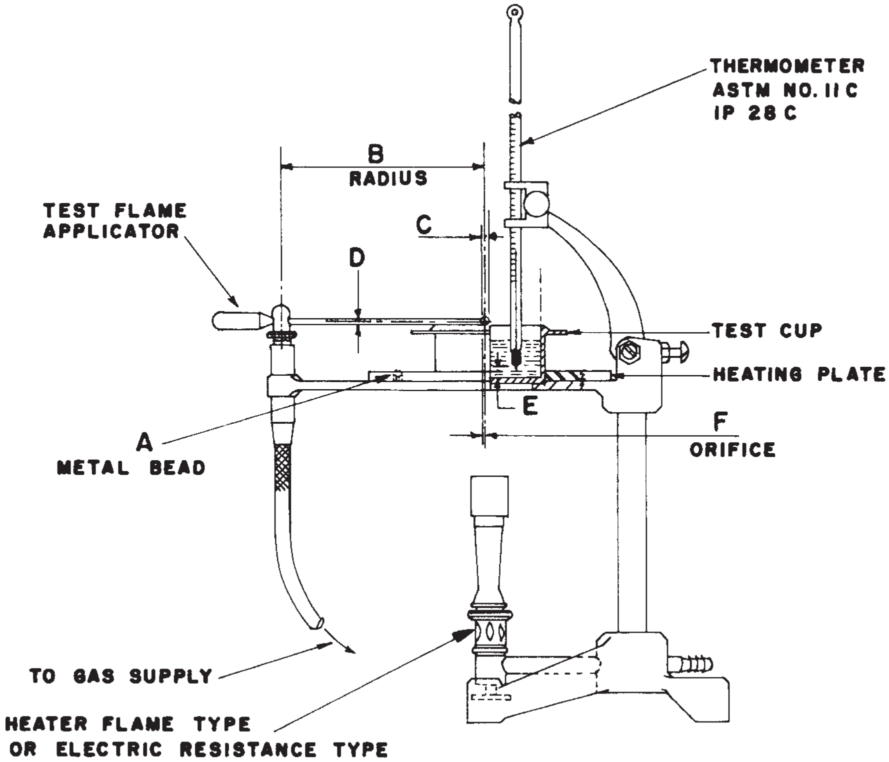
[6] *Annual Book of ASTM Standards*, Vol 06.01.

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

[8] Discontinued—See 2000 *Annual Book of ASTM Standards*, Vol 15.05.

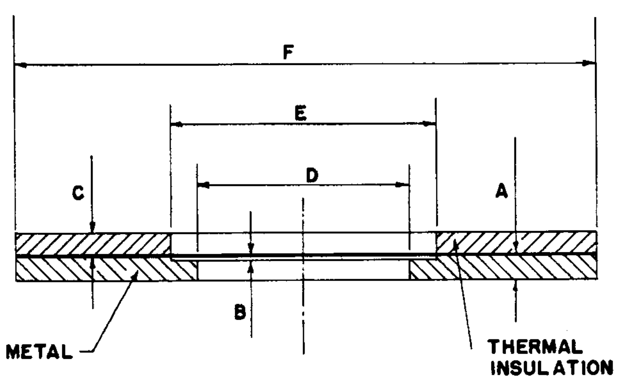
[9] Available from the Institute of Petroleum, 61 New Cavendish St., London, W1M 8AR, U.K.

[10] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.



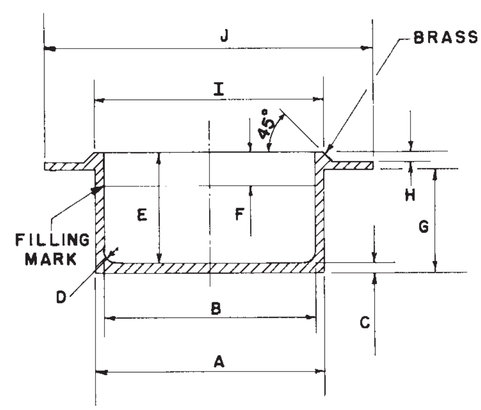
	millimetres		inches	
	min	max	min	max
A—Diameter	3.2	4.8	0.126	0.189
B—Radius	152	nominal	6	nominal
C—Diameter	1.6	nominal	0.063	nominal
D		2		0.078
E	6	7	0.236	0.276
F—Diameter	0.8	nominal	0.031	nominal

FIG. 1 Cleveland Open Cup Apparatus



	millimetres		inches	
	min	max	min	max
A	6	7	0.236	0.276
B	0.5	1.0	0.020	0.039
C	6	7	0.236	0.276
D—Diameter	55	56	2.165	2.205
E—Diameter	69.5	70.5	2.736	2.776
F—Diameter	146	159	5.748	6.260

FIG. 2 Heating Plate



	millimetres		inches	
	min	max	min	max
A	67.5	69	2.658	2.717
B	63	64	2.480	2.520
C	2.8	3.5	0.110	0.138
D—Radius	4	nominal	0.157	nominal
E	32.5	34	1.280	1.339
F	9	10	0.354	0.394
G	31	32.5	1.221	1.280
H	2.8	3.5	0.110	0.138
I	67	70	2.638	2.756
J	97	100	3.819	3.937

FIG. 3 Cleveland Open Cup

6.4 *Test Flame*—Natural gas (methane) flame and bottled gas (butane, propane) flame have been found acceptable for use as the ignition source. The gas flame device is described in detail in Annex A1. (**Warning—**Gas pressure supplied to the apparatus must not be allowed to exceed 3 kPa (12 in.) of water pressure.)

NOTE 3—A shield, of the approximate dimensions 460 mm (18 in.) square and 610 mm (24 in.) high, or other suitable dimensions, and having an open front is recommended to prevent drafts from disturbing the vapors above the test cup.

7. Reagents and Materials

7.1 *Cleaning Solvents*—Use suitable technical grade solvent capable of cleaning out the test specimen from the test cup and drying the test cup. Some commonly used solvents are toluene and acetone. (**Warning—**Toluene, acetone, and many solvents are flammable. Health hazard. Dispose of solvents and waste material in accordance with local regulations.)

8. Sampling

8.1 Obtain a sample in accordance with the instructions given in Practices D 140, D 4057, D 4177 or E 300.

8.2 At least 70 mL of sample is required for each test. Refer to Practice D 4057.

8.3 Erroneously high flash points may be obtained if precautions are not taken to avoid the loss of volatile material. Do not open containers unnecessarily; this will prevent loss of volatile material and possible introduction of moisture. Do not make a transfer of the sample unless the sample temperature is at least 56°C (100°F) below the expected flash point. When possible, flash point should be the first test performed on a sample and the sample should be stored at low temperature.

NOTE 4—Typical sample storage temperature is normal room temperature or lower.

8.4 Do not store samples in gas-permeable containers since volatile material may diffuse through the walls of the enclosure. Samples in leaky containers are suspect and not a source of valid results.

8.5 Light hydrocarbons may be present in the form of gases, such as propane or butane, and may not be detected by testing because of losses during sampling and filling of the test cup. This is especially evident on heavy residuums or asphalts from solvent extraction processes.

8.6 Samples of very viscous materials can be warmed until they are reasonably fluid before they are tested. However, no sample shall be heated more than is absolutely necessary. It shall never be heated above a temperature of 56°C (100°F) below its expected flash point. When the sample has been heated above this temperature, allow the sample to cool until it is at least 56°C (100°F) below the expected flash point before transferring.

NOTE 5—Typically, the sample containers for these types of samples will remain closed during the warming process.

8.7 Samples containing dissolved or free water can be dehydrated with calcium chloride or by filtering through a qualitative filter paper or a loose plug of dry absorbent cotton. Samples of very viscous materials can be warmed until they are reasonably fluid before they are filtered, but they shall not be heated for prolonged periods or above a temperature of 56°C (100°F) below its expected flash point.

NOTE 6—If the sample is suspected of containing volatile contaminants, the treatment described in 8.6 and 8.7 should be omitted.

9. Preparation of Apparatus

9.1 Support the manual or automated apparatus on a level steady surface, such as a table.

9.2 Tests are to be performed in a draft-free room or compartment. Tests made in a laboratory hood or in any location where drafts occur are not to be relied upon.

NOTE 7—A shield, of the approximate dimensions 460 mm (18 in.) square and 610 mm (24 in.) high, or other suitable dimensions, and having an open front is recommended to prevent drafts from disturbing the vapors above the test cup.

NOTE 8—With some samples whose vapors or products of pyrolysis are objectionable, it is permissible to place the apparatus along with a shield into a hood, the draft of which is adjusted so that the vapors may be withdrawn without causing air currents over the test cup during the final 56°C (100°F) rise in temperature prior to the flash point.

9.3 Wash the test cup with the cleaning solvent to remove any test specimen or traces of gum or residue remaining from a previous test. If any deposits of carbon are present, they should be removed with a material such as a very fine grade of steel wool. Ensure that the test cup is completely clean and dry before using again. If necessary, flush the test cup with cold water and dry for a few minutes over an open flame or a hot plate to remove the last traces of solvent and water. Cool the test cup to at least 56°C (100°F) below the expected flash point before using.

9.4 Support the temperature measuring device in a vertical position with the bottom of the device located 6.4 ± 0.1 mm (1⁄4 ± 1⁄50 in.) up from the bottom of the inside of the test cup and located at a point halfway between the center and the side of the test cup on a diameter perpendicular to the arc (or line) of the sweep of the test flame and on the side opposite to the test flame applicator mounting position.

NOTE 9—The immersion line engraved on the ASTM or IP thermometer will be 2 ± 0.1 mm (5⁄64 ± 1⁄50 in.) below the level of the rim of the cup when the thermometer is properly positioned.

NOTE 10—Some automated apparatus is capable of positioning the temperature measuring device automatically. Refer to the manufacturer's instructions for proper installation and adjustment.

9.5 Prepare the manual apparatus or the automated apparatus for operation according to the manufacturer's instructions for calibrating, checking, and operating the equipment.

10. Calibration and Standardization

10.1 Adjust the automated flash point detection system, when used, according to the manufacturer's instructions.

10.2 Calibrate the temperature measuring device according to the manufacturer's instructions.

10.3 Verify the performance of the manual apparatus or the automated apparatus at least once per year by determining the flash point of a certified reference material (CRM), such as those listed in Annex A2, which is reasonably close to the expected temperature range of the samples to be tested. The material shall be tested according to the procedure of this test method and the observed flash point obtained in 11.1.10 or 11.2.5 shall be corrected for barometric pressure (see Section 12). The flash point obtained shall be within the limits stated in

Table A2.1 for the identified CRM or within the limits calculated for an unlisted CRM (see Annex A2).

10.4 Once the performance of the apparatus has been verified, the flash point of secondary working standards (SWSs) can be determined along with their control limits. These secondary materials can then be utilized for more frequent performance checks (see Annex A2).

NOTE 11—The verification fluid is a material with a predetermined, interlaboratory tested, flash point temperature that is used to verify proper operation of the apparatus. Calibration is undertaken by the operator according to the apparatus manufacturers' instructions should the result of the verification be outside the stated reproducibility.

10.5 When the flash point obtained is not within the limits stated in 10.3 or 10.4, check the condition and operation of the apparatus to ensure conformity with the details listed in Annex A1, especially in regard to the position of the temperature measuring device, the application of the test flame, and the heating rate. After adjustment of the apparatus, repeat the test with a fresh test specimen (see 10.3) with special attention to the procedural details prescribed in Section 11.

11. Procedure

11.1 *Manual Apparatus*:

11.1.1 Fill the test cup with the sample so that the top of the meniscus of the test specimen is exactly at the filling mark, and place the test cup on the center of the heater. The temperature of the test cup and the sample shall not exceed 56°C (100°F) below the expected flash point. If too much test specimen has been added to the cup, remove the excess using a syringe or similar device for withdrawal of fluid. However, if there is test specimen on the outside of the test cup, empty, clean, and refill it. Destroy any air bubbles or foam on the surface of the test specimen with a sharp knife or other suitable device and maintain the required level of test specimen. If a foam persists during the final stages of the test, terminate the test and disregard any results.

11.1.2 Solid material shall not be added to the test cup. Solid or viscous samples shall be heated until they are fluid before being poured into the test cup; however, the temperature of the sample during heating shall not exceed 56°C (100°F) below the expected flash point.

11.1.3 Light the test flame and adjust it to a diameter of 3.2 to 4.8 mm (1⁄8 to 3⁄16 in.) or to the size of the comparison bead, if one is mounted on the apparatus (see Annex A1). (**Warning—**Gas pressure supplied to the apparatus must not be allowed to exceed 3 kPa (12 in.) of water pressure.) (**Warning—**Exercise care when using a gas test flame. If it should be extinguished it will not ignite the vapors in the test cup, and the gas for the test flame that then enters the vapor space can influence the result.) (**Warning—**The operator shall exercise care and take appropriate safety precautions during the initial application of the test flame since test specimens containing low-flash material can give an abnormally strong flash when the test flame is first applied.) (**Warning—**The operator shall exercise care and take appropriate safety precautions during the performance of this test method. The temperatures attained during this test, up to 400°C (752°F), are considered hazardous.)

11.1.4 Apply heat initially at such a rate that the temperature as indicated by the temperature measuring device increases 14 to 17°C (25 to 30°F)/min. When the test specimen temperature is approximately 56°C (100°F) below the expected flash point, decrease the heat so that the rate of temperature rise during the last 28°C (50°F) before the flash point is 5 to 6°C (9 to 11°F)/min.

11.1.5 Apply the test flame when the temperature of the test specimen is approximately 28°C below the expected flash point and each time thereafter at a temperature reading that is a multiple of 2°C. Pass the test flame across the center of the test cup at right angles to the diameter, which passes through the temperature measuring device. With a smooth, continuous motion, apply the test flame either in a straight line or along the circumference of a circle having a radius of at least 150 ± 1 mm (6.00 ± 0.039 in.). The center of the test flame shall move in a horizontal plane not more than 2 mm (5⁄64 in.) above the plane of the upper edge of the test cup and passing in one direction only. At the time of the next test flame application, pass the test flame in the opposite direction of the preceding application. The time consumed in passing the test flame across the test cup in each case shall be approximately 1 ± 0.1 s.

NOTE 12—When determining the flash point of asphalt, it is recommended to carefully move fully to one side, such as with a spatula, any surface film formed before each application of the ignition source. Available data indicate that higher flash point is observed for asphalt samples when surface film formed is not moved aside, compared to the flash point observed when the surface film is moved aside prior to the application of the ignition source.

NOTE 13—An alternative to the moving aside of the formed surface film can be found in Appendix X1.

11.1.6 During the last 28°C (50°F) rise in temperature prior to the expected flash point, care shall be taken to avoid disturbing the vapors in the test cup with rapid movements or drafts near the test cup.

11.1.7 When a foam persists on top of the test specimen during the last 28°C (50°F) rise in temperature prior to the expected flash point, terminate the test and disregard any results.

11.1.8 Meticulous attention to all details relating to the test flame, size of the test flame, rate of temperature increase, and rate of passing the test flame over the test specimen is required for proper results.

11.1.9 When testing materials where the expected flash point temperature is not known, bring the material to be tested and the test cup to a temperature no greater than 50°C (122°F), or when the material required heating to be transferred into the test cup, bring the material to that temperature. Apply the test flame, in the manner described in 11.1.5, beginning at least 5°C (9°F) above the starting temperature. Continue heating the test specimen at 5 to 6°C (9 to 11°F)/min and testing the material every 2°C (5°F) as described in 11.1.5 until the flash point is obtained.

NOTE 14—Flash point results determined in an unknown expected flash point mode should be considered approximate. This value can be used as the expected flash point when a fresh specimen is tested in the standard mode of operation.

11.1.10 Record, as the observed flash point, the reading on the temperature measuring device at the time the test flame causes a distinct flash in the interior of the test cup.

11.1.10.1 The sample is deemed to have flashed when a large flame appears and instantaneously propagates itself over the entire surface of the test specimen.

11.1.11 The application of the test flame can cause a blue halo or an enlarged flame prior to the actual flash point. This is not a flash point and shall be ignored.

11.1.12 When a flash point is detected on the first application of the test flame, the test shall be discontinued, the result discarded, and the test repeated with a fresh test specimen. The first application of the test flame with the fresh test specimen shall be at least 28°C (50°F) below the temperature found when the flash point was detected on the first application.

11.1.13 When the apparatus has cooled down to a safe handling temperature, less than 60°C (140°F), remove the test cup and clean the test cup and the apparatus as recommended by the manufacturer.

NOTE 15—Exercise care when cleaning the apparatus so as not to damage or dislocate the automated flash detection system, when used, or temperature measuring device. See the manufacturer's instructions for proper care and maintenance.

11.1.14 To determine the fire point, continue heating the test specimen after recording the flash point such that the test specimen temperature increases at a rate of 5 to 6°C (9 to 11°F)/min. Continue the application of the test flame at 2°C (5°F) intervals until the test specimen ignites and sustains burning for a minimum of 5 s. Record the temperature of the test specimen when the test flame, which caused the test specimen to ignite was applied. Sustain burning as the observed fire point of the test specimen.

11.1.15 When the apparatus has cooled down to a safe handling temperature, less than 60°C (140°F), remove the test cup and clean the test cup and the apparatus as recommended by the manufacturer.

11.2 *Automated Apparatus*:

11.2.1 The automated apparatus shall be capable of performing the procedure as described in 11.1, including control of the heating rate, application of the test flame, detection of the flash point, or fire point, or both, and recording the flash point or fire point, or both.

11.2.2 Fill the test cup with the sample so that the top of the meniscus of the test specimen is exactly at the filling mark, and place the test cup on the center of the heater. The temperature of the test cup and the sample shall not exceed 56°C (100°F) below the expected flash point. If too much test specimen has been added to the cup, remove the excess using a syringe or similar device for withdrawal of fluid; however, if there is test specimen on the outside of the test cup, empty, clean, and refill it. Destroy any air bubbles or foam on the surface of the test specimen with a sharp knife or other suitable device, and maintain the required level of test specimen. If a foam persists during the final stages of the test, terminate the test, and disregard any results.

11.2.3 Solid material shall not be added to the test cup. Solid or viscous samples shall be heated until they are fluid before being poured into the test cup; however, the temperature of the sample during heating shall not exceed 56°C (100°F) below the expected flash point.

11.2.4 Light the test flame, when necessary, and adjust it to a diameter of 3.2 to 4.8 mm (1⁄8 to 3⁄16 in.) or to the size of the comparison bead, if one is mounted on the apparatus. (**Warning—**Gas pressure supplied to the apparatus must not be allowed to exceed 3 kPa (12 in.) of water pressure.) (**Warning—**Exercise care when using a gas test flame. If it should be extinguished it will not ignite the vapors in the test cup, and the gas for the test flame that then enters the vapor space can influence the result.) (**Warning—**The operator shall exercise care and take appropriate safety precautions during the initial application of the test flame since test specimens containing low-flash material can give an abnormally strong flash when the test flame is first applied.) (**Warning—**The operator shall exercise care and take appropriate safety precautions during the performance of this test method. The temperatures attained during this test, up to 400°C (752°F), are considered hazardous.)

NOTE 16—Some automated apparatus can light the test flame automatically and the size of the flame is preset.

11.2.5 Start the automated apparatus according to the manufacturer's instructions. The apparatus shall follow the procedural details described in 11.1.4 through 11.1.15.

12. Calculations

12.1 Observe and record the ambient barometric pressure (see Note 17) at the time of the test. When the pressure differs from 101.3 kPa (760 mm Hg), correct the flash point or fire point, or both, as follows:

$$\text{Corrected flash point} = C + 0.25\,(101.3 - K) \quad (1)$$

$$\text{Corrected flash point} = F + 0.06\,(760 - P) \quad (2)$$

$$\text{Corrected flash point} = C + 0.033\,(760 - P) \quad (3)$$

where:
C = observed flash point, °C,
F = observed flash point, °F,
P = ambient barometric pressure, mm Hg, and
K = ambient barometric pressure, kPa.

NOTE 17—The barometric pressure used in this calculation is the ambient pressure for the laboratory at the time of test. Many aneroid barometers, such as those used at weather stations and airports, are precorrected to give sea level readings and would not give the correct reading for this test.

12.2 Using the corrected flash point or fire point, or both, as determined in 12.1, round the values to the nearest 1°C (2°F) and record.

13. Report

13.1 Report the corrected flash point or fire point value, or both, as the Test Method D 92 Cleveland open cup flash point or fire point, or both, of the test specimen.

14. Precision and Bias

14.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory test results is as follows:

14.1.1 *Repeatability*—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 the test method, exceed the following values in 1 case in 20.

Flash point	8°C (15°F)
Fire point	8°C (15°F)

14.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical 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 20.

Flash point	18°C (32°F)
Fire point	14°C (25°F)

14.2 The precision data for fire point is not known to have been developed in accordance with Precision Manual RR:D02-1007.[11]

NOTE 18—The precisions for fire point were not determined in the current interlaboratory program. Fire point is a parameter that is not commonly specified, although in some cases, this temperature may be desired.

NOTE 19—The precision for asphalt type samples which have had any formed surface film removed has not been determined.

NOTE 20—The precision for asphalt type samples which have utilized the procedure in Appendix X1 have not been determined.

14.3 *Bias*—The procedure of this test method has no bias because flash point and fire point can be defined only in terms of this test method.

14.4 *Relative Bias*—Statistical evaluation of the data did not detect any significant difference between the reproducibility variances of manual and automated Cleveland flash point results for the samples studied with the exception of multi-viscosity lubricating oil and white mineral oil. Evaluation of the data did not detect any significant difference between averages of manual and automated Cleveland flash point for the samples studied with the exception of multi-viscosity lubricating oil, which showed some bias. In any case of dispute, the flash point as determined by the manual procedure shall be considered the referee test.

14.5 The precision data for flash point were developed in a 1991 cooperative test program using seven samples of base oils, asphalt, and lubricating oils. Five laboratories participated with the manual apparatus and eight laboratories participated with the automated equipment. Information on the type of samples and their average flash point are in the research report available at ASTM Headquarters.[12]

15. Keywords

15.1 automated Cleveland open cup; Cleveland open cup; fire point open cup for flash point; flammability; flash point; petroleum products

[11] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1007.

[12] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: S15-1009.

ANNEXES

(Mandatory Information)

A1. CLEVELAND OPEN CUP TESTER

A1.1 *Test Cup,* conforming to Fig. 3 with dimensions as shown with the figure. The cup shall be made of brass or other non-rusting metal of equivalent heat conductivity. The cup may be equipped with a handle.

A1.2 *Heating Plate,* a brass, cast iron, wrought iron, or steel plate with a center hole surrounded by an area of plane depression, and a sheet of compressed insulating material (non-asbestos) that covers the metal plate except over the area of plane depression in which the test cup is supported. The essential dimensions of the heating plate are shown in Fig. 2; however, it may be square instead of round, and the metal plate may have suitable extensions for mounting the test flame applicator device and the thermometer support. Also, a metal bead, as mentioned in A1.3, may be mounted on the plate so that it extends through and slightly above a suitable small hole in the plate.

A1.3 *Ignition Source Applicator*—The device for applying the test flame may be of any suitable type. When using a test flame, it is suggested that the tip be 1.6 ± 0.05 mm (1⁄16 in.) in diameter at the end, and that the orifice be approximately 0.8 ± 0.05 mm (1⁄32 in.) in diameter. The device for operating the test flame applicator may be mounted in such a manner as to permit automated duplication of the sweep of the test flame, the radius of swing being not less than 150 mm (6 in.). The center of the test flame should be supported so that it swings in a plane not greater than 2 mm (5⁄64 in.) above the plane of the rim of the cup. It is desired that a bead, having a diameter of 3.2 to 4.8 mm (1⁄8 to 3⁄16 in.), be mounted in a convenient position on the apparatus so that the size of the test flame can be compared to it.

A1.4 *Heater*—Heat may be supplied from any convenient source. The use of a gas burner or alcohol lamp is permitted, but under no circumstances are products of combustion or free flame to be allowed to come up around the cup. An electric heater which can be controlled automatically or controlled by the user is preferred. The source of heat shall be centered under the opening of the heating plate with no local superheating. Flame-type heaters may be protected from drafts or excessive radiation by any suitable type of shield that does not project above the level of the upper surface of the heating plate.

A1.5 *Temperature Measuring Device Support*—Any convenient device that will hold the temperature measuring device in the specified position during a test and that will permit easy removal of the temperature measuring device from the test cup upon completion of a test may be used.

A1.6 *Heating Plate Support*—Any convenient support that will hold the heating plate level and steady may be employed.

A2. VERIFICATION OF APPARATUS PERFORMANCE

A2.1 *Certified Reference Material (CRM)*—CRM is a stable, pure (99 + mole % purity) hydrocarbon or other stable petroleum product with a method-specific flash point established by a method-specific interlaboratory study following RR:D02-1007[11] guidelines or ISO Guide 34 and 35.

A2.1.1 Typical values of the flash point corrected for barometric pressure for some reference materials and their typical limits are given in Table A2.1 (see Note A2.2). Suppliers of CRM's will provide certificates stating the method-specific flash point for each material of the current production batch. Calculation of the limits for these other CRM's can be determined from the reproducibility values of this test method, reduced by interlaboratory effect and then multiplied by 0.7 (see Research Report RR:S15-1008[13]).

[13] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: S15–1008.

TABLE A2.1 D 92 Typical Flash Point Values and Typical Limits for CRM

Hydrocarbon	Purity (mole %)	Flash Point (°C)	Limits (°C)
n–tetradecane	99 +	115.5	±8.0
n–hexadecane	99 +	138.8	±8.0

NOTE A2.1—Supporting data for the interlaboratory study to generate the flash point in Table A2.1 can be found in research report RR:S15-1010.[14]

NOTE A2.2—Materials, purities, flash point values, and limits stated in Table A2.1 were developed in an ASTM interlaboratory program to determine suitability of use for verification fluids in flash point test methods. Other materials, purities, flash point values, and limits can be suitable when produced according to the practices of RR:D02-1007 or ISO Guides 34 and 35. Certificates of performance of such materials should be consulted before use, as the flashpoint value will vary dependent on the composition of each CRM batch.

A2.2 *Secondary Working Standard (SWS)*—SWS is a stable, pure (99 + mole % purity) hydrocarbon, or other petroleum product whose composition is known to remain appreciably stable.

A2.2.1 Establish the mean flash point and the statistical control limits (3σ) for the SWS using standard statistical techniques.[15]

[14] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: S15–1010.

[15] *ASTM MNL7 Manual on Presentation of Data and Control Chart Analysis*, 6th ed., ASTM, 1990.

APPENDIX

(Nonmandatory Information)

X1. TECHNIQUE TO PREVENT SURFACE SKIN FORMATION WHEN TESTING FLASH POINT OF ASPHALTS BY TEST METHOD D 92

X1.1 Introduction

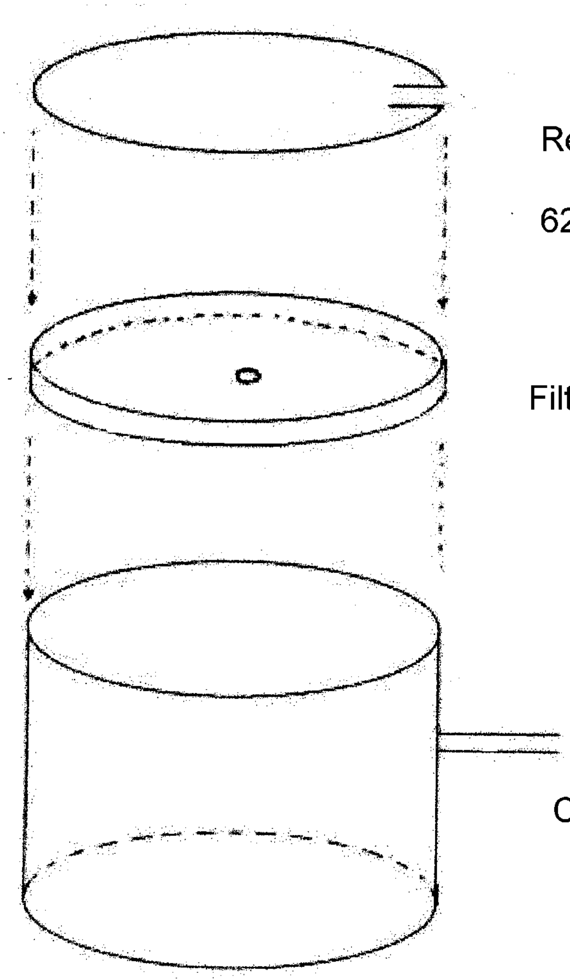
X1.1.1 This technique to prevent surface skin formation when testing flash point of asphalts by Test Method D 92 was developed by Imran Hussami of Frontier El Dorado Refining Company.

X1.2 Materials Required

X1.2.1 *Filter Paper*, qualitative, No. 417 (or equivalent), 7.5 cm diameter.

X1.2.2 *Restraining Tension-ring*, metal wire, circular, but with its 15 mm straight ends folded inwards, parallel to each other (see Fig. X1.1). Dimensions: wire about 2 mm thick, 62 to 63 outside diameter with bent ends 15 mm apart along the circumference of the circle. Total length of wire is about 210 mm. An ordinary metal coat hanger or similar material can be used to make the tension-ring.

X1.2.3 *Single-hole Punch*, (or equivalent) capable of making a 6 mm diameter hole in the center of the filter paper.



NOTE—Use of this alternate technique may cause bubbling in some samples. Bubbling could interfere with automatic flash detection devices, and it also may cause a slower heating rate in some samples.

FIG. X1.1 Technique to Prevent Surface Skin Formation When Testing Flash Point of Asphalts by Test Method D 92

X1.3 Procedure

X1.3.1 Determine the center of the filter paper by means of a ruler. Using the single-hole punch, punch a 6-mm diameter hole *in the center* of the 7.5 cm diameter qualitative filter paper.

X1.3.2 Curl up the sides of the filter paper, about 6 mm all around, and place it in the base of the Cleveland open cup flash point test cup, with the 6-mm skirt facing upward (see Fig. X1.1).

X1.3.3 Place the restraining tension-ring snugly over the curved portion of the filter paper in the base of the cup. (The tension-ring prevents the filter paper from moving upward during the test.)

X1.3.4 Fill the cup with the sample *4 to 5 mm below the filling mark* (this is to compensate for the sample that is absorbed by the filter paper which will be released during the test). (**Warning—**Filling all the way to the filling mark could produce premature flash point results.)

X1.3.5 Start the test either using a manual tester or an automatic unit (following manufacturer's instructions) and determine the flash point.

X1.3.6 Report the flash point corrected for bariometric pressure to the nearest 1°C.

SUMMARY OF CHANGES

Subcommittee D02.08 has identified the location of selected changes to this standard since the last issue (D 92–02a) that may impact the use of this standard.

(1) Added a paragraph to the Introduction about flash point values being dependent on the test procedure used.

Harris Testing Laboratories, Inc.

CERTIFICATE OF ANALYSIS

CLIENT
Megatrol Inc.
9469 S 500 W
Sandy, Ut 84070

PRODUCT: **FE^{26}** Premier
MARKS: NONE
DATE RECEIVED: 02/11/2008
LAB NO: HH0410-1202-P
SUBMITTED BY: Jon Rose

			SPECIFICATIONS	
METHOD	**TEST**	**RESULT**	**MIN.**	**MAX.**
D-97	**Pour Point, Degrees C**	**-31**	**XXX**	**XXX**

Comments:

Amos Mwangi

Date issued:
02/11/2008

CHEMIST

An American National Standard

Designation: D 97 – 04

Designation: 15/95

Standard Test Method for Pour Point of Petroleum Products[1]

This standard is issued under the fixed designation D 97; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method is intended for use on any petroleum product.[2] A procedure suitable for black specimens, cylinder stock, and nondistillate fuel oil is described in 8.8. A procedure for testing the fluidity of a residual fuel oil at a specified temperature is described in Appendix X1.

1.2 Several ASTM test methods offering alternative procedures for determining pour points using automatic apparatus are available. None of them share the same designation number as Test Method D 97. When an automatic instrument is used, the ASTM test method designation number specific to the technique shall be reported with the results.

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

2. Referenced Documents

2.1 *ASTM Standards:* [3]

D 117 Guide for Sampling, Test Methods, and Specifications for Electrical Insulating Oils of Petroleum Origin
D 396 Specification for Fuel Oils
D 1659 Test Method for Maximum Fluidity Temperature of Residual Fuel Oil[4]
D 2500 Test Method for Cloud Point of Petroleum Products
D 3245 Test Method for Pumpability of Industrial Fuel Oils
E 1 Specification for ASTM Liquid-in-Glass Thermometers

2.2 *Energy Institute Standards:*

Specifications for IP Standard Thermometers[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *black oil*, *n*—lubricant containing asphaltic materials. Black oils are used in heavy-duty equipment applications, such as mining and quarrying, where extra adhesiveness is desired.

3.1.2 *cylinder stock*, *n*—lubricant for independently lubricated engine cylinders, such as those of steam engines and air compressors. Cylinder stock are also used for lubrication of valves and other elements in the cylinder area.

3.1.3 *pour point*, *n—in petroleum products*, the lowest temperature at which movement of the test specimen is observed under prescribed conditions of test.

3.1.4 *residual fuel*, *n*—a liquid fuel containing bottoms remaining from crude distillation or thermal cracking; sometimes referred to as heavy fuel oil.

3.1.4.1 *Discussion*—Residual fuels comprise Grades 4, 5, and 6 fuel oils, as defined in Specification D 396.

4. Summary of Test Method

4.1 After preliminary heating, the sample is cooled at a specified rate and examined at intervals of 3°C for flow characteristics. The lowest temperature at which movement of the specimen is observed is recorded as the pour point.

5. Significance and Use

5.1 The pour point of a petroleum specimen is an index of the lowest temperature of its utility for certain applications.

6. Apparatus

6.1 *Test Jar*, cylindrical, of clear glass, flat bottom, 33.2 to 34.8-mm outside diameter, and 115 to 125 mm in height. The

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved April 1, 2004. Published April 2004. Originally approved in 1927, replacing D 47. Last previous edition approved in 2002 as D 97–02.

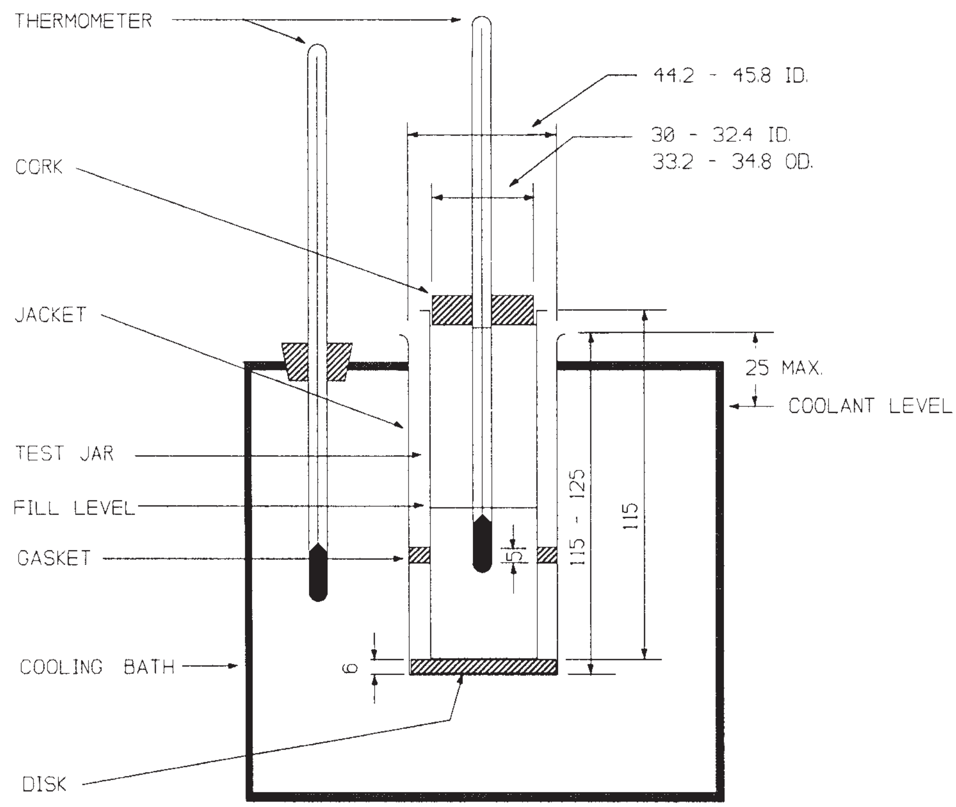
In the IP, this test method is under the jurisdiction of the Standardization Committee. This test method was adopted as a joint ASTM-IP Standard in 1965.

[2] Statements defining this test and its significance when applied to electrical insulating oils of mineral origin will be found in Guide D 117.

[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual 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.

[4] Withdrawn.

[5] Methods for Analysis and Testing, *IP Standards for Petroleum and its Products*, Part I, Vol 2.



NOTE—Dimensions are in millimetres (not to scale).

FIG. 1 Apparatus for Pour Point Test

inside diameter of the jar can range from 30.0 to 32.4 mm, within the constraint that the wall thickness be no greater than 1.6 mm. The jar shall have a line to indicate a sample height 54 ± 3 mm above the inside bottom. See Fig. 1.

6.2 *Thermometers*, having the following ranges and conforming to the requirements prescribed in Specification E 1 for thermometers:

Thermometer	Temperature Range	Thermometer Number ASTM	IP
High cloud and pour	−38 to +50°C	5C	1C
Low cloud and pour	−80 to +20°C	6C	2C
Melting point	+32 to +127°C	61C	63C

6.2.1 Since separation of liquid column thermometers occasionally occurs and may escape detection, thermometers should be checked immediately prior to the test and used only if they prove accurate within ±1°C (for example ice point).

6.3 *Cork*, to fit the test jar, bored centrally for the test thermometer.

6.4 *Jacket*, watertight, cylindrical, metal, flat-bottomed, 115 ± 3-mm depth, with inside diameter of 44.2 to 45.8 mm. It shall be supported in a vertical position in the cooling bath (see 6.7) so that not more than 25 mm projects out of the cooling medium, and shall be capable of being cleaned.

6.5 *Disk*, cork or felt, 6 mm thick to fit loosely inside the jacket.

6.6 *Gasket*, to fit snugly around the outside of the test jar and loosely inside the jacket. The gasket may be made of rubber, leather, or other material that is elastic enough to cling to the test jar and hard enough to hold its shape. Its purpose is to prevent the test jar from touching the jacket.

6.7 *Bath or Baths*, maintained at prescribed temperatures with a firm support to hold the jacket vertical. The required bath temperatures may be obtained by refrigeration if available, otherwise by suitable freezing mixtures. Freezing mixtures commonly used for temperatures down to those shown are as follows:

	For Temperatures Down
Ice and water	9°C
Crushed ice and sodium chloride crystals	−12°C
Crushed ice and calcium chloride crystals	−27°C
Acetone or petroleum naphtha (see Section 6) chilled in a covered metal beaker with an ice-salt mixture to −12°C then with enough solid carbon dioxide to give the desired temperature.	−57°C

7. Reagents and Materials

7.1 The following solvents of technical grade are appropriate for low-temperature bath media.

7.1.1 *Acetone*, (**Warning—**Extremely flammable).

7.1.2 *Alcohol, Ethanol* (**Warning**—Flammable).

7.1.3 *Alcohol, Methanol* (**Warning—**Flammable. Vapor harmful).

7.1.4 *Petroleum Naphtha*, (**Warning—**Combustible. Vapor harmful).

7.1.5 *Solid Carbon Dioxide*, (**Warning—**Extremely cold −78.5°C).

8. Procedure

8.1 Pour the specimen into the test jar to the level mark. When necessary, heat the specimen in a water bath until it is just sufficiently fluid to pour into the test jar.

NOTE 1—It is known that some materials, when heated to a temperature higher than 45°C during the preceding 24 h, do not yield the same pour point results as when they are kept at room temperature for 24 h prior to testing. Examples of materials which are known to show sensitivity to thermal history are residual fuels, black oils, and cylinder stocks.

8.1.1 Samples of residual fuels, black oils, and cylinder stocks which have been heated to a temperature higher than 45°C during the preceding 24 h, or when the thermal history of these sample types is not known, shall be kept at room temperature for 24 h before testing. Samples which are known by the operator not to be sensitive to thermal history need not be kept at room temperature for 24 h before testing.

8.1.2 Experimental evidence supporting elimination of the 24-h waiting period for some sample types is contained in a research report.[6]

8.2 Close the test jar with the cork carrying the high-pour thermometer (5.2). In the case of pour points above 36°C, use a higher range thermometer such as IP 3C or ASTM 61C. Adjust the position of the cork and thermometer so the cork fits tightly, the thermometer and the jar are coaxial, and the thermometer bulb is immersed so the beginning of the capillary is 3 mm below the surface of the specimen.

8.3 For the measurement of pour point, subject the specimen in the test jar to the following preliminary treatment:

8.3.1 *Specimens Having Pour Points Above −33°C*—Heat the specimen without stirring to 9°C above the expected pour point, but to at least 45°C, in a bath maintained at 12°C above the expected pour point, but at least 48°C. Transfer the test jar to a water bath maintained at 24°C and commence observations for pour point.

8.3.2 *Specimens Having Pour Points of −33°C and Below*—Heat the specimen without stirring to 45°C in a bath maintained at 48°C and cool to 15°C in a water bath maintained at 6°C. Remove the high cloud and pour thermometer, and place the low cloud and pour thermometer in position.

8.4 See that the disk, gasket, and the inside of the jacket are clean and dry. Place the disk in the bottom of the jacket. Place the gasket around the test jar, 25 mm from the bottom. Insert the test jar in the jacket. Never place a jar directly into the cooling medium.

8.5 After the specimen has cooled to allow the formation of paraffin wax crystals, take great care not to disturb the mass of specimen nor permit the thermometer to shift in the specimen; any disturbance of the spongy network of wax crystals will lead to low and erroneous results.

8.6 Pour points are expressed in integers that are positive or negative multiples of 3°C. Begin to examine the appearance of the specimen when the temperature of the specimen is 9°C above the expected pour point (estimated as a multiple of 3°C). At each test thermometer reading that is a multiple of 3°C below the starting temperature remove the test jar from the jacket. To remove condensed moisture that limits visibility wipe the surface with a clean cloth moistened in alcohol (ethanol or methanol). Tilt the jar just enough to ascertain whether there is a movement of the specimen in the test jar. The complete operation of removal, wiping, and replacement shall require not more than 3 s.

8.6.1 If the specimen has not ceased to flow when its temperature has reached 27°C, transfer the test jar to the next lower temperature bath in accordance with the following schedule:

Specimen is at +27°C, move to 0°C bath
Specimen is at +9°C, move to −18°C bath
Specimen is at −6°C, move to −33°C bath
Specimen is at −24°C, move to −51°C bath
Specimen is at −42°C, move to −69°C bath

8.6.2 As soon as the specimen in the jar does not flow when tilted, hold the jar in a horizontal position for 5 s, as noted by an accurate timing device and observe carefully. If the specimen shows any movement, replace the test jar immediately in the jacket and repeat a test for flow at the next temperature, 3°C lower.

8.7 Continue in this manner until a point is reached at which the specimen shows no movement when the test jar is held in a horizontal position for 5 s. Record the observed reading of the test thermometer.

8.8 For black specimen, cylinder stock, and nondistillate fuel specimen, the result obtained by the procedure described in 8.1 through 8.7 is the upper (maximum) pour point. If required, determine the lower (minimum) pour point by heating the sample while stirring, to 105°C, pouring it into the jar, and determining the pour point as described in 8.4 through 8.7.

8.9 Some specifications allow for a pass/fail test or have pour point limits at temperatures not divisible by 3°C. In these cases, it is acceptable practice to conduct the pour point measurement according to the following schedule: Begin to examine the appearance of the specimen when the temperature of the specimen is 9°C above the specification pour point. Continue observations at 3°C intervals as described in 8.6 and 8.7 until the specification temperature is reached. Report the sample as passing or failing the specification limit.

9. Calculation and Report

9.1 Add 3°C to the temperature recorded in 8.7 and report the result as the Pour Point, ASTM D 97. For black oil, and so forth, add 3°C to the temperature recorded in 8.7 and report the result as Upper Pour Point, ASTM D 97, or Lower Pour Point, ASTM D 97, as required.

10. Precision and Bias

10.1 *Lubricating Oil and Distillate and Residual Fuel Oil.* [7]

10.1.1 *Repeatability*—The difference between successive test results, obtained by the same operator using the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1377.

[7] The cloud point procedure formerly part of this test method now appears as Test Method D 2500.

operation of this test method, exceed 3°C only in one case in twenty. Differences greater than this should be considered suspect.

10.1.2 *Reproducibility*—The difference between two single and independent test 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 this test method, exceed 6°C only in one case in twenty. Differences greater than this should be considered suspect.

10.2 *Bias*—There being no criteria for measuring bias in these test-product combinations, no statement of bias can be made.

10.3 The precision statements were prepared with data on ten new (unused) mineral oil-based lubricants and sixteen assorted fuel oils tested by twelve cooperators. The mineral oil-based lubricants had pour points ranging from −48 to −6°C while the fuel oils had pour points ranging from −33 to +51°C. The following precision data were obtained:

	Mineral Oil Lubricants	Fuel Oils
95 % Confidence		
Repeatability, °C	2.87	2.52
Reproducibility, °C	6.43	6.59

APPENDIX

(Nonmandatory Information)

X1. TEST FOR FLUIDITY OF A RESIDUAL FUEL OIL AT A SPECIFIED TEMPERATURE

X1.1 General

X1.1.1 The low-temperature flow properties of a waxy fuel oil depend on handling and storage conditions. Thus, they may not be truly indicated by pour point. The pour point test does not indicate what happens when an oil has a considerable head of pressure behind it, such as when gravitating from a storage tank or being pumped along a pipeline. Failure to flow at the pour point is normally attributed to the separation of wax from the fuel; however, it can also be due to the effect of viscosity in the case of very viscous fuel oils. In addition pour points of residual fuels are influenced by the previous thermal history of the specimens. A loosely knit wax structure built up on cooling of the oil can be normally broken by the application of relatively little pressure.

X1.1.2 The usefulness of the pour point test in relation to residual fuel oils is open to question, and the tendency to regard the pour point as the limiting temperature at which a fuel will flow can be misleading. The problem of accurately specifying the handling behavior of fuel oil is important, and because of the technical limitations of the pour point test, various pumpability tests have been devised to assess the low-temperature flow characteristics of heavy residual fuel oils. Test Method D 3245 is one such method. However, most alternative methods tend to be time-consuming and as such do not find ready acceptance as routine control tests for determining low-temperature flow properties. One method which is relatively quick and easy to perform and has found limited acceptance as a "go-no-go" method is based on the appendix method to the former Test Method D 1659–65. The method is described as follows.

X1.2 Scope

X1.2.1 This method covers the determination of the fluidity of a residual fuel oil at a specified temperature in an as-received condition.

X1.3 Definition

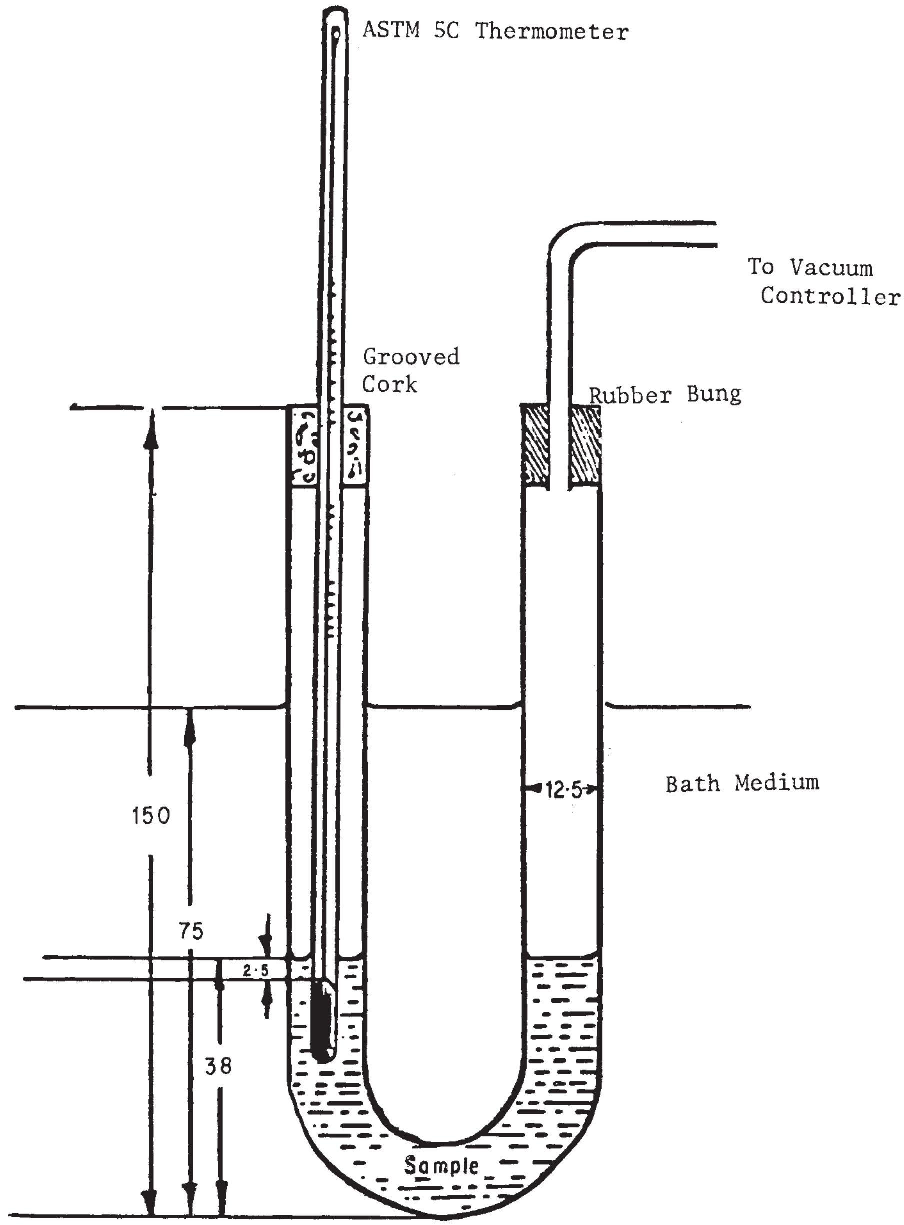
X1.3.1 *fluidity temperature*—the sample when tested in an as-received condition is considered "fluid at the temperature of the test" if it will flow 2 mm in 1 min in a 12.5 mm U-tube under a maximum pressure of 152 mm of mercury.

X1.4 Summary of Test Method

X1.4.1 A sample of fuel in its as-received condition is cooled at the specified temperature for 30 min in the standard U-tube and is tested for movement under prescribed pressure conditions.

X1.5 Significance and Use

X1.5.1 This method may be used as a "go-no-go" procedure for operational situations where it is necessary to ascertain the fluidity of a residual oil under prescribed conditions in an as-received condition. The conditions of this method simulate those of a pumping situation where the oil is expected to flow through a 12-mm pipe under slight pressure at a specified temperature. Fluidity, like Test Method D 97, is used to define cold flow properties. It differs from D 97, however, in that (*1*) it is restricted to residual fuel oil and (*2*) a prescribed pressure is applied to the sample. The latter represents an attempt to overcome the technical limitations of the Pour Point Method where gravity-induced flow is the criterion. Test Method D 3245, represents another method for predicting field performance in cold flow conditions. Test Method D 3245, however, does have limitations and may not be suitable for use with very waxy fuel oils which solidify so rapidly in the chilling bath that a reading cannot be obtained under the conditions of the test. It is also a time-consuming test and therefore not suitable for routine control testing.



NOTE—All dimensions are in millimetres

FIG. X1.1 Disposition of U-tube in Fluidity Temperature Test Bath

X1.6 Apparatus

X1.6.1 *Glass U-Tubes*, 150 mm high, having a uniform internal diameter of 12.5 ± 1 mm and a radius of curvature, measured to the outside curve of the tube of 35 mm (Fig. X1.1).

X1.6.2 *Thermometers*—Thermometers having a range from −38 to +50°C and conforming to the requirements of Thermometer 5C as prescribed in Specification E 1, shall be used for insertion in the glass U-tubes and for measuring the temperatures of the baths.

X1.6.3 *Fluidity Temperature Test Bath*,[8]

consists of a reservoir, a stirrer, and a motor and pump to circulate coolant through the coils of the tubing placed in the bottom of the test bath and passing through the cold bath. The flow of coolant through these coils can be controlled by a thermostat and a solenoid valve. It is possible that, where justified by the quantity of work, more than one such bath

[8] A kinematic viscosity bath is usually satisfactory.

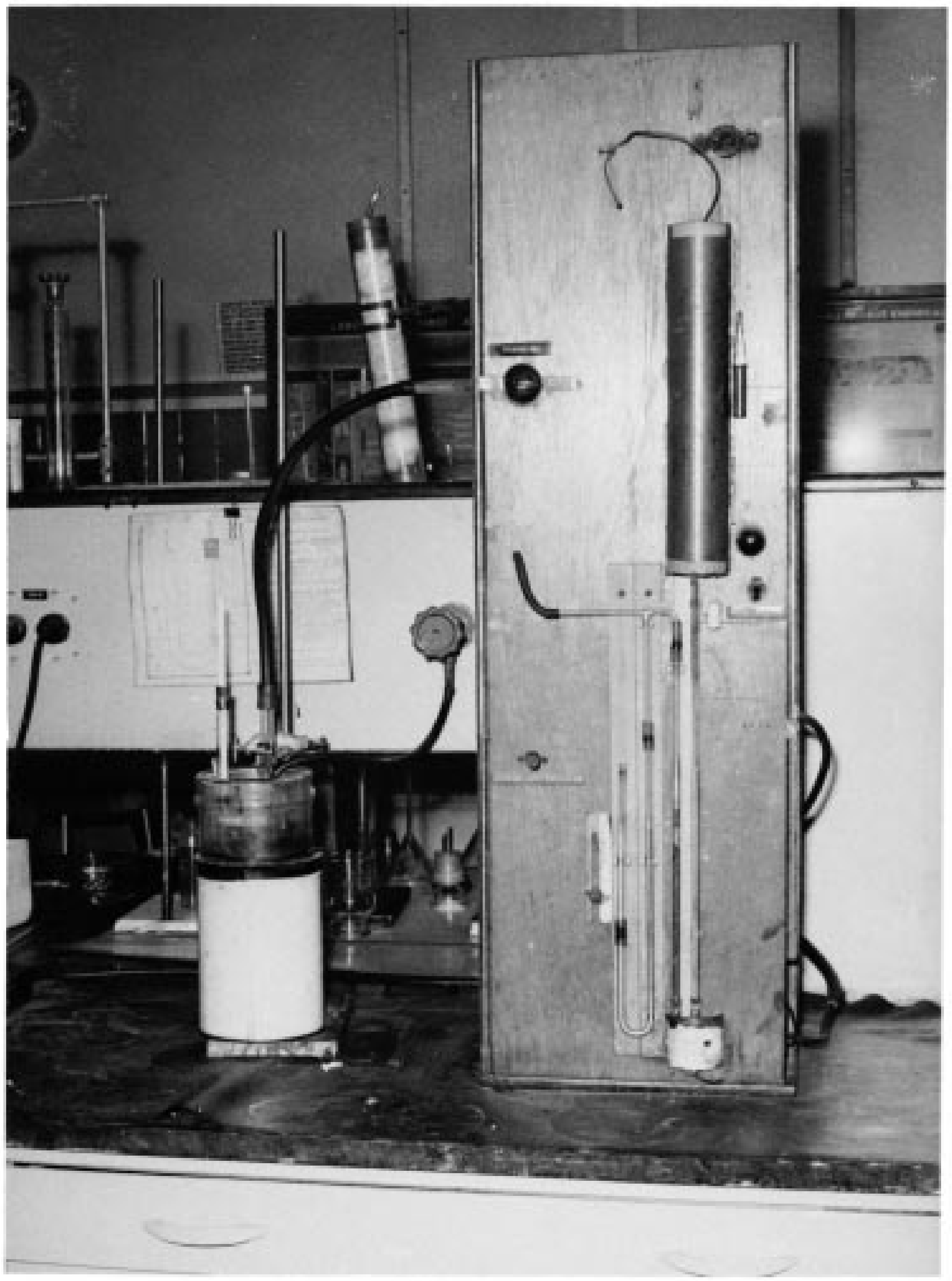


FIG. X1.2 Fluidity Temperature Apparatus

could be utilized to permit concurrent testing at more than one temperature (Fig. X1.2).

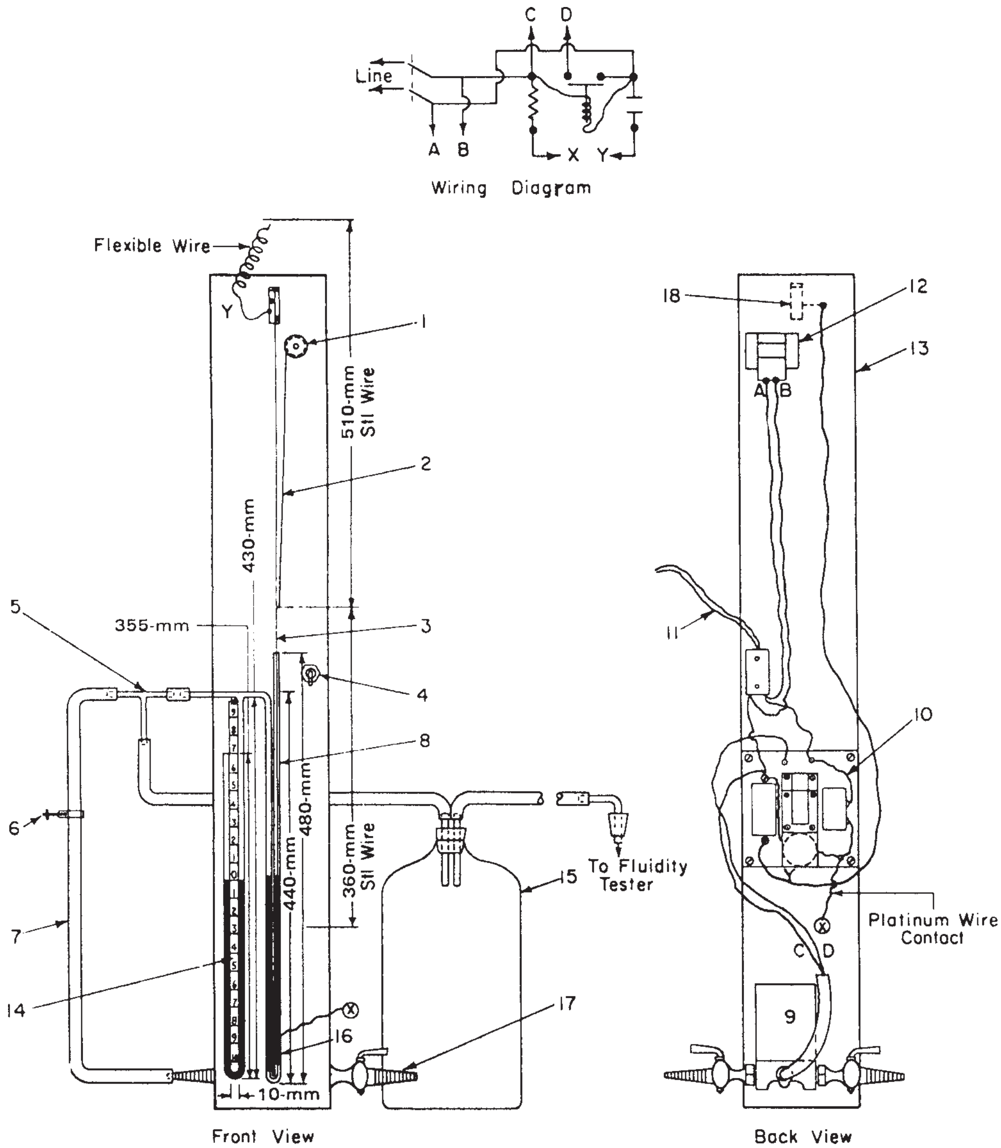
X1.6.4 *Mercury Manometer* calibrated in 10-mm divisions with a distinguishing mark at 152 mm (equivalent to 20.3 kPa).

X1.6.5 *Automatic Vacuum Controller*[9] *(as shown in Fig. X1.3 and Fig. X1.4)*—A device that gradually increased the vacuum applied to one end of the U-tube at the specified rate of 10 mm/4S.

[9] This apparatus may be shop fabricated. Details of special parts are indicated in Figs. X1.3 and X1.4. Alternatively the apparatus can be purchased.

X1.7 Preparation of Apparatus

X1.7.1 Adjust the automatic vacuum controller as follows: close the stopcock on the tube connecting the automatic vacuum controller to the fluidity tester. A pinchcock on the rubber tube will serve as well as a stopcock. Wind the thread attached to the steel rod around the pulley on the synchronous motor until the end of the rod is about 15 mm above the zero level of the mercury in the control manometer. Turn on the power switch. The thread will begin to unwind, lowering the steel rod. When the rod contacts the mercury, the relay will open the solenoid valve in the vacuum line and air will be



1—26-mm diameter face pulley
2—Thread
3—Steel rod
4—Switch-DPST
5—Tee, 90-mm long
6—Needle valve
7—Rubber or plastic tubing
8—6-mm heat-resistant glass tube
9—Solenoid valve
10—Electric relay
11—Electric cord to outlet
12—Synchronous motor
13—Plywood of approximately 10-mm thickness
14—Millimeter scale
15—4-L bottle
16—0.5-mm heat-resistant glass capillary
17—To vacuum line
18—Rod holder

FIG. X1.3 Assembly Automatic Vacuum Controller Apparatus

pumped from the system at a rate limited by the needle valve. Adjust this needle valve until the descending mercury in the control manometer just leads the rod, reducing the relay operation to a minimum. When properly adjusted, the pulsations caused by the opening and closing of the solenoid valve should not exceed ±1 mm. In this manner the pressure in the system will be reduced gradually at a rate governed by the descent of the steel rod.

X1.8 Procedure

X1.8.1 Pour the sample as received into a thoroughly cleaned and dry standard fluidity U-tube, without contacting the upper walls of the tube, until the vertical height of the sample in the U-tube is 38 mm. Insert in one leg of each U-tube an ASTM Thermometer 5C in a cork that has been grooved to permit the passage of air. The thermometer must be placed in

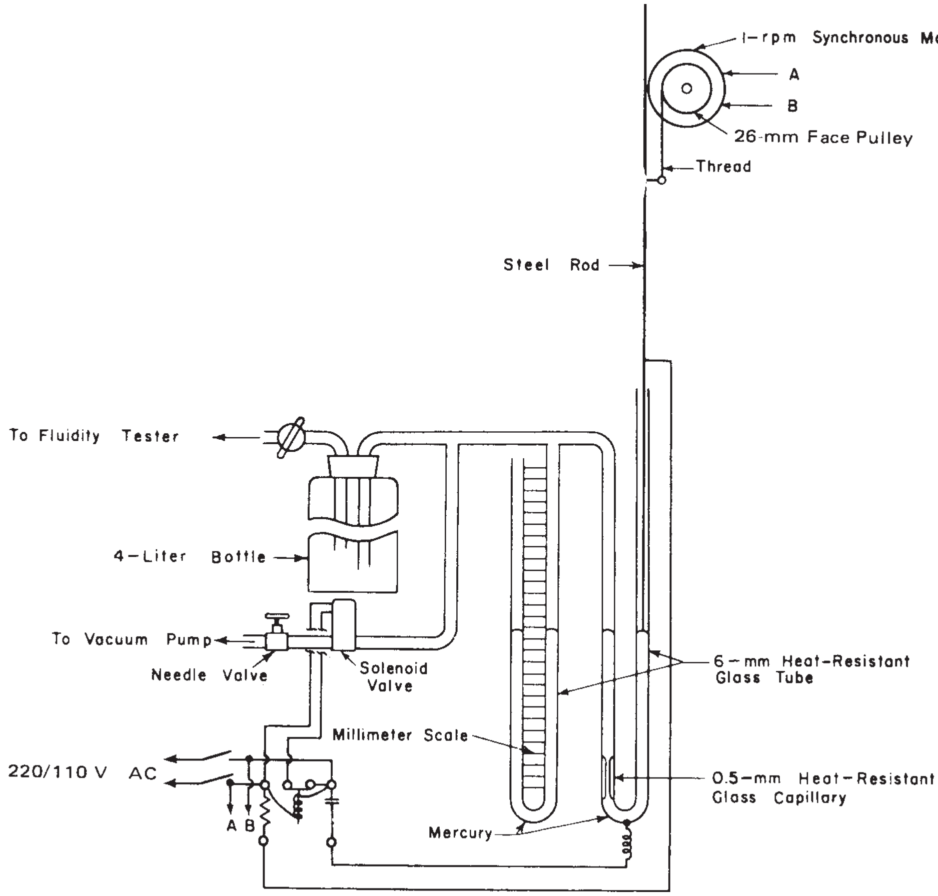


FIG. X1.4 Detail of Automatic Vacuum Controller

the center of the tube and its bulb immersed so that the beginning of the capillary is 3 mm below the surface of the specimen.

X1.8.2 Fix the tube in the bath set at the specific temperature, immersed to a depth of approximately 75 mm. Control the bath and sample temperatures within ±1°C and ±0.5°C, respectively, of the specified temperature of the test.

X1.8.3 Maintain the sample at the specified temperature for 30 min ± 30 s, with the U-tube connected to the automatic vacuum controller, and the stopcock or pinch-clamp open. Wind the thread on the pulley attached to the synchronous motor. Turn the power switch to the ON position. Apply suction automatically to the U-tube at the prescribed rate. Observe any movement of the specimen during a one-minute interval which is the time required to apply 152-mm Hg vacuum to the specimen in the U-tube. Immediately disconnect the U-tube from the automatic vacuum controller, turn off the power switch and rewind the thread. If the specimen has moved 2 mm or more during the time (1 min) the suction was applied, the specimen is considered fluid at the temperature of the test.

X1.9 Report

X1.9.1 Report the fluidity of the sample at a specified temperature as follows:

X1.9.1.1 If the sample fulfills the conditions of flow, as defined in X1.3.1, report fluidity: "Fluid at (temperature of test)" or fluidity at (temperature of test): "Pass."

X1.9.1.2 If the sample does not fulfill the conditions of flow, as defined in X1.3.1, report fluidity: "Not fluid at (temperature of test)" or fluidity at (temperature of test): "Fail."

X1.10 Precision and Bias

X1.10.1 As in the case of pass-fail data, no statement is made about either the precision or the bias of this method for measuring the fluidity of a residual fuel specimen since the result merely states whether there is conformance to the criteria for success specified in the procedure.

Harris Testing Laboratories, Inc.

CERTIFICATE OF ANALYSIS

CLIENT:
Megatrol Inc
9469 S 500 W
Sandy, Ut 84070

PRODUCT: **FE**^{26} Premier
MARKS: NONE
DATE RECEIVED: 02/11/2008
LAB NO: HH0410-1202-P
SUBMITTED BY: Jon Rose

			SPECIFICATIONS	
METHOD	**TEST**	**RESULT**	**MIN.**	**MAX.**
D-130	**Copper Corrosion**	**1 b**	**XXX**	**XXX**
		XXX	**XXX**	**XXX**
	Copper Corrosion 10% solution	**XXX**	**XXX**	**XXX**
	"measured in PPM"	**0.7**	**0**	**30**

Comments:

Amos Mwangi

Date issued:
02/11/2008

CHEMIST

Standard Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test[1]

This standard is issued under the fixed designation D 130; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of the corrosiveness to copper of aviation gasoline, aviation turbine fuel, automotive gasoline, cleaners (Stoddard) solvent, kerosine, diesel fuel, distillate fuel oil, lubricating oil, and natural gasoline or other hydrocarbons having a vapor pressure no greater than 124 kPa (18 psi) at 37.8°C. (**Warning**—Some products, particularly natural gasoline, may have a much higher vapor pressure than would normally be characteristic of automotive or aviation gasolines. For this reason, exercise extreme caution to ensure that the pressure vessel used in this test method and containing natural gasoline or other products of high vapor pressure is not placed in the 100°C (212°F) bath. Samples having vapor pressures in excess of 124 kPa (18 psi) may develop sufficient pressures at 100°C to rupture the pressure vessel. For any sample having a vapor pressure above 124 kPa (18 psi), use Test Method D 1838.)

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

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory requirements prior to use.* For specific warning statements, see 1.1, 6.1, and Annex A2.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 396 Specification for Fuel Oils
D 975 Specification for Diesel Fuel Oils
D 1655 Specification for Aviation Turbine Fuels
D 1838 Test Method for Copper Strip Corrosion by Liquefied Petroleum (LP) Gases
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
E 1 Specification for ASTM Liquid-in-Glass Thermometers

2.2 *ASTM Adjuncts:*

ASTM Copper Strip Corrosion Standard[3]

3. Summary of Test Method

3.1 A polished copper strip is immersed in a specific volume of the sample being tested and heated under conditions of temperature and time that are specific to the class of material being tested. At the end of the heating period, the copper strip is removed, washed and the color and tarnish level assessed against the ASTM Copper Strip Corrosion Standard.

4. Significance and Use

4.1 Crude petroleum contains sulfur compounds, most of which are removed during refining. However, of the sulfur compounds remaining in the petroleum product, some can have a corroding action on various metals and this corrosivity is not

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke and Carbon Material.

Current edition approved May 1, 2004. Published June 2004. Originally approved in 1922, replacing former D 89. Last previous edition approved in 2000 as D 130–94 (2000)$^{\epsilon1}$.

In the IP, this test method is under the jurisdiction of the Standardization Committee. It is issued under the fixed designation IP 154. The final number indicates the year of last revision.

This test method has been approved by the sponsoring committees and accepted by the cooperating societies in accordance with established procedures.

[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 ASTM International Headquarters. Order Adjunct No. ADJD0130. Names of suppliers in the United Kingdom can be obtained from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K. Two master standards are held by the IP for reference.

necessarily related directly to the total sulfur content. The effect can vary according to the chemical types of sulfur compounds present. The copper strip corrosion test is designed to assess the relative degree of corrosivity of a petroleum product.

5. Apparatus

5.1 *Copper Strip Corrosion Pressure Vessel*, constructed from stainless steel according to the dimensions as given in Fig. 1. The vessel shall be capable of withstanding a test pressure of 700 kPa gage (100 psi). Alternative designs for the vessel's cap and synthetic rubber gasket may be used provided that the internal dimensions of the vessel are the same as those shown in Fig. 1. The internal dimensions of the pressure vessel are such that a nominal 25-mm by 150-mm test tube can be placed inside the pressure vessel.

5.2 *Test Tubes*, of borosilicate glass of nominal 25-mm by 150-mm dimensions. The internal dimensions shall be checked as acceptable by use of a copper strip (see 6.3). When 30 mL of liquid is added to the test tube with the copper strip in it, a minimum of 5-mm of liquid shall be above the top surface of the strip.

5.3 *Test Baths*:

5.3.1 *General*—All test baths shall be able to maintain the test temperature to within ±1°C (2°F) of the required test temperature.

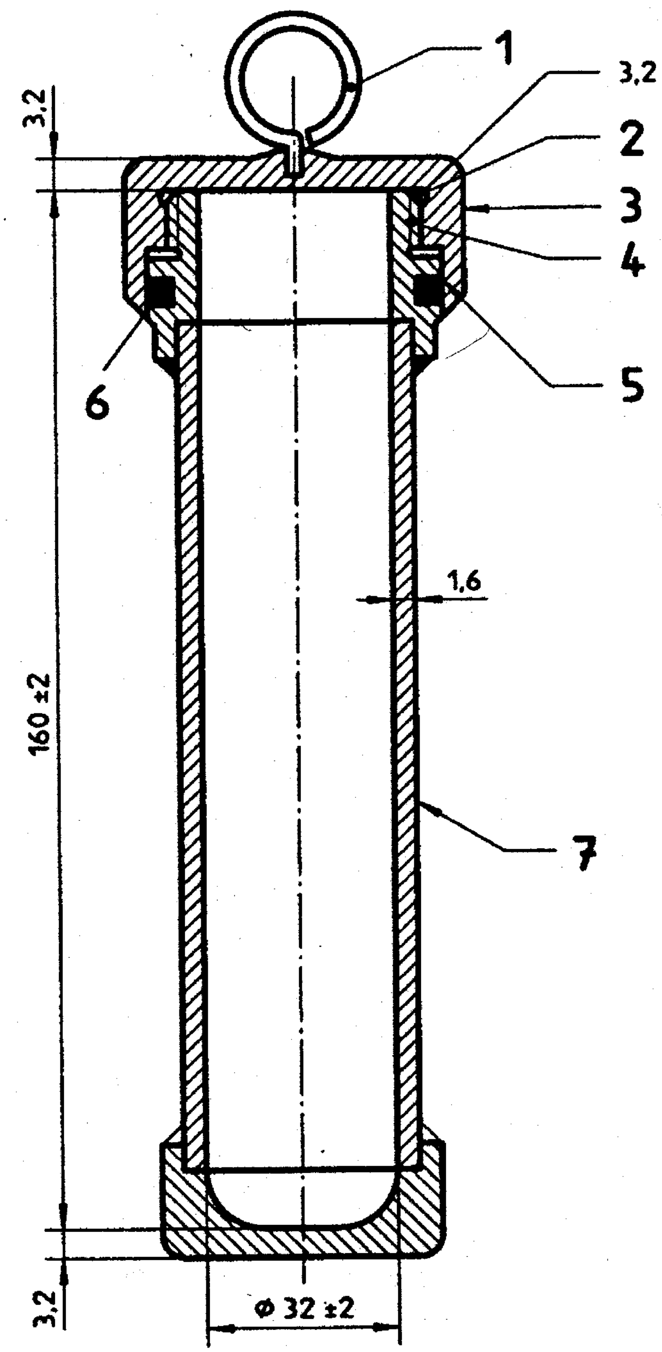
5.3.2 *Liquid Bath Used for Submerging Pressure Vessel(s)*—The bath shall be deep enough to submerge one or more pressure vessels (see 5.1) completely during the test. As the bath medium, use water or any liquid that can be satisfactorily controlled to the sample test temperature. The bath shall be fitted with suitable supports to hold each pressure vessel in a vertical position when submerged.

5.3.3 *Bath(s) Used for Test Tubes*—Liquid baths shall be fitted with suitable supports to hold each test tube (see 5.2) in a vertical position to a depth of about 100-mm (4-in.) as measured from the bottom of the test tube to the bath surface. As a liquid bath medium, water and oil have been found satisfactory and controllable at the specified test temperature. Solid block baths shall meet the same temperature control and immersion conditions and shall be checked for temperature measurement (heat transfer) for each product class by running tests on tubes filled with 30 mL of product plus a metal strip of the nominal dimensions given, plus a temperature sensor.

5.4 *Temperature Sensing Device (TSD)*, capable of monitoring the desired test temperature in the bath to within an accuracy of ±1°C or better. The ASTM 12C (12F) (see Specification E 1) or IP 64C (64F) total immersion thermometers have been found suitable to use in the test. If used, no more than 10-mm (0.4-in.) of the mercury should extend above the surface of the bath at the test temperature.

5.5 *Polishing Vise*, for holding the copper strip firmly without marring the edges while polishing. Any convenient type of holder (see Appendix X1) may be used provided that the strip is held tightly and that the surface of the strip being polished is supported above the surface of the holder.

5.6 *Viewing Test Tubes*, flat glass test tubes, are convenient for protecting corroded copper strips for close inspection or storage (see Appendix X1 for the description of a flat-glass



Key:
1 Lifting eye
2 Wide groove for pressure relief
3 Knurled cap
4 Twelve threads per inch NF thread or equivalent
5 Camber inside cap to protect "O" ring when closing pressure vessel
6 Synthetic rubber "O" ring without free sulfur
7 Seamless tube

Material: stainless steel
Welded construction
Maximum test gage pressure: 700 kPa

NOTE 1—Dimensions in millimetres.
NOTE 2—All dimensions without tolerance limits are nominal values.

FIG. 1 Pressure Vessel for Copper Strip Corrosion Test

viewing tube). The viewing test tube shall be of such dimensions as to allow the introduction of a copper strip (see 6.3) and made of glass free of striae or similar defects.

5.7 *Forceps*, with either stainless steel or polytetrafluoroethylene (PTFE) tips, for use in handling the copper strips, have been found suitable to use.

5.8 *Timing Device*, electronic or manual, capable of accurately measuring the test duration within the allowable tolerance.

6. Reagents and Materials

6.1 *Wash Solvent*—Any volatile, less than 5 mg/kg sulfur hydrocarbon solvent may be used provided that it shows no tarnish at all when tested for 3 h at 50°C (122°F). 2,2,4-trimethylpentane (isooctane) of minimum 99.75 % purity is the referee solvent and should be used in case of dispute. (**Warning**—extremely flammable, see A2.1.)

6.2 *Surface Preparation/Polishing Materials*, 00 grade or finer steel wool or silicon carbide grit paper or cloth of varying degrees of fineness including 65-µm (240-grit) grade; also a supply of 105-µm (150-mesh) size silicon carbide grain or powder and absorbent cotton (cotton wool). A commercial grade is suitable, but pharmaceutical grade is most commonly available and is acceptable.

6.3 *Copper Strips Specification*—Use strips approximately 12.5-mm (½-in.) wide, 1.5 to 3.0-mm (1⁄16 to ⅛-in.) thick, cut approximately 75-mm (3-in.) long from smooth-surfaced, hard-temper, cold-finished copper of 99.9 + % purity; electrical bus bar stock is generally suitable (see Annex A1). The strips may be used repeatedly but should be discarded when the strip's surface shows pitting or deep scratches that cannot be removed by the specified polishing procedure, or when the surface becomes deformed.

6.4 *Ashless Filter Paper or Disposable Gloves*, for use in protecting the copper strip from coming in contact with the individual during final polishing.

7. ASTM Copper Strip Corrosion Standards[3]

7.1 These consist of reproductions in color of typical test strips representing increasing degrees of tarnish and corrosion, the reproductions being encased for protection in plastic and made up in the form of a plaque.

7.1.1 Keep the plastic-encased ASTM Copper Strip Corrosion Standards protected from light to avoid the possibility of fading. Inspect for fading by comparing two different plaques, one of which has been carefully protected from light (for example, new plaque). Observe both sets in diffused daylight (or equivalent) first from a point directly above and then from an angle of 45°. If any evidence of fading is observed, particularly at the left-hand end of the plaque, it is suggested that the one that is the more faded with respect to the other be discarded.

7.1.1.1 Alternatively, place a suitably sized opaque strip (for example, 20-mm (¾-in.) black electrical tape) across the top of the colored portion of the plaque when initially purchased. At intervals remove the opaque strip and observe. When there is any evidence of fading of the exposed portion, the standards shall be replaced.

7.1.1.2 These plaques are full-color reproductions of typical strips. They have been printed on aluminum sheets by a 4-color process and are encased in plastic for protection. Directions for their use are given on the reverse side of each plaque.

7.1.2 If the surface of the plastic cover shows excessive scratching, it is suggested that the plaque be replaced.

8. Samples

8.1 In accordance with D 4057 or D 4177, or both, it is particularly important that all types of fuel samples, that pass a low-tarnish strip classification, be collected in clean, dark glass bottles, plastic bottles, or other suitable containers that will not affect the corrosive properties of the fuel. Avoid the use of tin plate containers for collection of samples, since experience has shown that they may contribute to the corrosiveness of the sample.

8.2 Fill the containers as completely as possible and close them immediately after taking the sample. Adequate headspace in the container is necessary to provide room for possible thermal expansion during transport. It is recommended that volatile samples be filled between 70 and 80 % of the container's capacity. Take care during sampling to protect the samples from exposure to direct sunlight or even diffused daylight. Carry out the test as soon as possible after receipt in the laboratory and immediately after opening the container.

8.3 If suspended water (that is, haze) is observed in the sample, dry by filtering a sufficient volume of sample through a medium rapid qualitative filter, into the prescribed clean, dry test tube. Carry out this operation in a darkened room or under a light-protected shield.

8.3.1 Contact of the copper strip with water before, during or after completion of the test run will cause staining, making it difficult to evaluate the strips.

9. Preparation of Test Strips

9.1 *Surface Preparation*—Remove all surface blemishes from all six sides of the strip obtained from a previous analysis (see Note 1). One way to accomplish this is to use 00 grade or finer steel wool or silicon carbide paper or cloth of such degrees of fineness as are needed to accomplish the desired results efficiently. Finish with 65-µm (240-grit) silicon carbide paper or cloth, removing all marks that may have been made by other grades of paper used previously. Ensure the prepared copper strip is protected from oxidation prior to final preparation, such as by immersing the strip in wash solvent from which it can be withdrawn immediately for final preparation (polishing) or in which it can be stored for future use.

NOTE 1—Only final preparation (9.2) is necessary for commercially purchased pre-polished strips.

9.1.1 As a practical manual procedure for surface preparation, place a sheet of silicon carbide paper or cloth on a flat surface and moisten it with kerosine or wash solvent. Rub the strip against the silicon carbide paper or cloth with a circular motion, protecting the strip from contact with the fingers by using ashless filter paper or wearing disposable gloves. Alternatively, the surface of the strip can be prepared by use of motor-driven machines using appropriate grades of dry paper or cloth.

9.2 *Final Preparation*—For strips prepared in 9.1 or new strips being used for the first time, remove a strip from its protected location, such as by removing it from the wash solvent. To prevent possible surface contamination during final preparation, do not allow fingers to come in direct contact with the copper strips, such as by wearing disposable gloves or holding the strips in the fingers protected with ashless filter paper. Polish first the ends and then the sides with the 105-mm (150-mesh) silicon carbide grains picked up with a pad of cotton (cotton wool) moistened with wash solvent. Wipe vigorously with fresh pads of cotton (cotton wool) and subsequently handle without touching the surface of the strip with the fingers. Forceps have been found suitable to use. Clamp in a vise and polish the main surfaces with silicon-carbide grains on absorbent cotton. Do not polish in a circular motion. Rub in the direction of the long axis of the strip, carrying the stroke beyond the end of the strip before reversing the direction. Clean all metal dust from the strip by rubbing vigorously with clean pads of absorbent cotton until a fresh pad remains unsoiled. When the strip is clean, immediately immerse it in the prepared sample.

9.2.1 It is important to polish the whole surface of the strip uniformly to obtain a uniformly stained strip. If the edges show wear (surface elliptical), they will likely show more corrosion than the center. The use of a vise (see Appendix X1) will facilitate uniform polishing.

9.2.2 It is important to follow the order of preparation with the correctly sized silicon carbide material as described in 9.1 and 9.2. The final preparation is with 105-µm silicon carbide powder. This is a larger grain size than the 65-µm paper used in the surface preparation stage. The reason for this use of larger silicon carbide grains in the final preparation is to produce asperities (controlled roughness) on the surface of the copper, which act as sites for the initiation of corrosion reactions.

10. Procedure

10.1 *General*—There are a variety of test conditions, which are broadly specific to given classes of product but, within certain classes, more than one set of test conditions of time or temperature, or both, may apply. In general, aviation gasoline shall be tested in a pressure vessel at 100°C and other high vapor pressure fuels, like natural gasoline, at 40°C. Other liquid products shall be tested in a test tube at 50°C, 100°C or even higher temperatures. The conditions of time and temperature given below are commonly used and are quoted in the ASTM specifications for these products where such specifications exist. They are, however, guides only. Other conditions can also be used when required by specifications or by agreement between parties. The test conditions of time and temperature shall be recorded as part of the result (see 12.1).

10.2 *Pressure Vessel Procedure*—For use with aviation gasoline and higher vapor pressure samples.

10.2.1 *For Aviation Gasoline and Aviation Turbine Fuel*—Place 30 mL of sample, completely clear and free of any suspended or entrained water (see 8.3) into a chemically clean and dry 25-mm by 150-mm test tube. Within 1 min after completing the final preparation (polishing), slide the copper strip into the sample tube. Place the sample tube into the pressure vessel (Fig. 1) and screw the lid on tightly. If more than one sample is to be analyzed at essentially the same time, it is permissible to prepare each pressure vessel in the batch before completely immersing each pressure vessel in the liquid bath at 100 ± 1°C (212 ± 2°F), provided the elapsed time between the first and last samples is kept to a minimum. After 2 h ± 5 min in the bath, withdraw the pressure vessel and immerse for a few minutes in cool water (tap water). Open the pressure vessel, withdraw the test tube and examine the strip as described in 10.4.

10.2.2 *For Natural Gasoline*—Carry out the test exactly as described in 10.2.1 but at 40°C (104°F) and for 3 h ± 5 min.

10.3 *Test Tube Procedure*—For use with most liquid products.

10.3.1 *For Diesel Fuel, Fuel Oil, Automotive Gasoline*—Place 30 mL of sample, completely clear and free of any suspended or entrained water (see 8.3), into a chemically clean, dry 25-mm by 150-mm test tube and, within 1 min after completing the final preparation (polishing), slide the copper strip into the sample tube. If more than one sample is to be analyzed at essentially the same time, it is permissible to prepare each sample in the batch by stoppering each tube with a vented stopper, such as a vented cork before placing each tube in a bath maintained at 50 ± 1°C (122 ± 2°F), provided the elapsed time between the first and last sample prepared is kept to a minimum. Protect the contents of the test tube from strong light during the test. After 3 h ± 5 min in the bath, examine the strip as described in 10.4. For tests on fuel oil and diesel fuel, to specifications other than Specifications D 396 and D 975, a temperature of 100°C (212°F) for 3 h is often used as an alternative set of conditions. Some automotive gasolines with vapor pressure above 80 kPa at 37.8°C have exhibited evaporation losses in excess of 10 % of their volume. If such evaporation losses are apparent, it is recommended that the Pressure Vessel Procedure (see 10.2) be used.

10.3.2 *For Cleaners (Stoddard) Solvent and Kerosine*—Carry out the test exactly as described in 10.3.1 but at 100 ± 1°C (212 ± 2°F).

10.3.3 *For Lubricating Oil*—Carry out the test exactly as described in 10.3.1, but the tests can be carried out for varying times and at elevated temperatures other than 100°C (212°F). For the sake of uniformity, it is suggested that even increments of 5°C, beginning with 150°C, be used.

10.4 *Strip Examination*:

10.4.1 Empty the contents of the test tube into a suitably sized receiver. If a receiver made out of glass is used, such as a 150-mL tall-form beaker, let the strip slide in gently so as to avoid breaking the glass. Immediately withdraw the strip with forceps and immerse in wash solvent. Withdraw the strip at once, dry and inspect for evidence of tarnishing or corrosion by comparison with the Copper Strip Corrosion Standards. The step of drying the strip may be done by blotting with filter paper, air drying, or by other suitable means. Hold both the test strip and the standard strip plaque in such a manner that light reflected from them at an angle of approximately 45° will be observed.

10.4.2 In handling the test strip during the inspection and comparison, the danger of marking or staining can be avoided if it is inserted in a flat glass tube (see Appendix X1), which can be stoppered with absorbent cotton.

11. Interpretation of Results

11.1 Interpret the corrosiveness of the sample in accordance with one of the classifications of the ASTM Copper Strip Corrosion Standard as listed in Table 1.

11.1.1 When a strip is in the obvious transition state between that indicated by any two adjacent standard strips, rate the sample at the more tarnished classification. Should a strip appear to have a darker orange color than Standard Strip 1b, consider the observed strip as still belonging in Classification 1; however, if any evidence of red color is observed, the observed strip belongs in Classification 2.

11.1.2 A claret red strip in Classification 2 can be mistaken for a magenta overcast on a brassy strip in Classification 3 if the brassy underlay of the latter is completely masked by a magenta overtone. To distinguish, immerse the strip in wash solvent; the former will appear as a dark orange strip while the latter will not change.

11.1.3 To distinguish multi-colored strips in Classifications 2 and 3, place a test strip in a 25-mm by 150-mm test tube and bring to a temperature of 340 ± 30°C in 4 to 6 min with the tube lying on a hot plate. Adjust to temperature by observing a high distillation thermometer inserted into a second test tube. If the strip belongs to Classification 2, it will assume the color of a silver and then a gold strip. If in Classification 3, it will take on the appearance of a transparent black, etc., as described in Classification 4a.

11.1.4 Repeat the test if blemishes due to fingerprints are observed, or due to spots from any particles or water droplets that may have touched the test strip during the digestion period.

11.1.5 Repeat the test also if the sharp edges along the flat faces of the strip appear to be in a classification higher than the greater portion of the strip; in this case, it is likely that the edges were burnished during preparation (polishing).

12. Report

12.1 Report the corrosiveness in accordance with one of the classifications listed in Table 1. State the duration of the test and the test temperature in the following format:

Corrosion copper strip (*X*h / *Y*°C), Classification *Zp*

where:
X = test duration, in hours,
Y = test temperature, °C,
Z = classification category (that is, 1, 2, 3, or 4), and
p = classification description for the corresponding *Z* (for example, a, b).

13. Precision and Bias

13.1 In the case of pass/fail data, no generally accepted method for determining precision or bias is currently available.

14. Keywords

14.1 automotive gasoline; aviation gasoline; aviation turbine fuel; copper corrosion; copper strip; corrosiveness to copper; natural gasoline

TABLE 1 Copper Strip Classifications

Classification	Designation	Description[A]
Freshly polished strip	...	[B]
1	slight tarnish	a. Light orange, almost the same as freshly polished strip
		b. Dark orange
2	moderate tarnish	a. Claret red
	...	b. Lavender
	...	c. Multicolored with lavender blue or silver, or both, overlaid on claret red
	...	d. Silvery
	...	e. Brassy or gold
3	dark tarnish	a. Magenta overcast on brassy strip
	...	b. Multicolored with red and green showing (peacock), but no gray
4	corrosion	a. Transparent black, dark gray or brown with peacock green barely showing
	...	b. Graphite or lusterless black
	...	c. Glossy or jet black

[A] The ASTM Copper Strip Corrosion Standard is a colored reproduction of strips characteristic of these descriptions.
[B] The freshly polished strip is included in the series only as an indication of the appearance of a properly polished strip before a test run; it is not possible to duplicate this appearance after a test even with a completely noncorrosive sample.

ANNEXES

(Mandatory Information)

A1. COPPER QUALITY

A1.1 Copper Quality

A1.1.1 Hard-temper, cold-finished type-(ETP) electrolytic tough pitch copper.[4]

[4] Conforming to Copper Development Association (CDA), United States of America No. 110, or to British Standard (BS) EN 1652 or BS 4608, which have proper quality.

A2. WARNING STATEMENTS

A2.1 Isooctane

Harmful if inhaled. Vapors may cause flash fire.
Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid build-up of vapors and eliminate all sources of ignition, especially nonexplosion-proof electrical apparatus and heaters.
Avoid prolonged breathing of vapor or spray mist.
Avoid prolonged or repeated skin contact.

A2.2 Aviation Turbine Fuel (Jet A or A-1, see Specification D 1655)

Keep away from heat, sparks, and open flames.
Keep container closed.
Use with adequate ventilation.
Avoid breathing vapor or spray mist.
Avoid prolonged or repeated contact with skin.

A2.3 Gasoline (Containing Lead)

Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid build-up of vapors and eliminate all sources of ignition, especially nonexplosion-proof electrical apparatus and heaters.
Avoid prolonged breathing of vapor or spray mist.
Avoid prolonged or repeated skin contact.

A2.4 Gasoline (White or Unleaded)

Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid build-up of vapors and eliminate all sources of ignition, especially nonexplosion-proof electrical apparatus and heaters.
Avoid prolonged breathing of vapor or spray mist.
Avoid prolonged or repeated skin contact.

A2.5 Kerosine

Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid breathing vapor or spray mist.
Avoid prolonged or repeated contact with skin.

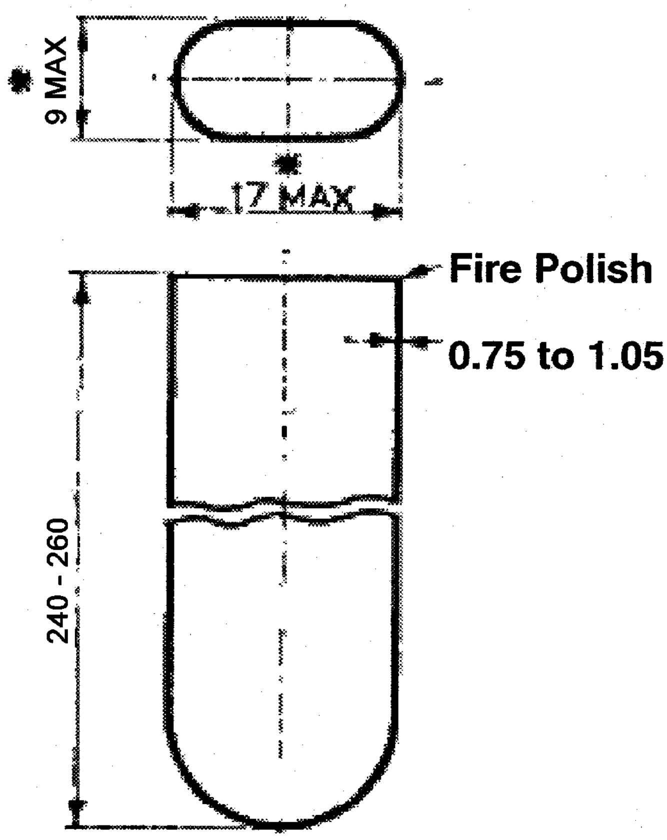
A2.6 Stoddard Solvent

Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid prolonged breathing of vapor or spray mist.
Avoid prolonged or repeated skin contact.

APPENDIX

(Nonmandatory Information)

X1. OPTIONAL USEFUL EQUIPMENT



NOTE 1—Dimensions in millimetres.

NOTE 2—The dimensions are the minimum dimensions that shall allow the introduction of a copper strip.

NOTE 3—The tube shall be free of striae or similar defects.

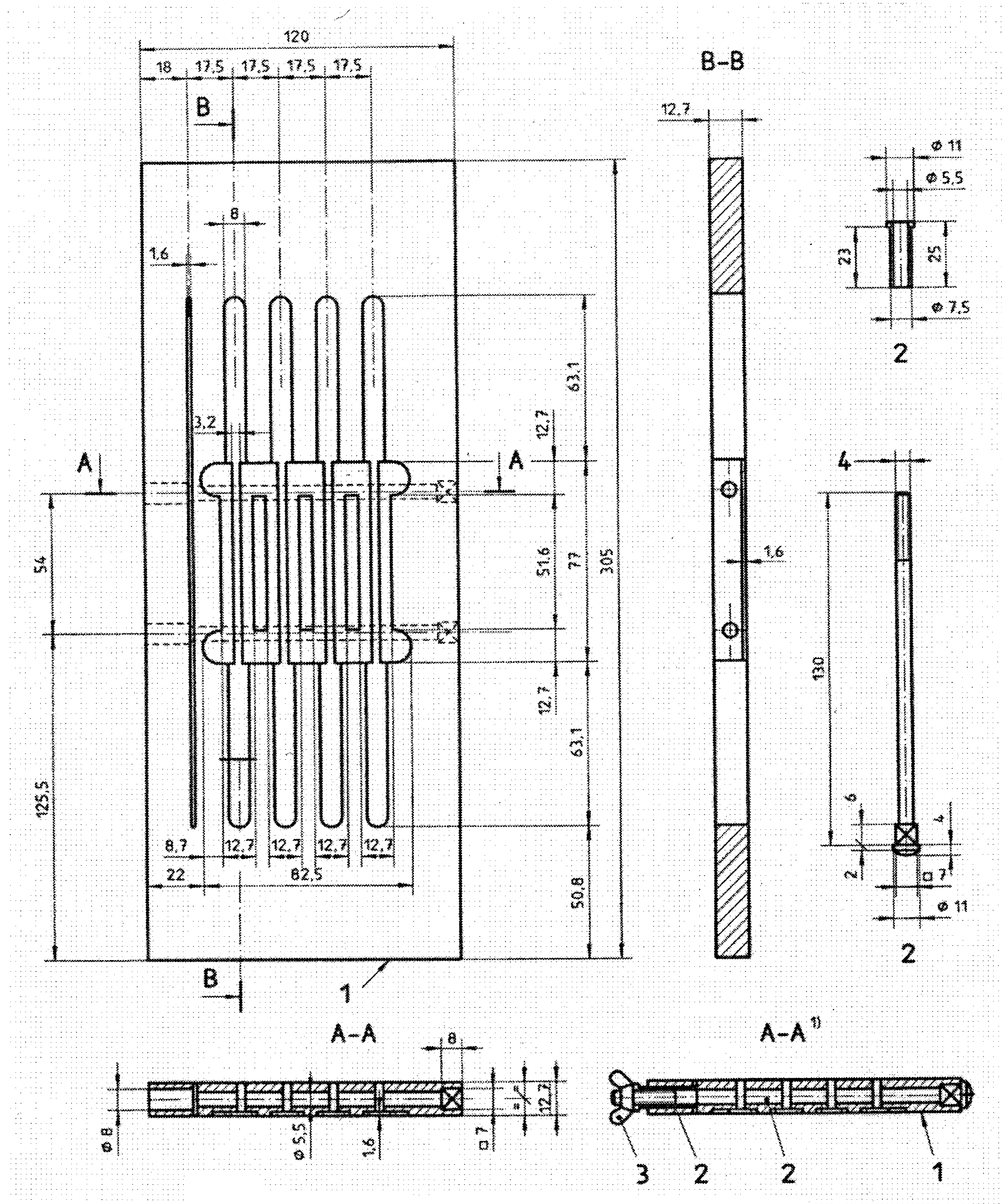
FIG. X1.1 Flat Glass Viewing Test Tube

X1.1 Viewing Tube

X1.1.1 A useful flat glass test tube for holding tarnished copper strips for inspection or for storage for later inspection is illustrated and dimensioned in Fig. X1.1.

X1.2 Strip Vise

X1.2.1 A useful and convenient vise for holding up to four copper strips during final polishing is illustrated and dimensioned in Fig. X1.2.



Key:
1 Material: Plastic
2 Material: Brass
3 Wing nut
4 Ø 5-mm metric thread or equivalent

NOTE—Dimensions in millimetres.

FIG. X1.2 Multistrip Vise

Harris Testing Laboratories, Inc.

CERTIFICATE OF ANALYSIS

CLIENT:
Megattol Inc.
9469 S 500 W
Sandy, Ut 84070

PRODUCT: **FE^{26}** Premier
MARKS: NONE
DATE RECEIVED: 02/11/2008
LAB NO: HH0410-1202-P
SUBMITTED BY: Jon Rose

			SPECIFICATIONS	
METHOD	**TEST**	**RESULT**	**MIN.**	**MAX.**
D-665	**Rust Prevention Test**	**Pass**	**XXX**	**XXX**

Comments:

Amos Mwangi

Date issued:
02/11/2008

CHEMIST

Designation: D 665 – 03

An American National Standard

Designation: 135/93

Standard Test Method for Rust-Preventing Characteristics of Inhibited Mineral Oil in the Presence of Water[1]

This standard is issued under the fixed designation D 665; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the evaluation of the ability of inhibited mineral oils, particularly steam-turbine oils, to aid in preventing the rusting of ferrous parts should water become mixed with the oil. This test method is also used for testing other oils, such as hydraulic oils and circulating oils. Provision is made in the procedure for testing heavier-than-water fluids.

NOTE 1—For synthetic fluids, such as phosphate ester types, the plastic holder and beaker cover should be made of a chemically resistant material, such as polytetrafluoroethylene (PTFE).

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.* For specific warning statements, see 6.4-6.6.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

A 108 Specification for Steel Bars, Carbon, Cold-Finished, Standard Quality

A 240/A 240M Specification for Chromium and Chromium-Nickel Stainless Steel Plate, Sheet, and Strip for Pressure Vessels and for General Applications

D 91 Test Method for Precipitation Number of Lubricating Oils

D 1193 Specification for Reagent Water

D 2422 Classification of Industrial Fluid Lubricants by Viscosity System

D 3603 Test Method for Rust-Preventing Characteristics of Steam Turbine Oil in the Presence of Water (Horizontal Disk Method)

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

E 1 Specification for ASTM Thermometers

Motor Fuels, Section I, Annex A2, Table 32, Reference Materials and Blending Accessories[3]

2.2 *Other Documents:*

Specifications-IP Standard Thermometers, Appendix A[4]

Specifications for IP Standard Reference Liquids, Appendix B[4]

SAE Standard J405 Chemical Composition of SAE Wrought Stainless Steels[5]

BS 871 Specification for abrasive papers and cloths[4]

BS 970: Part 1: Carbon and Carbon Manganese Steels Including Free Cutting Steels[4]

3. Summary of Test Method

3.1 A mixture of 300 mL of the oil under test is stirred with 30 mL of distilled water or synthetic sea water, as required, at a temperature of 60 ± 1°C (140 ± 2°F) with a cylindrical steel test rod completely immersed therein. It is recommended to run the test for 4 h; however, the test period may, at the discretion of the contracting parties, be for a shorter or longer period. The test rod is observed for signs of rusting and, if desired, degree of rusting.

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[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.C0 on Turbine Oils.

Current edition approved Nov. 1, 2003. Published November 2003. Originally approved in 1942. Last previous edition approved in 2002 as D 665–02.

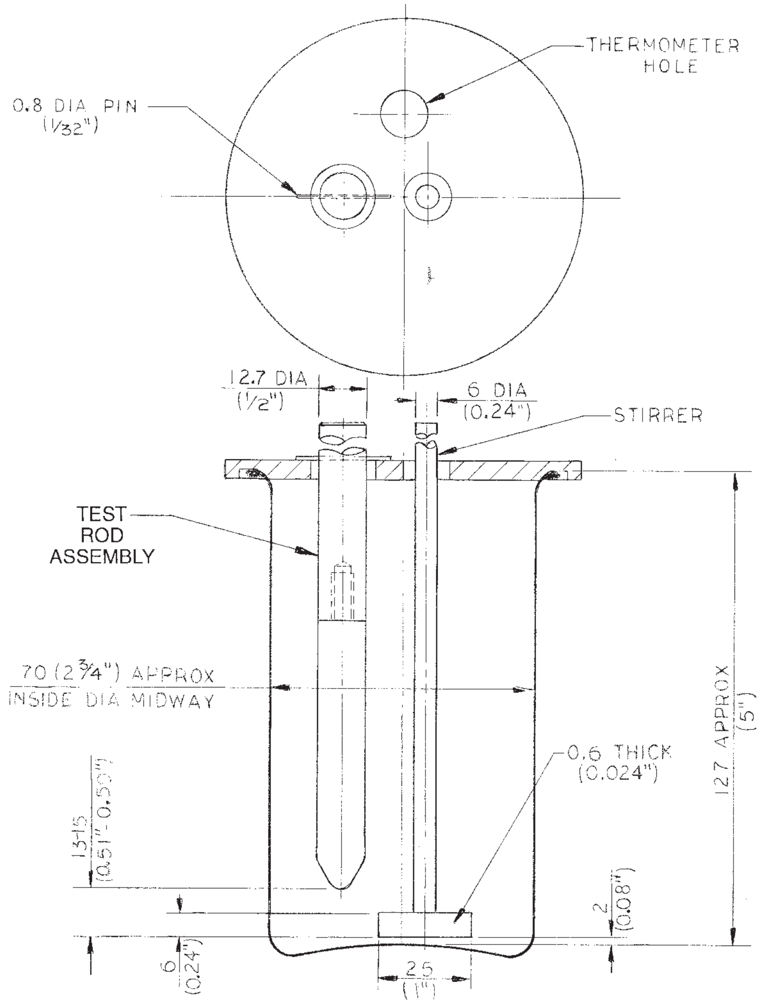
In the IP, this test method is under the jurisdiction of the Standardization Committee. This test method was adopted as a joint ASTM–IP standard in 1964.

[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] See 1996 *Annual Book of ASTM Standards*, Vol 05.04.

[4] *Annual Book of IP Standard Methods for Analysis and Testing of Petroleum and Related Products*, Vol 2.

[5] *1995 SAE Handbook*, Vol 1.



NOTE—All units are in millimetres, unless otherwise specified.

FIG. 1 Rusting Test Apparatus

NOTE 2—Until 1999 it was customary to run the test for 24 h. A round robin with comparisons of different test times showed that no statistically significant differences in rating were found for any sample, between the 4 and 24 h results.[6]

4. Significance and Use

4.1 In many instances, such as in the gears of a steam turbine, water can become mixed with the lubricant, and rusting of ferrous parts can occur. This test indicates how well inhibited mineral oils aid in preventing this type of rusting. This test method is also used for testing hydraulic and circulating oils, including heavier-than-water fluids. It is used for specification of new oils and monitoring of in-service oils.

NOTE 3—This test method was used as a basis for Test Method D 3603. Test Method D 3603 is used to test the oil on separate horizontal and vertical test rod surfaces, and can provide a more discriminating evaluation.

5. Apparatus

5.1 *Oil Bath*—A thermostatically controlled liquid bath capable of maintaining the test sample at a temperature of 60 ± 1°C (140 ± 2°F) (see Note 4). An oil having a viscosity of approximately ISO VG 32 (see Classification D 2422) is suitable for the bath. The bath shall have a cover with holes to accommodate the test beakers.

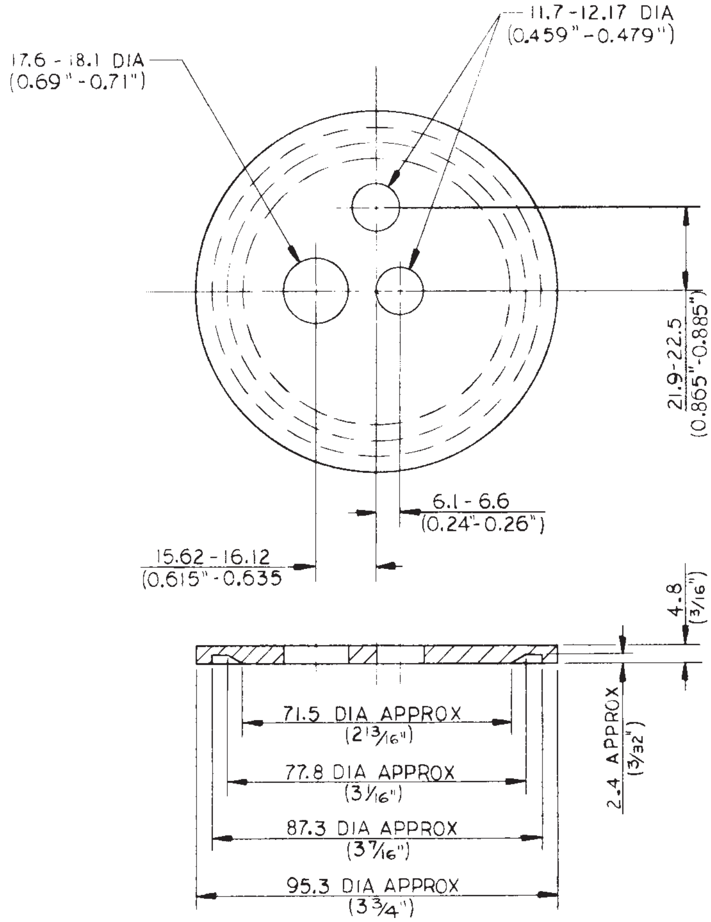
NOTE 4—ASTM Thermometer 9C (9F), as prescribed in Specification E 1, or IP Thermometer 21C in accordance with IP Volume 2, Appendix A, is suitable to indicate the temperature. Alternatively, calibrated thermocouples may be used.

5.2 *Beaker*—A 400-mL, Berzelius-type, tall-form heat-resistant glass[7] beaker without pourout, as shown in Fig. 1, approximately 127 mm (5 in.) in height measured from the inside bottom center and approximately 70 mm (2¾ in.) in inside diameter measured at the middle.

5.3 *Beaker Cover*—A flat beaker cover of glass or poly (methyl methacrylate) (PMMA) (Note 5), kept in position by

[6] Supporting data (results of the cooperative test program with modified test duration) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1474.

[7] Borosilicate glass is satisfactory for this purpose.



NOTE—All units are in millimetres, unless otherwise specified.

FIG. 2 Beaker Cover

suitable means such as a rim or groove. Two holes shall be provided on any diameter of the cover; one for a stirrer 12 mm (15⁄32 in.) in diameter with its center 6.4 mm (1⁄4 in.) from the center of the cover; and the other, on the opposite side of the center of the cover, for the test rod assembly (see Section 8), 18 mm (45⁄64 in.) in diameter with its center 16 mm (5⁄8 in.) from the center of the cover. In addition, a third hole 12 mm (15⁄32 in.) in diameter shall be provided for a temperature measuring device, with its center 22.5 mm (7⁄8 in.) from the center of the cover and on a diameter of the cover at right angles to the diameter through the other two holes.

NOTE 5—An inverted petri dish makes a suitable cover, as the sides of the dish aid in keeping it in position. Fig. 2 shows a PMMA resin cover for the beaker which has been found to be suitable. An optional feature is shown, consisting of a slot, 1.6 by 27 mm (1⁄16 by 11⁄16 in.), which is centered on a diameter of the stirrer hole at right angles to the cover diameter through the test rod hole and stirrer hole. This feature allows withdrawal of the stirrer while the beaker cover is in place. When the test method test is used for other fluids such as synthetics, the beaker cover should be made from chemically resistant material such as polymonochlorotrifluoroethylene (PCTFE).

5.4 The plastic holder shall be made of PMMA resin in accordance with the dimensions shown in Fig. 3 (two types of holders are illustrated). When testing synthetic fluids, the plastic holder should be made from chemically resistant material such as polytetrafluoroethylene (PTFE).

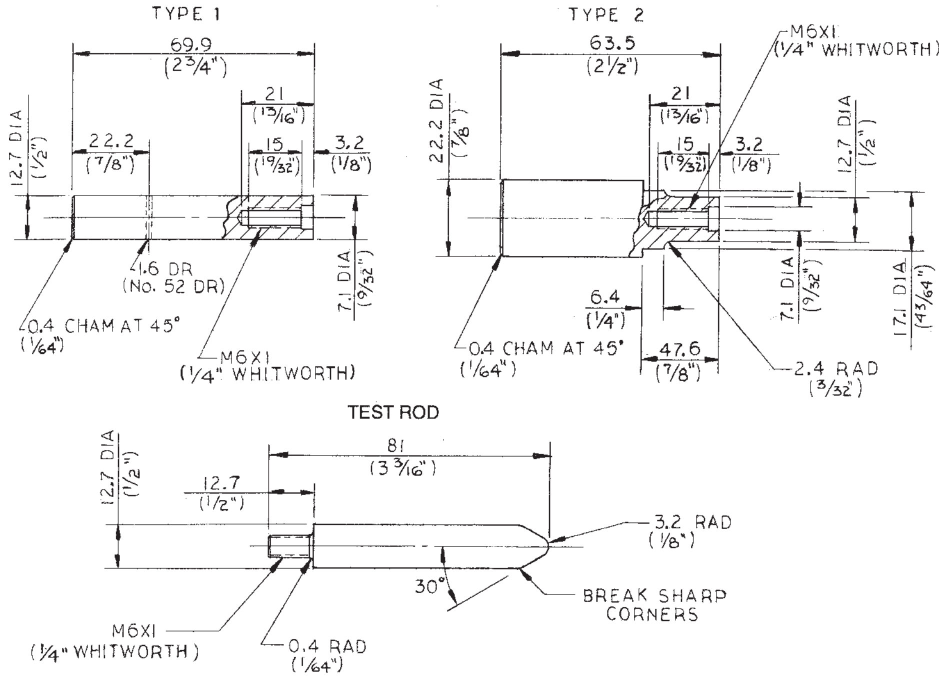
5.5 *Stirrer*—A stirrer constructed entirely from stainless steel (Note 6 and Note 7) in the form of an inverted T. A flat blade 25 by 6 by 0.6 mm (1 by 0.24 by 0.024 in.) shall be attached to a 6-mm (0.24-in.) rod in such a way that the blade is symmetrical with the rod and has its flat surface in the vertical plane.

NOTE 6—A suitable material is an 18 % chromium, 8 % nickel alloy steel conforming to Type 304, of Specification A 240/A 240M, or SAE No. 30304 (see SAE J405), or BS 970: Part 1: 1983: 302S31.

NOTE 7—If stainless steel is not obtainable, stirrers made of heat-resistant glass[7] and having approximately the same dimensions as the stainless steel stirrers specified can be used.

5.6 *Stirring Apparatus*—Any convenient form of stirring apparatus capable of maintaining a speed of 1000 ± 50 rpm.

5.7 *Grinding and Polishing Equipment*—A 150-grit (99-µm) and a 240-grit (53.5-µm) in accordance with BS 871 or its equivalent, metalworking aluminum oxide abrasive cloth coat



NOTE—All units are in millimetres, unless otherwise specified.

FIG. 3 Test Rod and Holders

on a jeans backing, a suitable chuck (see Fig. 4) for holding the test rod, and a means of rotating the test rod at a speed of 1700 to 1800 rpm.

5.8 *Oven*, capable of maintaining a temperature of 65°C (150°F).

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[8] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D 1193.

6.3 The synthetic sea water shall have the following composition:

Salt	g/L
NaCl	24.54
$MgCl_2 \cdot 6H_2O$	11.10
Na_2SO_4	4.09
$CaCl_2$	1.16
KCl	0.69
$NaHCO_3$	0.20
KBr	0.10
H_3BO_3	0.03
$SrCl_2 \cdot 6H_2O$	0.04
NaF	0.003

6.3.1 The solution can be conveniently prepared as follows. This procedure avoids any precipitation in concentrated solutions with subsequent uncertainty of complete resolution. Using certified pure (cp) chemicals and distilled water, prepare the following stock solutions:

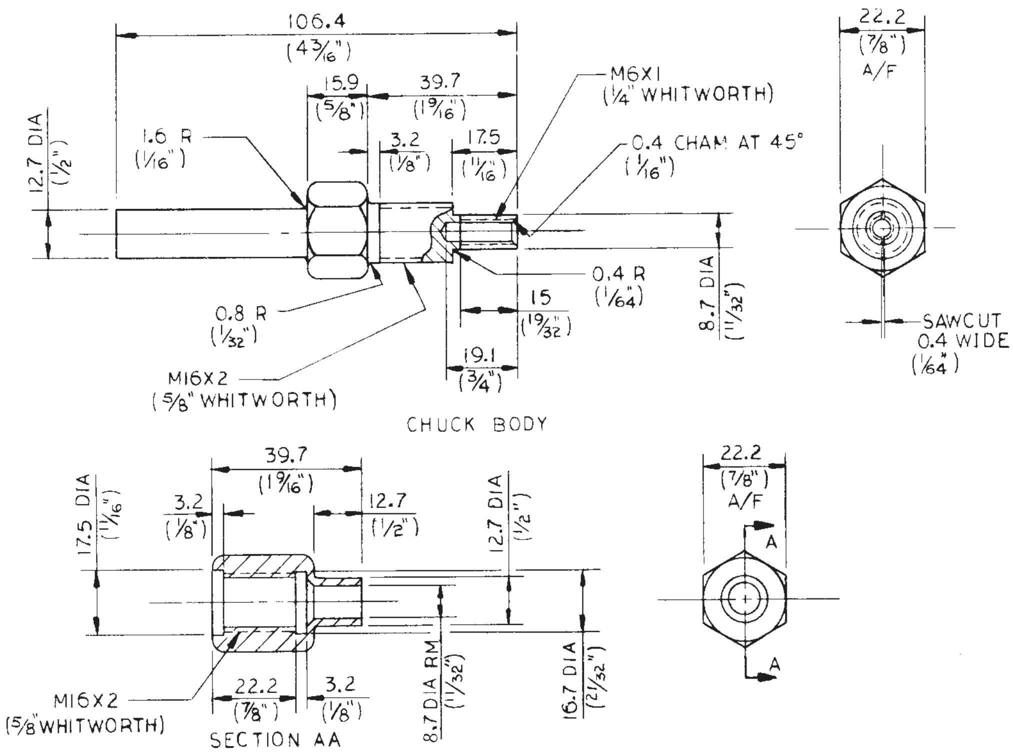
Stock Solution No. 1:

$MgCl_2 \cdot 6H_2O$	3885 g
$CaCl_2$(anhydrous)	406 g
$SrCl_2 \cdot 6H_2O$	14 g
Dissolve and dilute to 7 L	

Stock Solution No. 2:

KCl	483 g
$NaHCO_3$	140 g
KBr	70 g
H_3BO_3	21 g
NaF	2.1 g
Dissolve and dilute to 7 L	

[8] *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.



NOTE—All units are in millimetres, unless otherwise specified.

FIG. 4 Chuck for Polishing Test Rods

6.3.2 To prepare the synthetic sea water, dissolve 245.4 g of NaCl and 40.94 g of Na_2SO_4 in a few litres of distilled water, add 200 mL of Stock Solution No. 1 and 100 mL of Stock Solution No. 2 and dilute to 10 L. Stir the 10-L batch and add 0.1 *N* Na_2CO_3 solution until the pH is between 7.8 and 8.2. One or two millilitres of the carbonate solution will be required.

6.4 *Precipitation Naphtha*, as specified in Test Method D 91. (**Warning—**Flammable. Health hazard.)

6.5 *Isooctane*, as specified in Table 32, Motor Fuels Section I, Annex A2, Reference Materials and Blending Accessories. (**Warning**—Flammable. Health hazard.)

6.6 *IP 60/80 Petroleum Spirit*, as specified by IP Standard Reference Liquids. (**Warning**—Flammable. Health hazard.)

6.7 *Steel Test Rod*, as specified in Section 8.

7. Sampling

7.1 Sample for this test can come from tanks, drums, small containers, or even operating equipment, and they should be truly representative of the entire quantity. Therefore, use the applicable apparatus and techniques described in Practice D 4057, or other comparable standard practice.

8. Test Rod and Its Preparation

8.1 For each test oil, prepare two steel test rods. These may either be new or from a previous test (see Note 8) and shall be prepared in accordance with 8.2 and 8.3.

8.2 The test rod assembly shall consist of a round steel test rod fitted to a plastic holder. The round steel test rod, when new, shall be 12.7 mm (0.5 in.) in diameter and approximately 68 mm (2 11⁄16 in.) in length exclusive of the threaded portion which screws into the plastic holder and shall be tapered at one end as shown in Fig. 3. It shall be made of steel conforming to Grade 10180 of Specification A 108 or to BS 970 Part I: 1983-070M20. If these steels are not available, other equivalent steels may be used, provided they are found to be satisfactory by comparative test using this Test Method D 665 – IP 135.

NOTE 8—When making a check test, the steel test rod that showed rust should not be reused. Test rods that repeatedly show rust in tests of various oils can be imperfect. Such test rods should be used with oils known to pass the test. If rusting occurs in repeat tests, these test rods should be discarded.

8.3 *Preliminary Grinding*—If the test rod has been used previously and is free of rust or other irregularities, the preliminary grinding may be omitted, and it may be subjected only to final polishing as prescribed in 8.4. If the test rod is new or if any part of its surface shows rust or other irregularities, clean it with ASTM precipitation naphtha or *iso*octane or IP 60/80 petroleum spirit and grind with medium 150-grit aluminum oxide cloth to remove all irregularities, pits, and scratches, as determined by visual inspection (Note 9). Perform the grindings by mounting the test rod in the chuck of the grinding and polishing apparatus, and turning it at a speed of 1700 to 1800 rpm while applying the 150-grit aluminum oxide cloth. Old 150-grit aluminum oxide cloth may be used to remove rust or major irregularities, but complete the grinding with new cloth. Proceed at once with the final polishing with 240-grit aluminum oxide cloth, or remove the test rod from the chuck and store in *iso*octane until needed. Discard reused test rods when the diameter is reduced to 9.5 mm (0.375 in.).

NOTE 9—Do not touch the test rods with the hands at any stage after cleaning with naphtha or *iso*octane or the petroleum spirit (which precedes either preliminary grinding or final polishing) until the test is completed. Forceps or a clean, lintless cloth may be used.

8.4 *Final Polishing*:

8.4.1 Just before the test is to be made, subject the test rod to final polishing with 240-grit aluminum oxide cloth. If the preliminary grinding has just been completed, stop the motor that rotates the test rod. Otherwise, remove the test rod from the *iso*octane (previously used unrusted test rods shall be stored in this reagent), dry with a clean cloth, and place in the chuck. Rub a new piece of 240-grit aluminum oxide cloth longitudinally over the static test rod until the rounded end, and the entire surface show visible scratches. Rotate the test rod at a speed of 1700 to 1800 rpm. Take the cloth and place it halfway around the test rod, and apply a firm but gentle downward pull to the loose ends of the cloth for about 1 to 2 min so as to produce a uniform finely scratched surface free of longitudinal scratches. Carry out the final stages of the polishing with new cloth.

8.4.2 To ensure that the flat shoulder (that portion of the test rod perpendicular to the threaded stem) is free of rust, polish this area. This can be done by holding a strip of 240-grit aluminum oxide cloth between the chuck and the shoulder while rotating the test rod for a brief period.

8.4.3 Remove the test rod from the chuck without touching with the fingers; wipe lightly with a clean, dry, lintless cloth or tissue (or brush the test rod lightly with a camel's hair brush); attach to the plastic holder; and immediately immerse in the oil to be tested. This can be either the hot oil sample (see 9.1) or a clean test tube containing a portion of the sample. The test rod can be removed later from this tube and allowed to drain briefly before being placed in the hot oil.

9. Procedure A for Distilled Water

9.1 Clean the beaker in accordance with good laboratory procedure, wash with distilled water, and dry in an oven. Clean glass beaker cover and a glass stirrer by the same procedure. To clean a stainless steel stirrer and a PMMA cover, use ASTM precipitation naphtha or *iso*octane or IP 60/80 petroleum spirit, wash thoroughly with hot water and finally with distilled water, and dry in an oven at a temperature not over 65°C (150°F). Pour 300 mL of the oil to be tested (see Practice D 4057) into the beaker and place the beaker in the oil bath held at a temperature that will maintain the oil sample at 60 ± 1°C (140 ± 2°F). Insert the beaker into a hole of the bath cover and suspend in the hole with the beaker rim resting on the bath cover. The oil level in the bath shall not be below the oil level in the test beaker. Cover the beaker with the beaker cover with the stirrer in position in the proper opening. Adjust the stirrer so that the shaft is 6 mm (0.24 in.) off center in the beaker containing the oil sample and the blade is not more than 2 mm (0.08 in.) from the bottom of the beaker. Then suspend a temperature measuring device (see Note 10) through the hole in the cover intended for that purpose so that it is immersed to a depth of about 56 mm (2.2 in.). Start the stirrer and when the temperature reading reaches 60 ± 1°C (140 ± 2°F), insert the steel test rod prepared in accordance with Section 8.

9.2 Insert the test rod assembly through the test rod hole in the beaker cover and suspend so that its lower end is 13 to 15 mm (0.51 to 0.59 in.) from the bottom of the beaker. Either type of plastic test rod holder (see Fig. 3) may be used. The hole through which the test rod is suspended shall be unobstructed (see Note 12).

NOTE 10—When analyzing multiple samples of a similar nature that are introduced into a thermostatically controlled bath at approximately the same time (that is, individual samples being analyzed as a batch), data collected has shown that it is not necessary to suspend a temperature measuring device through the hole in the cover intended for that purpose in each of the samples, since a thermostatically controlled bath is capable of maintaining the proper bath temperature within the allowed limits at each of the sample beaker locations. As such, it is permissible to suspend a temperature measuring device through the hole in the cover intended for that purpose in as few as one of the samples being analyzed, immersed to a depth of about 56 mm (2.2 in.). The temperature reading measured in the sample beaker location selected is the basis for determining when the temperature reaches 60 ± 1°C (140 ± 2°F) in order to begin stirring each of the beakers and inserting the steel test rods.

NOTE 11—In order not to disturb the thermal equilibrium in the oil bath once stirring of the samples has begun, no additional samples are to be added to the oil bath.

NOTE 12—Fig. 1 shows the arrangement of the apparatus.

9.3 Continue stirring for 30 min to ensure complete wetting of the steel test rod. With the stirrer in motion, remove the temperature measuring device (if applicable, see Note 10) temporarily and add 30 mL of distilled water through this hole, discharging the water on the bottom of the beaker, and replace the temperature measuring device (if applicable). Continue stirring at a speed of 1000 ± 50 rpm for 4 h (see Note 13) from the time water was added, maintaining the temperature of the oil-water mixture at 60 ± 1°C (140 ± 2°F). Stop stirring at the end of the 4-h period, remove the test rod, allow to drain, and then wash with ASTM precipitation naphtha or *iso*octane, or IP 60/80 petroleum spirit. If desired, the test rod may be preserved by lacquering.

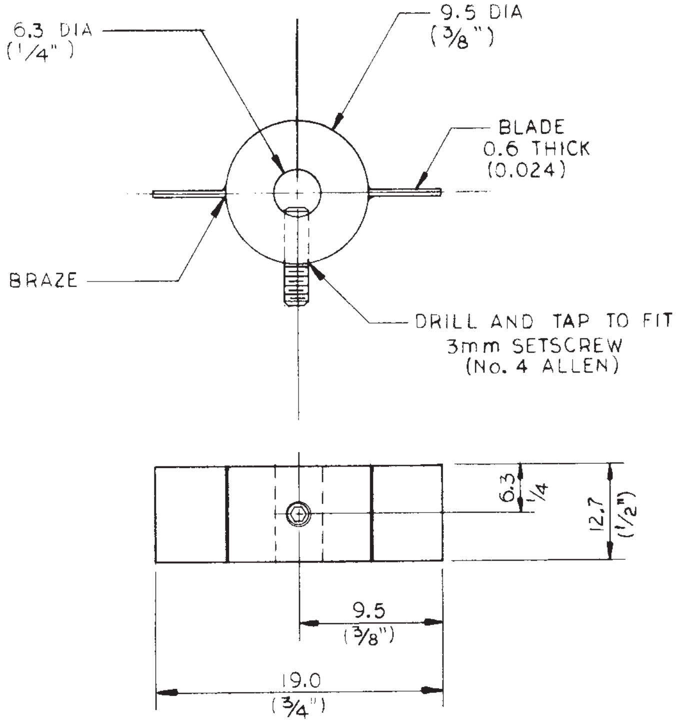
NOTE 13—It is recommended to run the test for 4 h; however, the test period may, at the discretion of the contracting parties, be for a shorter or longer period.

10. Procedure B for Synthetic Sea Water

10.1 The procedure for rust-preventing characteristics of inhibited mineral oils in the presence of synthetic sea water shall be identical with that described in 9.1, 9.2, and 9.3, except use synthetic sea water in place of distilled water in that portion of the procedure described in 9.3.

11. Procedure C for Heavier-Than-Water Fluids

11.1 The stirring action provided by the stirrer prescribed in 5.5 is not sufficient to thoroughly mix the water and test sample when the fluid under test is heavier than water. This section is meant to define changes in the test method to accommodate heavier-than-water fluids. Except as specifically stated, all the requirements of the previous Sections 1-10 shall apply. Since this procedure can be performed with distilled water or synthetic sea water, take care to include this information in the report.



NOTE—All units are in millimetres, unless otherwise specified.

FIG. 5 Auxiliary Stirrer Blade (not to scale)

11.2 *Apparatus*:

11.2.1 *Beaker Cover*—Same as prescribed in 5.3 (see Note 14).

NOTE 14—Some heavier-than-water fluids can attack or dissolve PMMA beaker covers and test rod holders. It is recommended that PCTFE beaker covers and PTFE test rod holders be used when testing heavier-than-water fluids.

11.2.2 *Stirrer*—Same as prescribed in 5.5 but with an auxiliary blade attached to the stirrer shaft. The auxiliary blade shall be of stainless steel, 19.0 by 12.7 by 0.6 mm (0.75 by 0.50 by 0.25 in.) as shown in Fig. 5. The auxiliary blade shall be positioned on the stirrer shaft so that the bottom edge of the auxiliary blade is 57 mm (2.25 in.) above the top edge of the fixed lower blade, and so that the flat surfaces of both blades are in the same vertical plane.

11.3 *Test Rod and Its Preparation*—Same as prescribed in Section 8.

12. Interpretation of Results

12.1 Perform all inspections at the end of the test to determine the condition of test rods without magnification under normal light. For the purpose of this test, normal light is considered to be illumination of about 60 footcandles (650 lx). Within the meaning of this test method, a rusted test rod is one on which any rust spot or rust streak is visible by the above inspection procedure.

12.2 For the purpose of this test method, rust is an area of corrosion of the test surface that is identified by color and is confirmed by the presence of pits or roughness if the surface is wiped with a lintless cloth or tissue paper. Neither surface discoloration nor specks, which can easily be removed with a lintless or tissue paper cloth with no evidence of pitting or roughness, shall be considered to be rust.

12.3 In order to report an oil as passing or failing, conduct the test in duplicate. Report an oil as passing the test if both test rods are rust-free at the end of the test period. Report an oil as failing the test if both test rods are rusted at the end of the test period (see Note 15). If one test rod is rusted while the other is free of rust, test two additional test rods (see Note 8). If either of these latter test rods shows rusting, report the oil as not passing the test. If neither of these latter test rods shows rusting, report the oil as passing the test.

NOTE 15—An indication of the degree of rusting occurring in this test may be desired. For uniformity in such cases, use of the following classifications of rusting severity is recommended:

Light Rusting—Rusting confined to not more than six spots, each of which is 1 mm or less in diameter.

Moderate Rusting—Rusting in excess of the above but confined to less than 5 % of the surface of the test rod.

Severe Rusting—Rusting covering more than 5 % of the surface of the test rod.

12.4 A reference oil with a Pass in Procedure A and a Fail in Procedure B can be prepared as follows: Add 0.0150 mass % of an additive concentrate[9] to a white mineral oil.[10] The additive concentrate consists of 60 mass % of dodecenyl succinic acid and 40 mass % of normal paraffin oil, ISO VG 22 (see Classification D 2422).[11]

13. Report

13.1 The test report shall contain the following:

13.1.1 The type and identification of the product used,

13.1.2 The date of the test,

13.1.3 A reference to this ASTM-IP standard, indicating whether Procedure A, B, or C was used. Since Procedure C may be followed with either distilled water or sea water, be sure to include the type of water used in this case,

13.1.4 The duration of the tests,

13.1.5 Any deviation from the procedure specified, and

13.1.6 The result of the test, including degree of severity of rusting, if desired.

14. Precision and Bias

14.1 No generally accepted method for determining precision or bias is currently available.[11]

15. Keywords

15.1 circulating oils; heavier-than-water fluids; hydraulic oils; inhibited mineral oil; rust-preventing characteristics; steam-turbine oils

[9] A suitable additive concentrate is Lubrizol 850, as used in the ASTM round robin. Lubrizol 850 is available from Lubrizol Corporation, Wickliffe, OH.

[10] A suitable white mineral oil is USP Mineral Oil with a viscosity in the order of ISO VG 32 (Penreco Drakeol 19 was used in the ASTM round robin and is available from Penreco, Karns City, PA).

[11] Supporting data (results of the cooperative test program using this reference oil) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1284.

Harris Testing Laboratories, Inc.

CERTIFICATE OF ANALYSIS

CLIENT:
Megatrol Inc.
9469 S 500 W
Sandy, Ut. 84070

PRODUCT: **FE**^{26} Premier
MARKS: NONE
DATE RECEIVED: 02/11/2008
LAB NO: HH0410-1202-P
SUBMITTED BY: Jon Rose

			SPECIFICATIONS	
METHOD	**TEST**	**RESULT**	**MIN.**	**MAX.**
D-892	**Foaming Tendency, Sequence 1**	**XXX**	**XXX**	**XXX**
	Foaming Volume, ml @ the end of 5-min blowing period	**18**	**XXX**	**XXX**
	Foaming Volume, ml @ the end of 10-min setting period	**0**	**XXX**	**XXX**

Comments:

Amos Mwangi

Date issued:
02/11/2008

CHEMIST

Designation: D 892 – 03

An American National Standard
British Standard 5092

Designation: 146/2000

Standard Test Method for Foaming Characteristics of Lubricating Oils[1]

This standard is issued under the fixed designation D 892; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This test method covers the determination of the foaming characteristics of lubricating oils at 24°C and 93.5°C. Means of empirically rating the foaming tendency and the stability of the foam are described.

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

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific warning statements, see Sections 7, 8, and 9.1.1.

2. Referenced Documents

2.1 *ASTM Standards:*

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids and the Calculation of Dynamic Viscosity[2]

D 4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants[3]

E 1 Specification for ASTM Thermometers[4]

E 128 Test Method for Maximum Pore Diameter and Permeability of Rigid, Porous Filters for Laboratory Use[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *diffuser*, *n—for gas*, a device for dispersing gas into a fluid.

3.1.1.1 *Discussion*—In this test method the diffuser may be made of either metallic or non-metallic materials.

3.1.2 *entrained air (or gas)*, *n*—in liquids, a two-phase mixture of air (or gas) dispersed in a liquid in which the volume of the liquid is the major component.

3.1.2.1 *Discussion*—The air (or gas) is in the form of discrete bubbles of about 10 to 1000 µm in diameter. The bubbles are not uniformly dispersed. In time they tend to rise to the surface to coalesce to form larger bubbles which break or form foam. Subsurface coalescence can also occur, in which case, the bubbles rise more rapidly.

3.1.3 *foam, n—in liquids*, a collection of bubbles formed in the liquid or on (at) its surface in which the air (or gas) is the major component on a volumetric basis.

3.1.4 *lubricant*, *n*—any material interposed between two surfaces that reduces the friction or wear between them. **D 4175–88**

3.1.4.1 *Discussion*—In this test method, the lubricant is an oil which can or can not contain additives such as foam inhibitors.

3.1.5 *maximum pore diameter*, *n—in gas diffusion*, the diameter a capillary of circular cross section which is equivalent (with respect to surface tension effects) to the largest pore of the diffuser under consideration.

3.1.5.1 *Discussion*—The pore dimension is expressed in micrometres in this test method.

3.1.6 *permeability*, *n—in gas diffusion*, the flow of gas, through the gas diffuser.

3.1.6.1 *Discussion*—In this test method, the permeability is measured at a pressure of 2.45 kPa (250 mm of water) in millilitres per minute.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *dynamic bubble*, *n*—the first bubble to pass through and escape from the diffuser followed by a continuous succession of bubbles when testing for the maximum pore diameter in Annex A1.

3.2.1.1 *Discussion*—When a diffuser is immersed in a liquid, air can be trapped in the pores. It can escape eventually or as soon as a pressure is applied to the diffuser. When testing

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.06 on Analysis of Lubricants.

Current edition approved May 10, 2003. Published May 2003. Originally approved in 1946. Last previous edition approved in 2002 as D 892–02.

In the IP, this test method is under the jurisdiction of the Standardization Committee. This test method has been approved by the sponsoring committees and accepted by the cooperating societies in accordance with established procedures.

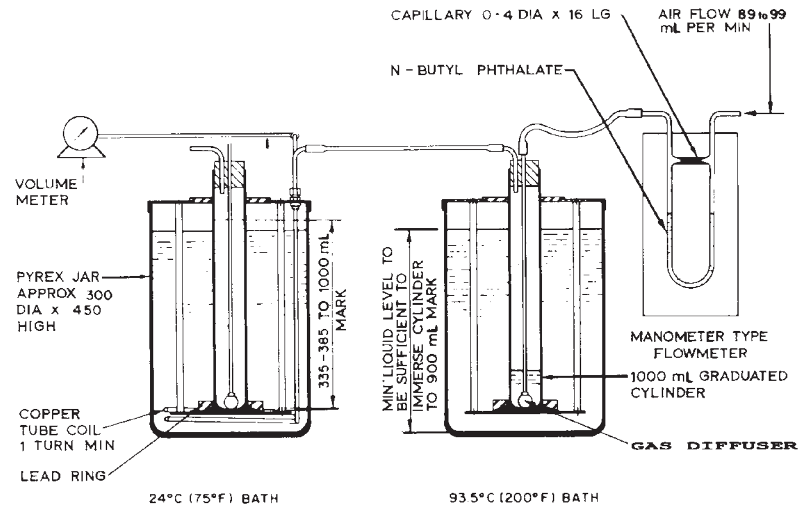
[2] *Annual Book of ASTM Standards*, Vol 05.01.

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

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

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

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



All dimensions in millimetres.

FIG. 1 Foaming Test Apparatus

for maximum pore diameter (Annex A1) the escape of such bubble shall be ignored.

3.2.2 *foam stability, n*—in foam testing, the amount of foam remaining at the specified time following the disconnecting of the air supply.

3.2.2.1 *Discussion—In this test method,* foam stability is determined from measurements made 10 min ± 10 s after disconnecting the air supply.

3.2.3 *foaming tendency*, *n*—in foam testing, the amount of foam determined from measurements made immediately after the cessation of air flow.

4. Summary of Test Method

4.1 The sample, maintained at a temperature of 24°C (75°F) is blown with air at a constant rate for 5 min, then allowed to settle for 10 min. The volume of foam is measured at the end of both periods. The test is repeated on a second sample at 93.5°C (200°F), and then, after collapsing the foam, at 24°C (75°F).

5. Significance and Use

5.1 The tendency of oils to foam can be a serious problem in systems such as high-speed gearing, high-volume pumping, and splash lubrication. Inadequate lubrication, cavitation, and overflow loss of lubricant can lead to mechanical failure. This test method is used in the evaluation of oils for such operating conditions.

6. Apparatus

6.1 *Foaming Test Apparatus*, an example of a suitable set-up is shown in Fig. 1, consisting of a 1000-mL graduated cylinder or cylinders held in position when placed in the baths, such as fitted with a heavy ring or clamp assembly to overcome the buoyancy, and an air-inlet tube, to the bottom of which is fastened a gas diffuser. The gas diffuser can be either a 25.4-mm (1-in.) diameter spherical gas diffuser stone[6] made of fused crystalline alumina grain, or a cylindrical metal diffuser[7] made of sintered five micron porous stainless steel (Note 1). The cylinder shall have a diameter such that the distance from the inside bottom to the 1000-mL graduation mark is 360 ± 25 mm. It shall be circular at the top (Note 2) and shall be fitted with a stopper, such as those made of rubber, having one hole at the center for the air-inlet tube and a second hole off-center for an air-outlet tube. The air-inlet tube shall be adjusted so that, when the stopper is fitted tightly into the cylinder, the gas diffuser (Note 3) just touches the bottom of the cylinder and is approximately at the center of the circular cross section. Gas diffusers shall meet the following specification when tested in accordance with the method given in Annex A1:

Maximum pore diameter, µm	Not greater than 80
Permeability at pressure of 2.45 kPa (250 mm) water, mL of air/min	3000 to 6000

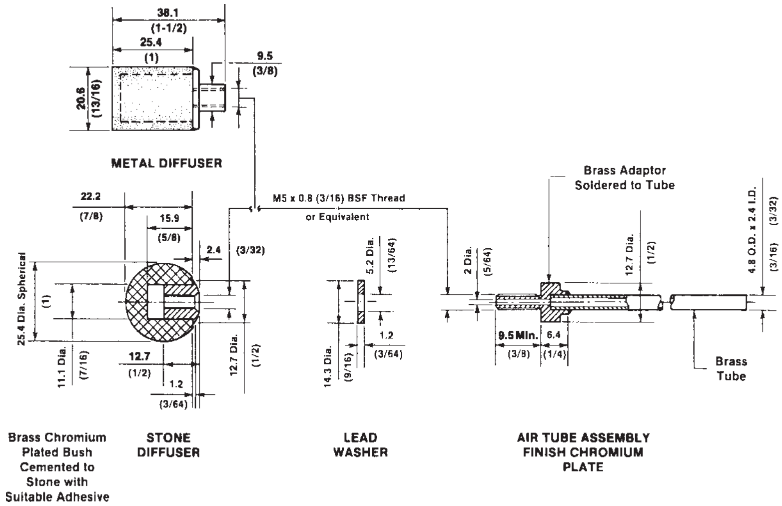
NOTE 1—Gas diffuser permeability and porosity can change during use; therefore, it is recommended that diffusers be tested when new and periodically thereafter preferably after each use.

NOTE 2—Graduated cylinders with circular tops can be prepared from cylinders with pouring spouts by cutting them off below the spouts. The cut surface is to be smoothed before use by fire polishing or grinding.

NOTE 3—Gas diffusers may be attached to air-inlet tubes by any suitable means. A convenient arrangement is shown in Fig. 2.

[6] The sole source of supply of the diffuser stones known to the committee at this time is Norton Co., Industrial Ceramics Div., Worcester, MA 01606, under the designation AX536, Alundum porous spheres.. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee [1], which you may attend.

[7] The sole source of supply of the metal diffusers known to the committee at this time is Petrolab Corp., 874 Albany-Shaker Road, Latham, NY 12110 under the designation M13-0653. The names of suitable suppliers of diffuser stones and metal diffusers in the United Kingdom may be obtained from the Institute of Petroleum.. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee [1], which you may attend.



Dimensions in millimetres (inches)

FIG. 2 Attachment of Gas Diffusers to Air-Inlet Tubes

6.2 *Test Baths*, large enough to permit the immersion of the cylinder at least to the 900-mL mark and capable of being maintained at temperatures constant to 0.5°C (1°F) at 24°C (75°F) and 93.5°C (200°F), respectively. Both bath (Note 5) and bath liquid shall be clear enough to permit observation of the graduations on the cylinder.

Note 4—Air baths may also be utilized for heating purposes. Limited data has shown that both liquid and air baths give equivalent results. However, the precision estimates given in Section 13 are based on using only liquid baths.[8]

Note 5—Heat-resistant cylindrical glass jars approximately 300 mm (12 in.) in diameter and 450 mm (18 in.) in height make satisfactory baths.

6.3 *Air Supply*, from a source capable of maintaining an air flow rate of 94 ± 5 mL/min through the gas diffuser. The air shall be passed through a drying tower 300 mm in height packed as follows: just above the constriction place a 20-mm layer of cotton, then a 180-mm layer of indicating desiccant, and a 20-mm layer of cotton. The cotton serves to hold the desiccant in place. Refill the tower when the indicating desiccant begins to show presence of moisture. A flowmeter sensitive to the required tolerances can be used to measure the air flow (Note 6).

Note 6—A manometer type flowmeter, in which the capillary between the two arms of the U-tube is approximately 0.4 mm in diameter and 16 mm in length, and in which *n*-butylphthalate is the manometric liquid, is suitable.

6.3.1 The total volume of air leaving the foaming test apparatus shall be measured by a volume measuring device (Note 8) capable of accurately measuring gas volumes of about 470 mL. The air shall be passed through at least one loop of copper tubing placed around the inside circumference of the cold bath so that the volume measurement is made at approximately 24°C (75°F). Precautions are to be taken to avoid leaks at any point in the system.

Note 7—Alternatively, a 1 L cylinder (with 10 mL graduation marks) full of water is inverted in a tall, large beaker also filled with water. There should be no air bubbles inside. Air leaving the copper loop in the bath is connected below the cylinder. When the test is started, air will flow into the cylinder, displacing the water. At the end of the test, the volume of air in the cylinder is measured by equalizing the water levels inside and outside the cylinder. Alternatively, the total volume of air passed would be the difference between the final and the initial volumes of water in the cylinder.

Note 8—A wet test meter calibrated in hundredths of a litre is suitable.

6.4 *Timer*, graduated and accurate to 1 s or better.

6.5 *Thermometer*, having a range as shown below and conforming to the requirements as prescribed in Specification E 1 or specifications for IP thermometers:

Temperature Range	Thermometer ASTM	No. IP
−5 to 215°F	12F	64F
−20 to 102°C	12C	64C

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all cases. Unless indicated otherwise, it is intended that all reagents conform to the specifications of the committee on Analytical Reagents of the American Chemical Society where such specifications are available.[9] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Acetone*—(**Warning**—Extremely flammable, vapors can cause a flash fire).

7.3 *Compressed Air*, hydrocarbon free and dry to a dew point of −60°C or lower.

Note 9—If the source of compressed air is ensured to the stated

[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1516.

[9] *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 *Annual 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.

specifications, the drying tower in 6.3 may not be necessary.

7.4 *Heptane*—(**Warning**—Flammable, vapor harmful).

7.5 *Propan-2-ol*—(Solvents with equivalent cleaning and solvency characteristics may be substituted for propan-2-ol.)

7.6 *Toluene (Methylbenzene)*—(Solvents with equivalent cleaning and solvency characteristics may be substituted for toluene.)

8. Hazards

8.1 (**Warning**—Users of this test method shall be trained and familiar with all normal laboratory practices, or under the immediate supervision of such a person. It is the responsibility of the operator to ensure that all local legislative and statutory requirements are met.)

8.2 (**Warning**—Cleaning solvents have flash points lower than ambient temperatures. Avoid the possibility of fire or explosion.)

8.3 (**Warning**—The fumes from the test oil and the bath shall be vented in a manner compatible with local government regulations.)

8.4 (**Warning**—Some apparatus assemblies can have as much as 20 L of heat transfer oil at 93.5°C. Therefore, in the event of breakage of the containing vessel, provisions for suitable containment of the spill is advisable.)

9. Preparation of Apparatus

9.1 Thorough cleansing of the test cylinder and air-inlet tube is essential after each use to remove any additive remaining from previous tests which can seriously interfere with results of subsequent tests.

9.1.1 *Cylinder*—Rinse the cylinder with heptane. (**Warning**—Flammable, vapor harmful.) Wash the cylinder with a suitable detergent. Rinse the cylinder, in turn, with distilled water, then acetone (**Warning**—Extremely flammable, vapors can cause a flash fire) and dry in a current of the compressed air or in a drying oven. Interior walls that drain the water cleanly, that is without drops forming, are adequately cleaned.

NOTE 10—Certain detergents are notorious for adhering to glass; therefore, it is important to realize that such a circumstance can affect the test result. Several rinsings with water and acetone may be required.

9.1.2 *Gas Diffuser and Air Tube*—Clean the gas diffuser with toluene (solvents with equivalent cleaning and solvency characteristics may be substituted for toluene.) and heptane. Immerse the gas diffuser in about 300 mL of toluene. Flush a portion of the toluene back and forth through the gas diffuser at least five times with vacuum and air pressure. Repeat the process with heptane. After the final washing, dry both the tube and the gas diffuser thoroughly by forcing clean air through them. Wipe the outside of the air inlet tube, first with a cloth moistened with heptane, then a dry cloth. Do not wipe the gas diffuser.

NOTE 11—Certain samples may contain ingredients which may not be adequately removed by this process and, because these can affect the next test, more rigorous cleaning may be required; this is recommended. When alternate diffuser cleaning methods are used certain cautions should be observed: (*1*) Non-metallic diffusers may have absorbed as well as adsorbed these interfering ingredients or the cleaners, or both, and this shall be considered before proceeding to the next test. (*2*) So that all tests performed start off under the same circumstances, when alternate diffuser cleaning methods are used, the final rinsing process shall be as detailed in 9.1.2. (*3*) See also Note 1.

10. Procedure

10.1 *Sequence I—Without* mechanical shaking or stirring, decant approximately 200 mL of sample into a beaker. Heat to 49 ± 3°C (120 ± 5°F) and allow to cool to 24 ± 3°C (75 ± 5°F). See Option A for stored sample (see 10.5). Each step of the procedure described in 10.3 and 10.4, respectively, shall be carried out within 3 h after completion of the previous step. In 10.5.1, the test shall be carried out as soon as compatible with the temperature specification and not more than 3 h after immersion of the cylinder in the 93.5°C (200°F) bath.

10.2 Pour the sample into the 1000-mL cylinder until the liquid level is at the 190-mL mark. Immerse the cylinder at least to the 900-mL mark in the bath maintained at 24 ± 0.5°C (75 ± 1°F). When the oil has reached the bath temperature, insert the gas diffuser and the air-inlet tube with the air source disconnected, and permit the gas diffuser to soak for about 5 min. Connect the air-outlet tube to the air volume measuring device. At the end of 5 min, connect to the air source, adjust the air flow rate to 94 ± 5 mL/min, and force clean dry air through the gas diffuser for 5 min ± 3 s, timed from the first appearance of air bubbles rising from the gas diffuser. At the end of this period, shut off the air flow by disconnecting the hose from the flow meter and immediately record the volume of foam; that is, the volume between the oil level and the top of the foam. The total air volume which has passed through the system shall be 470 ± 25 mL. Allow the cylinder to stand for 10 min ± 10 s and again record the volume of foam.

10.3 *Sequence II*— Pour a second portion of sample into a cleaned 1000-mL cylinder until the liquid level is at the 180-mL mark. Immerse the cylinder at least to the 900-mL mark in the bath maintained at 93.5 ± 0.5°C (200 ± 1°F). When the oil has reached a temperature of 93 ± 1°C (199 ± 2°F), insert a clean gas diffuser and air-inlet tube and proceed as described in 10.2, recording the volume of foam at the end of the blowing and settling periods.

10.4 *Sequence III*— Collapse any foam remaining after the test at 93.5°C (200°F) (10.3), by stirring. Cool the sample to a temperature below 43.5°C (110°F) by allowing the test cylinder to stand in air at room temperature, then place the cylinder in the bath maintained at 24 ± 0.5°C (75 ± 1°F). After the oil has reached bath temperature, insert a cleaned air-inlet tube and gas diffuser and proceed as described in 10.2, recording the foam value at the end of the blowing and settling periods.

10.5 Some lubricants with modern additives can pass their foam requirements when blended (with the antifoam properly dispersed in small particle sizes) but fail to meet the same requirements after two or more weeks' storage. (It appears that the polar dispersant additives have the potency to attract and hold antifoam particles, such that the apparent increased antifoam size results in decreased effectiveness to control foam in Test Method D 892.) However, if the same stored oil is merely decanted and poured into engines, transmissions, or gear boxes and those units operated for a few minutes, the oil again meets its foam targets. Similarly, *decanting* the stored oil into a blender, followed by agitation as described for Option A (see 10.5.1), redisperses the antifoam held in suspension and the oil again will give good foam control in Test Method D 892. For such oils, Option A can be used. On the other hand, if the antifoam is not dispersed into sufficiently small particles when the oil is blended, the oil cannot meet its foam requirements. If this freshly blended oil were vigorously stirred according to Option A, it is very possible that the oil would then meet its foam targets whereas the plant blend would never do so. Therefore, it is inappropriate and misleading to apply Option A for quality control of freshly made blends.

10.5.1 *Option A*—Clean the container of a 1-L (1-qt), high-speed blender[10] using the procedure given in 9.1.1. Place 500 mL of sample measured from 18 to 32°C (65 to 90°F) into the container, cover, and stir at maximum speed for 1 min. Because it is normal for considerable air to be entrained during this agitation, allow to stand until entrained bubbles have dispersed and the temperature of the oil has reached 24 ± 3°C (75 ± 5°F). Within 3 h following the agitation (solvents with equivalent cleaning and solvency characteristics may be substituted for toluene), start with testing as specified in 10.2.

NOTE 12—In case of viscous oils, 3 h can be insufficient time to disperse the entrained air. If a longer time is required, record the time as a note on the results.

11. Alternative Procedure

11.1 For routine testing a simplified testing procedure can be used. This procedure differs from the standard method in only one respect. The total air volume used during the 5-min blowing period is not measured after the air has passed through the gas diffuser. This eliminates the volume measuring equipment and the airtight connections necessary to carry the exit air from the graduated cylinder to the volume measuring device, but requires that the flowmeter be correctly calibrated and that the flow rate be carefully controlled. Results obtained by this procedure shall be reported as D 892 – IP 146 (Alternative).

12. Report

12.1 Report the data in the following manner:

Test	Foaming Tendency ASTM D 892 IP 146 Foam Volume, mL, at end of 5-min blowing period	Foam Stability ASTM D 892 IP 146 Foam Volume, mL, at end of 10-min settling period
As received:		
Sequence I	...	...
Sequence II	...	...
Sequence III	...	...
After agitation: (Option A, 9.5.1)		
Sequence I	...	...
Sequence II	...	...
Sequence III	...	...

12.2 For the purpose of reporting results, when the bubble layer fails to completely cover the oil surface and a patch or eye of clear fluid is visible, the value shall be reported as nil foam.

[10] Blender (Waring type) or equivalent.

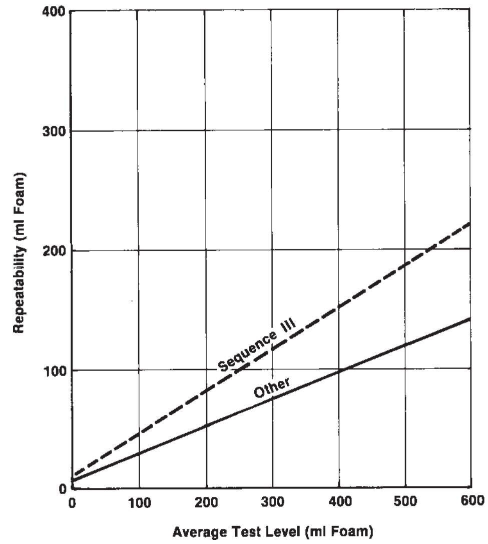


FIG. 3 Precision Chart—Repeatability

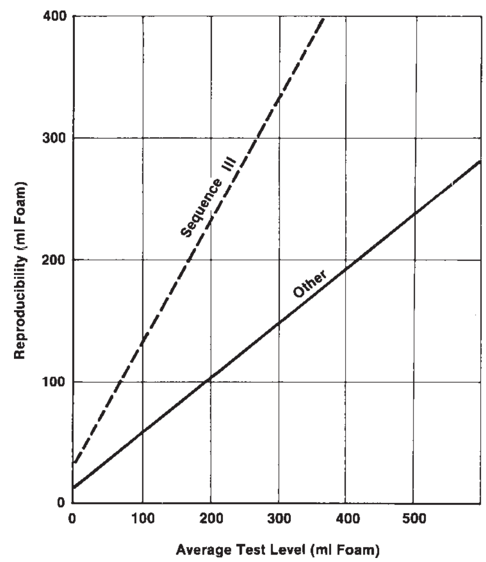


FIG. 4 Precision Chart—Reproducibility

13. Precision and Bias [11]

13.1 *Precision*—The precision values in this statement were determined in a cooperative laboratory program.[12]

13.1.1 *Repeatability*—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 the test method exceed the following values in only one case in twenty (see Fig. 3).

13.1.2 *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, exceed the following values in only one case in twenty (see Fig. 4).

[11] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1244.

[12] Supporting data has been filed at ASTM International Headquarters. Request RR: D02-1007.

NOTE 13—The dashed lines in Fig. 3 and Fig. 4 are for foam stability of Sequence III and the solid lines are for foam height for Sequences I, II, and III and foam stability for Sequences I and II.

13.1.3 For those oils which have been tested by Option A (10.5.1), no precision statement is yet available.

NOTE 14—The majority of the results in the cooperative work that led to Option A were nil foam; hence, no precision statement can be calculated.

13.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure for measuring foaming characteristics in Test Method D 892, bias cannot be determined.

14. Keywords

14.1 foam (foaming)

ANNEX

(Mandatory Information)

A1. TEST FOR MAXIMUM PORE DIAMETER AND PERMEABILITY OF GAS DIFFUSERS (BASED ON TEST METHOD E 128)

A1.1 Apparatus

A1.1.1 Apparatus for the maximum pore diameter determination consists of a regulated source of clean, dry, compressed air, a U-tube water manometer of sufficient length to read a pressure differential of 7.85 kPa (800 mm of water) and a cylinder of a size sufficient (250 mL is suitable) to conveniently immerse a gas diffuser to a depth of 100 mm (see Fig. A1.1).

A1.1.2 Additional apparatus for permeability determination consists of a gas volume metre of sufficient capacity to measure flow rates of at least 6000 mL/min while generating a back pressure of no more than 10 mm water. A filtering flask large enough that the 25.4-mm (1-in.) diameter diffuser will pass through the neck. This flask shall be fitted with a rubber stopper with a single hole to admit the air-inlet tube (see Fig. A1.2). A supply of tubing having an internal diameter of 8 mm (0.3 in.) shall be used to make the connections between the various parts of the apparatus as shown in Fig. A1.1 and Fig. A1.2.

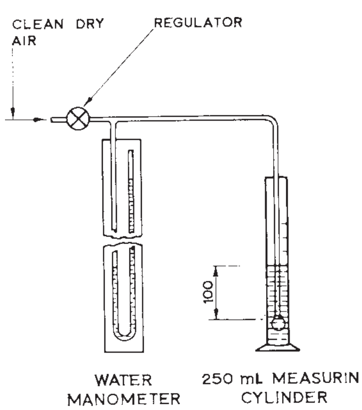


FIG. A1.1 Apparatus for Measuring Maximum Pore Size

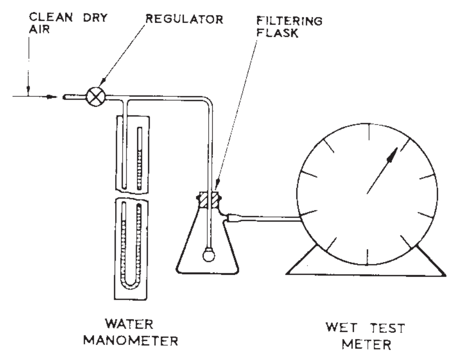


FIG. A1.2 Apparatus for Measuring Permeability

A1.2 Procedure

A1.2.1 *Maximum Pore Diameter*—Connect the diffuser to the manometer using an adaptor as shown in Fig. 2 (but without the brass tubing) and a 1.0-m length of 8-mm bore tubing. Support the clean diffuser to a depth of 100 mm, as measured to the top of the diffuser, in distilled water if the diffuser is non-metallic and propan-2-ol if the diffuser is metallic. Allow to soak for at least 2 min. Connect the air-inlet tube to a controllable source of clean, compressed air as shown in Fig. A1.1. Increase the air pressure at a rate of about 490 Pa (50 mm of water)/min until the first dynamic bubble passes through the filter and rises through the water. The first dynamic bubble is recognized by being followed by a succession of additional bubbles. Read the water level in both legs of the manometer and record the difference *p*. The uniformity of distribution of pores approaching maximum pore size may be observed by gradually increasing the air pressure and noting the uniformity with which streams of bubbles are distributed over the surface.

A1.2.1.1 Calculate the maximum pore diameter, *D*, in micrometres, as follows:

(*1*) For non-metallic diffusers and water as the diffuser medium:

$$D = 29\,225/(p - 100) \tag{A1.1}$$

where:

p = mm of water.

(*2*) For metallic diffusers and propan-2-ol as the diffuser medium:

$$D = 8930/(p - 80) \tag{A1.2}$$

where:

p = water in the manometer, mm.

A1.2.1.2 Calibration of diffusers have been found to be a critical factor in this test.[13]

A1.2.2 *Permeability*—Connect the clean, dry diffuser with a controllable source of clean, dry, compressed air, again using a 1-m length of 8-mm-bore tubing, and place it in a filtering flask connected to a suitable flowmeter using a further 0.5-m length of tubing as shown in Fig. A1.2. Adjust the pressure differential to 2.45 kPa (250 mm of water) and measure the rate of flow of air through the gas diffuser in millilitres per minute. Depending on the sensitivity of the flowmeter used, this observation may be made for a suitably longer period and the average flow rate per minute recorded.

[13] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1369.

APPENDIX

(Nonmandatory Information)

X1. HELPFUL HINTS IN OPERATION OF TEST METHOD D 892

X1.1 Helpful Hints

X1.1.1 The test should be performed exactly as described to obtain good results.

X1.1.2 Norton stone diffusers are known to be unreliable regarding their porosity and permeability; hence, new stones (as well as the metal diffusers) need to be checked in accordance with Annex A1.

X1.1.3 The diffusers should be checked periodically for porosity and permeability, depending upon the usage; checking is recommended at least once a week. Out of specification diffusers are a major cause of inaccuracy in this test method.

X1.1.4 The connection between the gas diffusers and the air inlet tubes should be airtight.

X1.1.5 The inlet air should be dried by passing through a desiccant drying tower. The indicator desiccant needs to be changed when it shows the presence of moisture by changing its color from blue to pink.

X1.1.6 Thermometer calibration should be checked at least annually against a master thermometer.

X1.1.7 Thorough cleaning of the test cylinder and the air inlet tube is essential after each use to remove any residual additive from the previous analysis.

X1.1.7.1 The cylinders are cleaned with heptane, a suitable detergent, distilled water, acetone, and dried with air or in an oven, in sequence.

X1.1.7.2 The gas diffusers are cleaned at least five times with toluene, heptane, and clean dry air in sequence.

X1.1.8 Oil or water baths must be used to control testing temperatures within 0.5°C (1°F).

X1.1.9 The total volume of the air passing through the system should be measured to 470 ± 25 mL. Without this step, there is no way of ascertaining that the system is airtight.

X1.1.10 It is recommended that the stopwatches be calibrated against a national standard at least once a year. Annex A3 (Timer Accuracy) of Test Method D 445 is a good source for guidance on how to check the timers for accuracy.

X1.1.11 If using Option A, all entrained air bubbles after stirring should be dispersed before testing.

X1.1.12 It is misleading and inappropriate to apply Option A for quality control of freshly made blends, or comparing/reporting Option A and regular foam test results.

X1.1.13 If the alternative procedure is used for measurements, the data should not be reported as that obtained by Test Method D 892.

SUMMARY OF CHANGES

Committee D02.06 has identified the location of selected changes to this standard since the last issue (D 892-02) that may impact the use of this standard.

(1) Updated 6.1.

Harris Testing Laboratories, Inc.

CERTIFICATE OF ANALYSIS

CLIENT:
Megatrol Inc.
9469 S 500 W
Sandy, UT 84070

PRODUCT: **FE**^{26} Premier
MARKS: NONE
DATE RECEIVED: 02/11/2008
LAB NO: HH0410-1202-P
SUBMITTED BY: Jon Rose

			SPECIFICATIONS	
METHOD	**TEST**	**RESULT**	**MIN.**	**MAX.**
D-2272	**Rotating Bomb Oxidation,** Minutes	**15**	**XXX**	**XXX**

Comments:

Amos Mwangi

CHEMIST

Date issued:
02/11/2008

Standard Test Method for Oxidation Stability of Steam Turbine Oils by Rotating Pressure Vessel[1]

This standard is issued under the fixed designation D 2272; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method[2] utilizes an oxygen-pressured vessel to evaluate the oxidation stability of new and in-service turbine oils having the same composition (base stock and additives) in the presence of water and a copper catalyst coil at 150°C.

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

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific warning statements, see 6.2, 6.4, 6.5, 6.6, 6.10, and 6.11.

2. Referenced Documents

2.1 *ASTM Standards:*

B 1 Specification for Hard-Drawn Copper Wire[3]

D 235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent)[4]

D 943 Test Method for Oxidation Characteristics of Inhibited Mineral Oils[5]

D 1193 Specification for Reagent Water[6]

D 2112 Test Method for Oxidation Stability of Inhibited Mineral Insulating Oil by Pressure Vessel[7]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[8]

D 4742 Test Method for Oxidation Stability of Gasoline Automotive Engine Oils by Thin-Film Oxygen Uptake (TFOUT)[8]

2.2 *British Standard:[9]*

B2 2000 Part 0: Section 0.1,

IP 37C Thermometer

2.3 *Institute of Petroleum Standard:[10]*

IP 229

3. Summary of Test Method

3.1 The test oil, water, and copper catalyst coil, contained in a covered glass container, are placed in a vessel equipped with a pressure gage. The vessel is charged with oxygen to a gage pressure of 620 kPa (90 psi, 6.2 bar) (see Eq 1), placed in a constant-temperature oil bath set at 150°C, and rotated axially at 100 rpm at an angel of 30° from the horizontal. The number of minutes required to reach a specific drop in gage pressure is the oxidation stability of the test sample.

$$100 \text{ kPa} = 1.00 \text{ bar} = 14.5 \text{ psi} \quad (1)$$

4. Significance and Use

4.1 The estimate of oxidation stability is useful in controlling the continuity of this property for batch acceptance of production lots having the same operation. It is not intended that this test method be a substitute for Test Method D 943or be used to compare the service lives of new oils of different compositions.

4.2 This test method is also used to assess the remaining oxidation test life of in-service oils.

NOTE 1—A modification of the rotating vessel method has been published as Test Method D 2112, which uses a similar procedure and apparatus but a lower (140°C) bath temperature. Test Method D 2112 requires duplicate testing and Test Method D 2272 conducted duplicate testing in the past.

5. Apparatus

5.1 *Oxidation Vessel, Glass Sample Container with Four-Hole PTFE Disk, Hold-Down Spring, Catalyst-Coil, Pressure*

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.09 on Oxidation.

Current edition approved August 10, 2002. Published October 2002. Originally published as D 2272-64 T. Last previous edition D 2272-98.

[2] von Fuchs, G. H., Claridge, E. L., and Zuidema, H. H., "The Rotary Bomb Oxidation Test for Inhibited Turbine Oils," *Materials Research and Standards*, MTRSA (formerly ASTM Bulletin), No. 186, December 1952, pp. 43-46; von Fuchs, G. H., "Rotary Bomb Oxidation Test," *Lubrication Engineering*, Vol 16, No. 1, January 1960, pp. 22-31.

[3] *Annual Book of ASTM Standards*, Vol 02.03.

[4] *Annual Book of ASTM Standards*, Vol 06.04.

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

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

[7] *Annual Book of ASTM Standards*, Vol 10.03.

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

[9] Available from British Standards Institute, 389 Chiswick High Rd., London, W4 4AL, United Kingdom.

[10] Available from Institute of Petroleum, 61 New Cavendish St., London, W1G 7AR, United Kingdom.

Gage, Thermometer, and Test Bath, as described in Annex A1. The assembled apparatus is shown schematically in Fig. 1 and Fig. A1.6.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[11] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Isopropyl Alcohol*, reagent grade. (**Warning—**Flammable. Health hazard.)

6.3 *Liquid Detergent*.

6.4 *n-Heptane*, 99.0 minimum mol % (pure grade). (**Warning—**Flammable. Health hazard.)

6.5 *Oxygen*, 99.5 %, with pressure regulation to 620 kPa (90 psi, 6.2 bar). (**Warning—**Vigorously accelerates combustion.)

6.6 *Potassium Hydroxide, Alcohol Solution (1 %)*—Dissolve 12 g of potassium hydroxide (KOH) pellets in 1 L of the isopropyl alcohol. (**Warning—**Flammable. Health hazard.)

6.7 *Silicone Carbide Abrasive Cloth*, 100-grit with cloth backing.

6.8 *Silicone Stopcock Grease*.

6.9 *Wire Catalyst, Electrolytic Copper Wire*, 1.63 ± 1 % mm (0.064 ± 1 % in.) in diameter (No. 16 Imperial Standard Wire Gage or No. 14 American Wire Gage, 99.9 % purity, conforming to Specification B 1. Soft copper wire of an equivalent grade may also be used.

6.10 *Petroleum Spirit*, conforming to Specification D 235 for petroleum spirit (mineral spirits). (**Warning—**Combustible. Health hazard.)

6.11 *Acetone*, reagent grade. (**Warning—**Flammable. Health hazard.)

6.12 *Reagent Water*, conforming to Specification D 1193, Type II.

7. Sampling

7.1 Samples for this test method can come from tanks, drums, small containers, or even operating equipment. Therefore, use the applicable apparatus and techniques described in Practice D 4057.

8. Preparation of Apparatus

8.1 *Catalyst Preparation*—Before use, polish approximately 3 m of the copper wire with a silicon carbide abrasive cloth and wipe free from abrasives with a clean, dry cloth. Wind the wire into a coil having an outside diameter 44 to 48 mm and weight of 55.6 ± 0.3 g and stretched to a height of 40 to 42 mm. Clean the coil thoroughly with isopropyl alcohol, air-dry, and insert inside the glass sample container by a turning motion, if necessary. A new coil is used for each sample. For extended storage, the prepared coil may be packaged in a dry, inert atmosphere. For overnight storage (less than 24 h), the coils may be stored in *n*-Heptane.

NOTE 2—Commercially available and prepackaged coils prepared as described in 8.1 can also be used for the test.[12]

8.2 *Cleaning of Vessel*—Wash the vessel body, cap, and inside of vessel stem with hot detergent solution and rinse thoroughly with water. Rinse the inside of the stem with

[11] *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.

[12] Prepackaged coils were provided for Spring 1995 round robin.

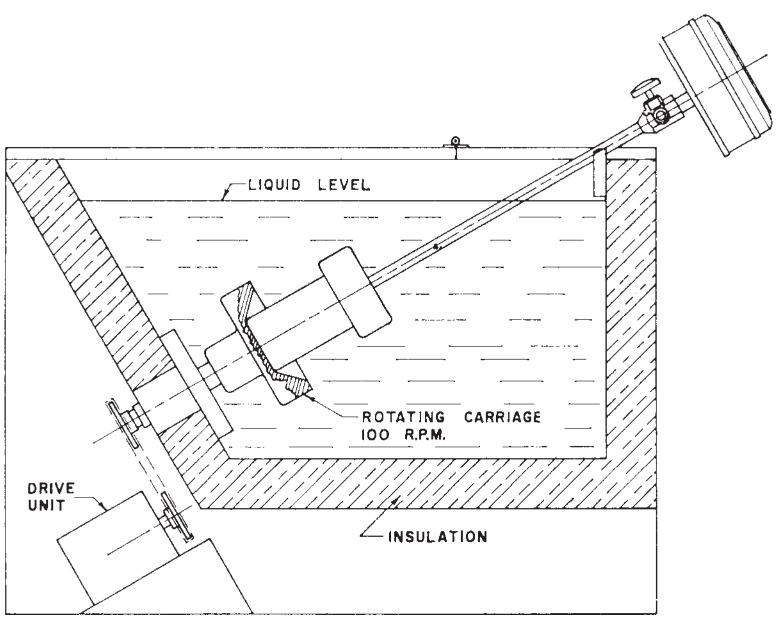


FIG. 1 Schematic Drawing of the Rotary Vessel Test Apparatus

isopropyl alcohol and blow dry with clean compressed air. If the vessel body, cap, or inside of the stem smells sour after simple cleaning, wash with 1 % alcoholic KOH solution and repeat as before. (**Warning—**Failure to remove oxidation residue can adversely affect test results.)

8.3 *Cleaning of Glass Container*—Drain and rinse with a suitable solvent (for example, petroleum spirit or acetone). Soak or scrub in an aqueous detergent solution. Brush thoroughly and flush thoroughly with tap water. Rinse with isopropyl alcohol, followed by distilled water and air dry. If any insolubles remain, soak overnight in an acid-type cleaning solution and repeat the above procedure starting from the tap water flush.

8.4 *Cleaning of Polytetrafluoroethylene (PTFE) Disk*—Remove any residual oil with a suitable solvent and clean by brushing with detergent solution. Rinse thoroughly with tap water, followed by distilled water rinse and air dry.

9. Procedure

9.1 *Charging*—Weigh the glass sample container with a freshly cleaned catalyst coil. Weigh 50 ± 0.5 g of oil sample into the container; also add 5 mL of reagent water. Add another 5 mL of reagent water to the vessel body and slide the sample container into the vessel body (see Note 3). Cover the glass container with a 57.2–mm (2 ¼–in.) PTFE disk and place a hold-down spring[13] on top of the PTFE disk. Apply a thin coating of silicone stopcock grease to the O-ring vessel seal located in the gasket groove of the vessel cap to provide lubrication, and insert the cap into the vessel body.

NOTE 3—The water between the vessel wall and the sample container aids heat transfer.

9.1.1 Tighten the closure ring by hand. Cover the threads of the gage-nipple with a thin coating of stopcock grease (PTFE pipe tape is a suitable alternative to the use of stopcock grease) and screw the gage into the top center of the vessel stem. Attach the oxygen line with an inline pressure gage to the inlet valve on the vessel stem. Slowly turn on the oxygen supply valve until the pressure has reached 620 kPa (90 psi, 6.2 bar). Turn off the oxygen supply valve. Slowly release pressure by loosening the fitting or by using an inline bleeder valve. Repeat purging process two more times; purge step should take approximately 3 min. Adjust the regulating valve on the oxygen supply tank to 620 kPa (90 psi, 6.2 bar) at a room temperature of 25°C (77°F). For each 2.0°C (3.6°F) above or below this temperature, 5 kPa (0.7 psi, 0.05 bar) shall be added or subtracted to attain the required initial pressure. Fill the vessel to this required pressure and close the inlet valve securely by hand. If desired, test the vessel for leaks by immersing in water (see Note 4).

NOTE 4—If the vessel was immersed in water to check for leaks, dry the outside of the wet vessel by any convenient means such as airblast or a towel. Such drying is advisable to prevent subsequent introduction of free water into the hot oil bath which would cause sputtering.

9.2 *Oxidation*—Bring the heating bath to the test temperature while the stirrer is in operation. Switch off stirrer, insert the vessel into the carriages, and note the time. Restart the stirrer. If an auxiliary heater is used, keep it on for the first 5 min of the run and then turn it off (see Note 5). The bath temperature shall stabilize at the test temperature within 15 min after the vessel is inserted. Maintain the test temperature within ± 0.1°C (see Note 6).

NOTE 5—The time for the bath to reach the operating temperature after insertion of the vessel may differ for different apparatus assemblies and should be observed for each unit. The objective is to find a set of conditions that does not permit a drop of more than 2°C after insertion of the vessel and allows the vessel pressure to reach a plateau within 30 min as shown in Curve A of Fig. 2.

NOTE 6—Maintaining the correct temperature within the specified limits of ± 0.1°C during the entire test run is an important factor assuring both repeatability and reproducibility of test results.

[13] PTFE disk with 4-holes and hold down spring were provided for Spring 1995 round robin.

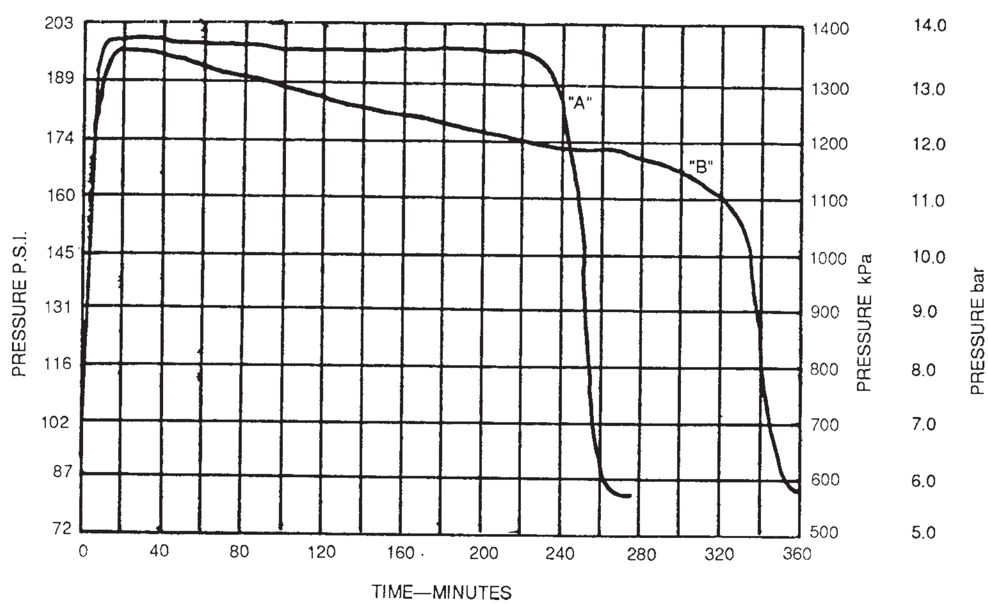


FIG. 2 Pressure Versus Times Plot of Two Rotary Vessel Oxidation Test Runs

9.3 Keep the vessel completely submerged and maintain continuous and uniform rotation throughout the test. A standard rotational speed of 100 ± 5 rpm is required; any appreciable variations in this speed could cause erratic results.

9.4 The test is complete after the pressure drops more than 175 kPa (25.4 psi, 1.75 bar) below the maximum pressure (see Note 7). The 175 kPa pressure drop usually, but not always, coincides with an induction-type *period of rapid pressure drop*. When it does not, the operator may question whether he has produced a valid experiment (see Note 8).

NOTE 7—While termination of the test at a 175 kPa (25.4 psi, 1.75 bar) pressure drop is the standard procedure, some operators may elect to stop the test at lesser pressure drops or to observe the condition of the oil after a predetermined test period of perhaps 100 min; that is, well within the normal induction period of new inhibited oils.

NOTE 8—A typical experiment is shown in Fig. 2 as Curve A. The maximum pressure is expected to be reached within 30 min, a pressure plateau is established, and an induction-type pressure drop is observed. Curve B, in which there is a gradual decrease in pressure before the induction break is recorded, is more difficult to evaluate. The gradual decrease in pressure could be due to a vessel leak, although some synthetic fluids will generate this type of curve. If a leak is suspected, repeat the test in a different vessel. If the same type of curve is derived when the test is repeated, the experiment is likely valid.

9.5 After termination of the test, the vessel shall be removed from the oil bath and cooled to room temperature. The vessel can be briefly dipped into and swirled around in a bath of light mineral oil to wash off the adhering bath oil. The vessel is rinsed off with hot water, then immersed into cold water to quickly bring it to room temperature. Alternately, the vessel can be cooled to room temperature in air. The excess oxygen pressure is bled off and the vessel opened.

10. Report

10.1 *Interpretation of Results*:

10.1.1 In reference to Fig. 2, Curve A, observe the plot of the recorded pressure versus time and establish the plateau (see Note 8). Record the time at the point on the falling part of the curve where the pressure is 175 kPa (25.4 psi, 1.75 bar) less than the established plateau pressure. If the test is repeated, the plateau pressures in repeat tests should not differ by more than 35 kPa (5.1 psi, 0.35 bar).

10.1.2 In reference to Fig. 2, Curve B, observe the plot of the recorded pressure versus time and establish the maximum pressure obtained during the initial 30 min of the experiment (see Note 8). Record the time on the falling part of the curve where the pressure is 175 kPa (25.4 psi, 1.75 bar) less than the established maximum pressure. If the test is repeated, maximum pressures in repeat tests should not differ by more than 35 kPa (5.1 psi, 0.35 bar).

10.2 *Report the Results*:

10.2.1 In reference to Fig. 2, Curve A, the vessel life of the sample is the time in minutes from the start of the test to a 175 kPa (25.4 psi, 1.75 bar) pressure drop from the level of the established plateau.

10.2.2 In reference to Fig. 2, Curve B, the vessel life of the sample is the time in minutes from the start of the test to a 175 kPa (25.4 psi, 1.75 bar) pressure drop from the maximum pressure.

NOTE 9—In reporting test results, it is recommended that it be indicated whether tests were made with stainless steel or chrome-plate copper vessels.

11. Precision and Bias [14]

11.1 The precision and bias statement is generated from the research report (95 % confidence). The data range of results in RR:D02-1409 is from 30 to 1000 min.

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

$$0.12\,X \tag{2}$$

where:
X = denotes mean value.

11.1.2 *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:

$$0.22\,X \tag{3}$$

where:
X = denotes mean value.

NOTE 10—This precision statement was prepared with data on seven oils (an uninhibited base oil and three new and three used steam turbine oils) tested by eleven cooperators. The oils covered values in the ranges from 30 to 1000 min. Oils with results greater than 1000 min exhibited poor precision in the Spring 1995 round robin.

11.2 *Bias*—There being no criteria for measuring bias in these test-product combinations, no statement of bias can be made.

[14] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1409. Supporting data are also provided in Supplement I.

ANNEX

(Mandatory Information)

A1. APPARATUS FOR ROTARY PRESSURE VESSEL OXIDATION TEST

A1.1 *Oxidation Vessel*, with body, cap, closure ring, and stem, constructed as shown in Figs. A1.1-A1.4.

A1.1.1 *Vessel Body and Cap*, shall be constructed of 18-8 or 321S12/321S20 Part 1 (BSI) stainless steel to ensure a proper rate of heat transfer. The interior surface shall be given a smooth finish to facilitate cleaning. Alternatively, the vessel body and cap may be machined from 76.2-mm (3-in.) solid copper rod and then heavily chrome plated.

A1.1.2 *Vessel Stem*, shall be constructed of stainless steel, the stem having an inside diameter of 6.4 mm (¼ in.) and shall be equipped with a 6.4-mm (¼-in.) needle valve.

A1.1.3 *Closure Ring*, shall be made of chrome-plated steel or chrome-plated aluminum bronze BS 2032.

A1.1.4 The vessel shall withstand a working pressure of 3450 kPa (500 psi, 34.5 bar) at 150°C.

A1.1.5 *O-ring Gaskets*, Viton or silicon, 50.8 mm (2 in.) in inside diameter by 60.3 mm (2⅜ in.) in outside diameter (BS/USA size No. 329). Caps with larger seal recess diameters will require 54 mm (2⅛ in.) inside diameter by 60.3 mm (2⅜ in.) in outside diameter (BS/USA size No. 227).

A1.2 *Glass Sample Container*, with copper catalyst coil, 175-mL capacity as shown in Fig. A1.5, constructed of borosilicate glass. Glass sample container shall have a sliding fit in the vessel with no excess side clearance. The container alone shall have a maximum wall thickness of 2.5 mm and shall weigh no more than 100 g.

A1.2.1 *Top of Sample Container*, shall be covered with 57.2-mm (2¼-in.) diameter PTFE disk. The disk will have four 3.2-mm (⅛-in.) diameter holes evenly spaced in a 9.5-mm (⅜-in.) radius from the center of the disk. The disk shall have a thickness of 1.6 mm (1⁄16 in.). A stainless steel hold-down spring as shown in Fig. A1.6 shall be used to ensure rotation of the sample container. The assembly is shown in Fig. A1.7.

A1.3 *Recording Devices*:

A1.3.1 *Recording Gage*[15], as shown in Fig. A1.8 or indicating, with a range from 0 to 1400 kPa (or 0 to 200 psi or 0 to 14 bar) and graduated in 25-kPa (or 5 psi or 0.25 bar) divisions. The accuracy shall be 2.5 % or less of the total scale interval. Recording gages should be mounted so that the face is perpendicular to the axis of rotation.

A1.3.2 *Pressure Measurement System*, consisting of electronic pressure transducers, power source, mounting equipment and connecting cables. The rotary transducer couplings can be mounted directly on the vessel stem in place of the standard mechanical pressure recorders. The pressure transducer shall have a span of 0 to 1400 kPa (or 0 to 200 psi or 0 to 14 bar). The accuracy should be valid over a wide compensated temperature range. The output signal from the transducer can be channeled into a datalogger, microprocessor based recorder, or a computer for data acquisition. The data acquisition package should be capable of logging pressure data vessel time. The overall system accuracy of the data should be within 2.5 % of the total scale.

A1.4 *Oxidation Bath*, equipped with an efficient stirrer and a suitable device from holding and rotating the vessel axially at an angle of 30° at 100 ± 5 rpm while submerged in oil to a point at least 25 mm (1 in.) below the level of the bath liquid.

A1.4.1 A bath at least 230 mm (9 in.) deep, filled with 30 L (8 gal) of heavy bath oil per vessel, has the proper heat capacity. Silicone oil shall be necessary to house the oil bath under a fume hood to contain any oil vapor generated.

A1.4.2 Provide thermal regulation to maintain the bath within 0.1°C of the test temperature. There should be sufficient, immediately available heat to bring the bath to operating temperature within 15 min after the vessels have been inserted.

A1.5 *Thermometer*, IP 37C sludge test thermometer having a range from 144 to 156°C graduated in 0.2°C intervals or other temperature measuring device, having an accuracy of 0.1°C.

A1.6 *Gage*, for pressurizing vessel to 620 kPa (90 psi) graduated in 1.5 kPa (0.2 psi) increments.[15]

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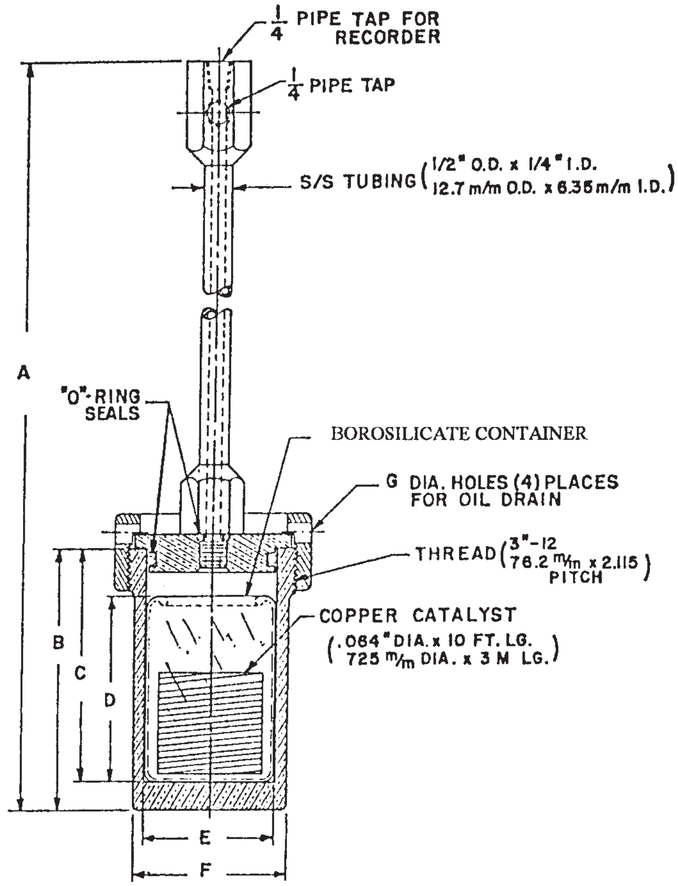
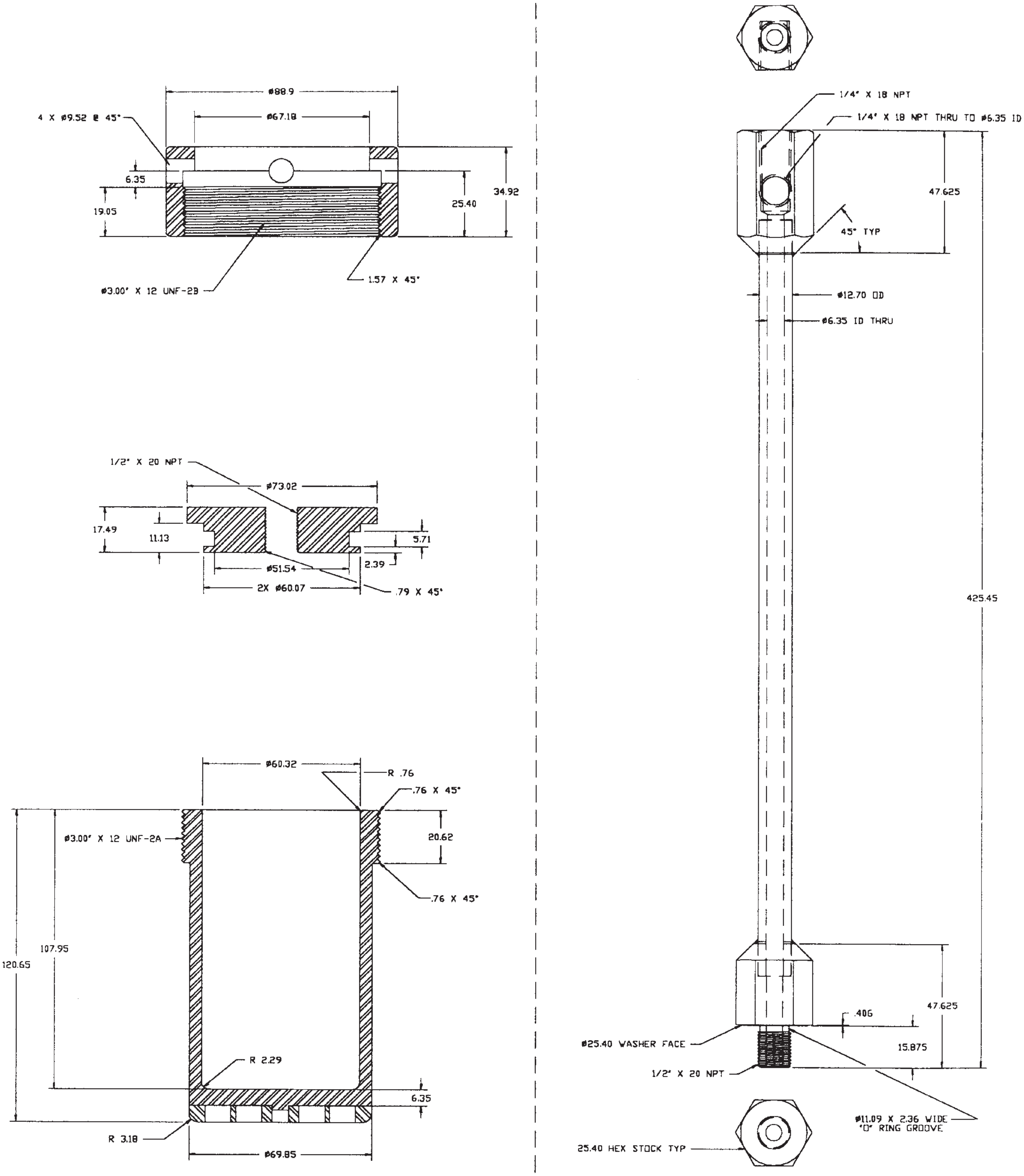
[15] The sole source of supply of the Heise gage, Model CM known to the committee at this time is Dresser Industries, 153 South Main St., Newtown, CT 06470. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee [1], which you may attend.



	Inches	Millimeters
A	21 1/8	536.58
B	4 3/4	120.65
C	4 1/4	107.95
D	3 3/8 to 3 1/2	86 to 89
E	2.375 {+ 0.010 − 0.000}	60.325/60.579
F	2 3/4	69.85
G	3/8	9.525

FIG. A1.1 Oxidation Vessel



Unless otherwise specified all dimensions in millimeters.

NOTE—The vessel shown in Figs. A1.1 and A1.2 can also be used for Test Method D 4742 (TFOUT). Test Method D 2272 and IP 229 utilize different drive mechanisms for the vessel; hence, US and UK vessels/baths are not interchangeable.

FIG. A1.2 Construction of Oxidation Vessel

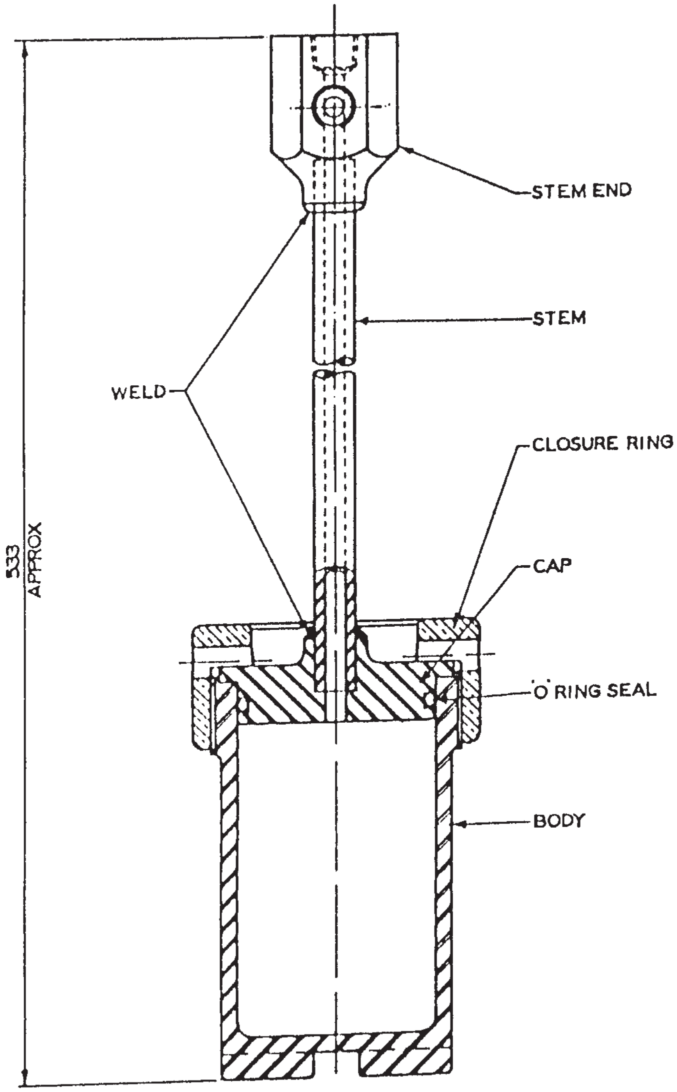
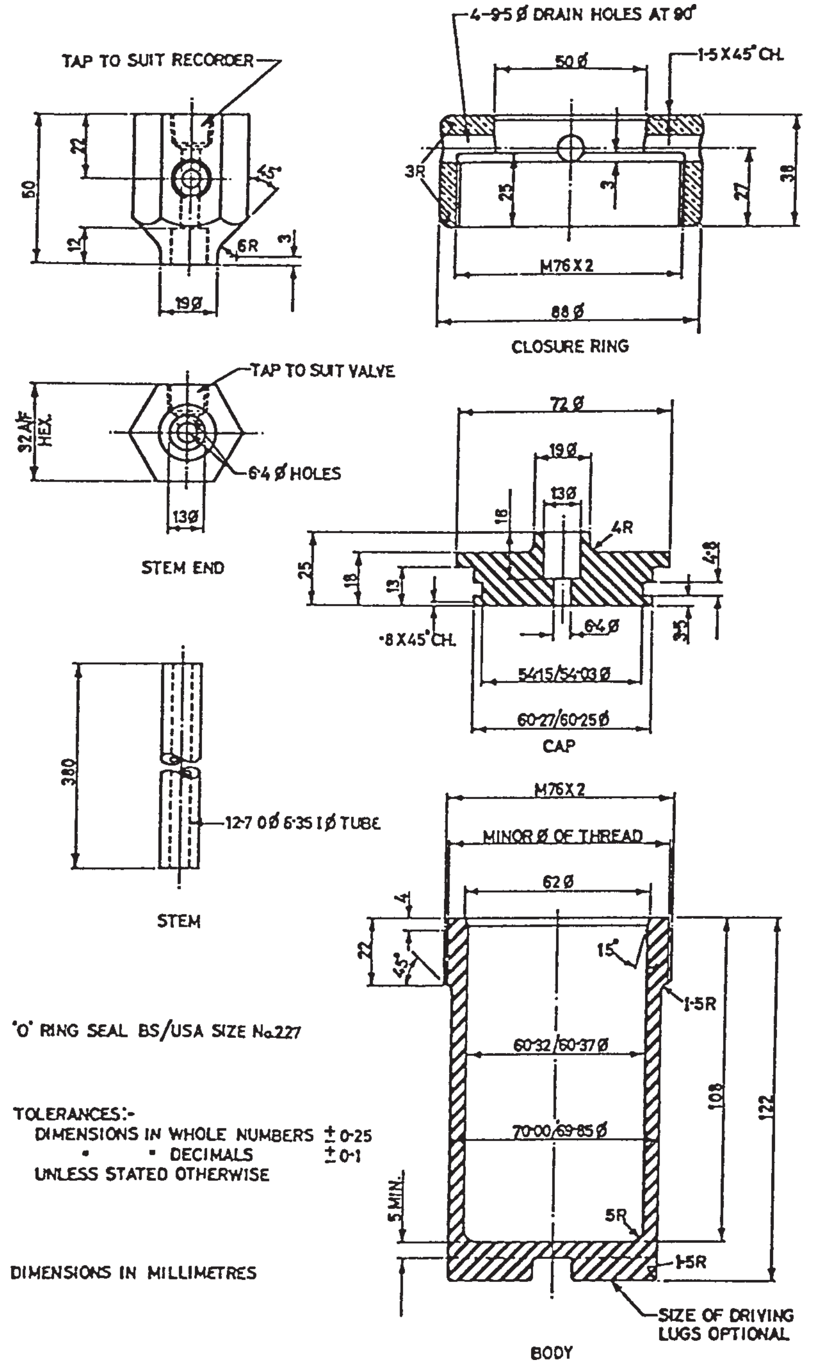


FIG. A1.3 Oxidation Vessel



NOTE—The vessel shown in Figs. A1.3 and A1.4 is not applicable for Test Method D 4742.

FIG. A1.4 Details of Oxidation Vessel

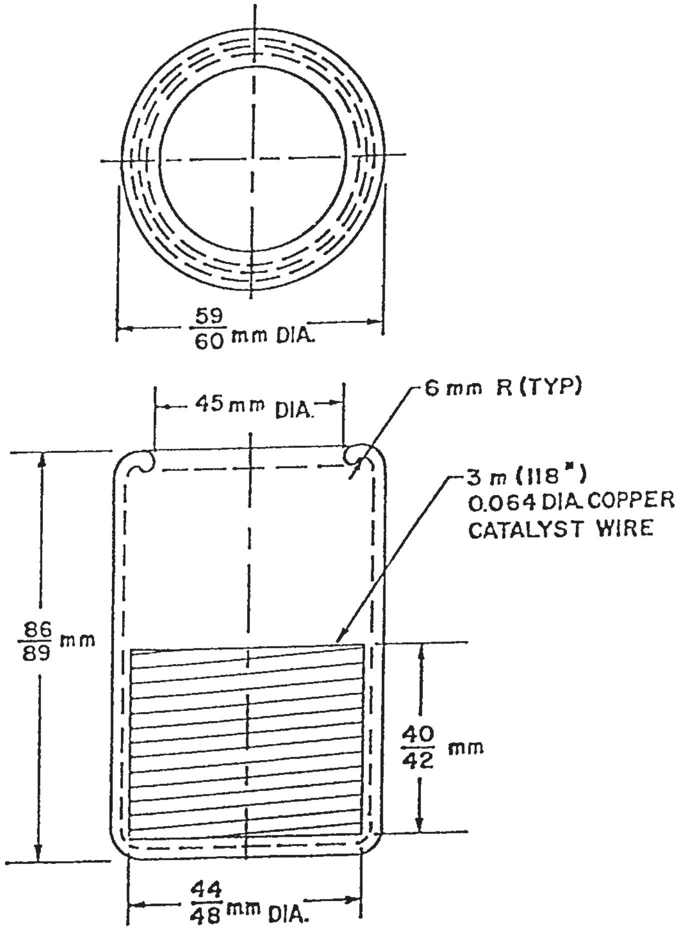


FIG. A1.5 Borosilicate Glass Sample Container

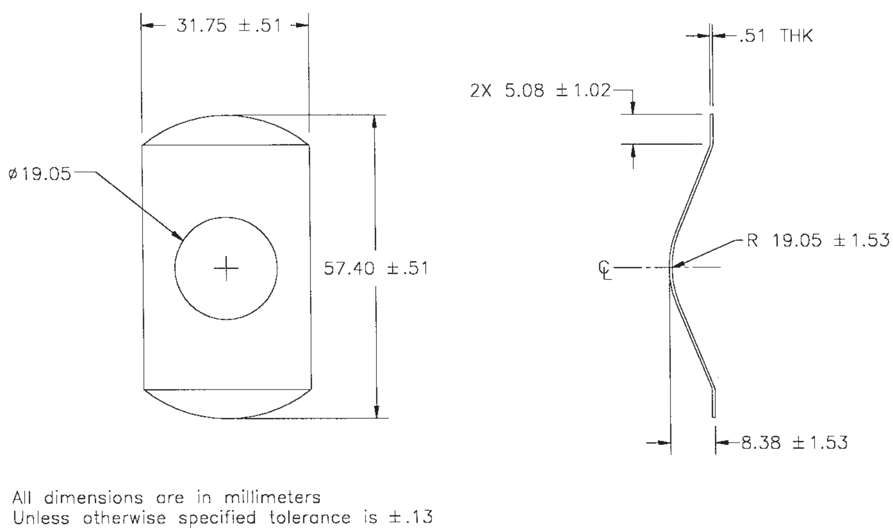


FIG. A1.6 Hold-down Spring

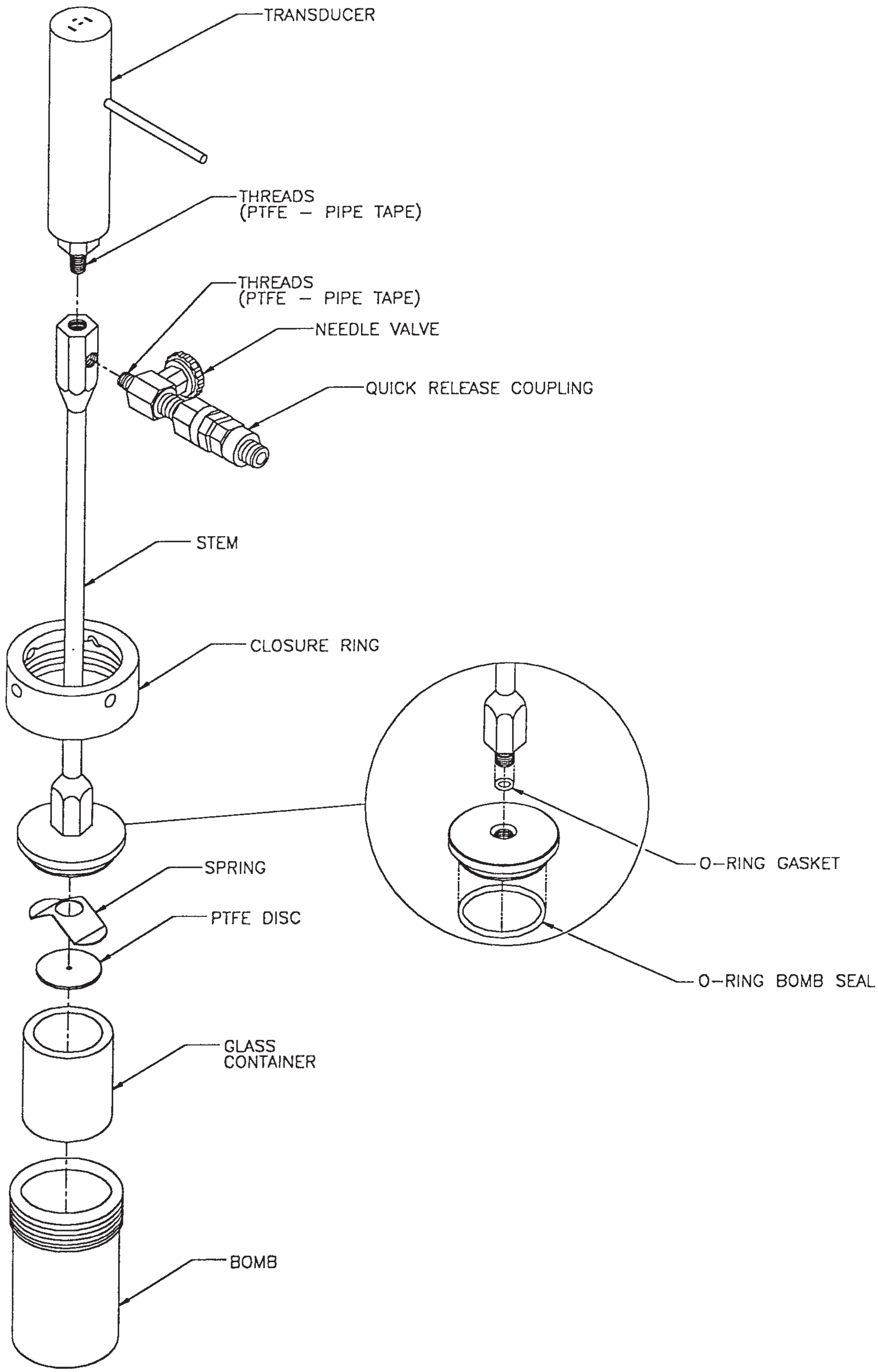


FIG. A1.7 Oxidation Vessel Assembly

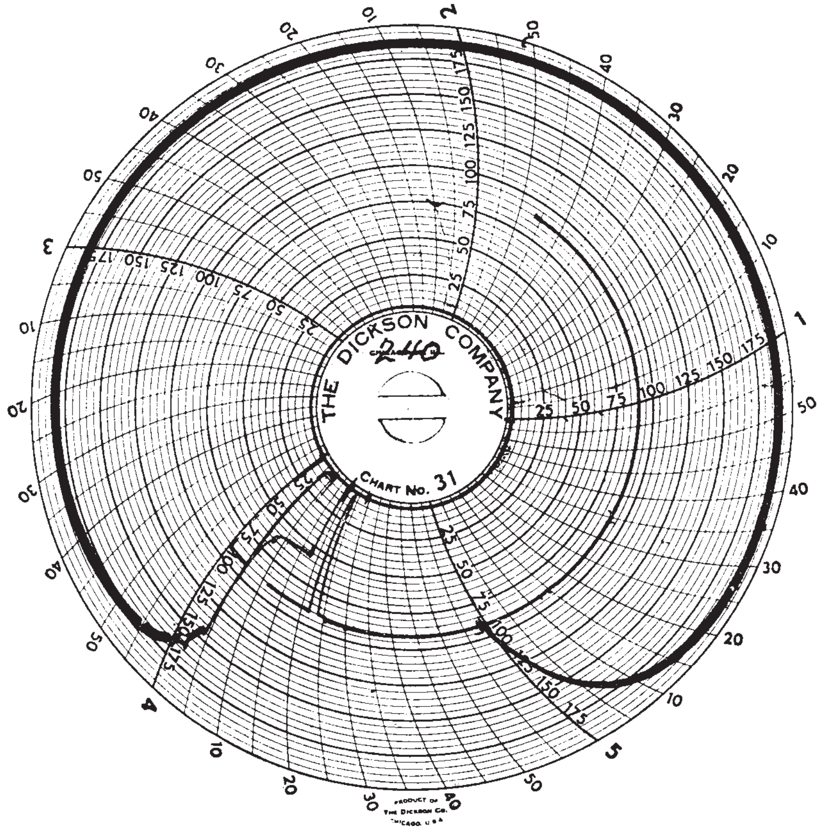


FIG. A1.8 Chart of Recording Pressure Gage (Actual Size = 4½ in.)

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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 International 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, at the address shown below.

Harris Testing Laboratories, Inc.

CERTIFICATE OF ANALYSIS

CLIENT
Megatrol Inc.
9469 S 500 W
Sandy, UT 84070

PRODUCT: FE[26] Premier
MARKS: NONE
DATE RECEIVED: 02/11/2008
LAB NO: HH0410-1202-P
SUBMITTED BY: Jon Rose

METHOD	TEST	RESULT
D-3233	Falex Pin & V-Block 4,500 lb Gauge Pre-Heat to 120 ºF	Unable to attain load load 4,500 lb. load

Load, lbs		Starting Torque		Final Torque		Comments
300	lbs @ 3 min	5	lb - in	7	lb - in	
500	lbs @ 1 min	10	lb - in	10	lb - in	
750	lbs @ 1 min	13	lb - in	13	lb - in	
1,000	lbs @ 1 min	16	lb - in	16	lb - in	
1,250	lbs @ 1 min	20	lb - in	20	lb - in	
1,500	lbs @ 1 min	22	lb - in	23	lb - in	
1,750	lbs @ 1 min	28	lb - in	29	lb - in	
2,000	lbs @ 1 min	30	lb - in	31	lb - in	
2,250	lbs @ 1 min	32	lb - in	33	lb - in	
2,500	lbs @ 1 min	34	lb - in	35	lb - in	
2,750	lbs @ 1 min	36	lb - in	36	lb - in	very slight loss of load
3,000	lbs @ 1 min	38	lb - in	37	lb - in	very slight loss of load
3,250	lbs @ 1 min	42	lb - in	41	lb - in	
3,500	lbs @ 1 min	46	lb - in	45	lb - in	
3,750	lbs @ 1 min	49	lb - in	52	lb - in	slight loss of load
4,000	lbs @ 1 min	54	lb - in	56	lb - in	slight loss of load
4,250	lbs @ 1 min	57	lb - in	57	lb - in	loss of load

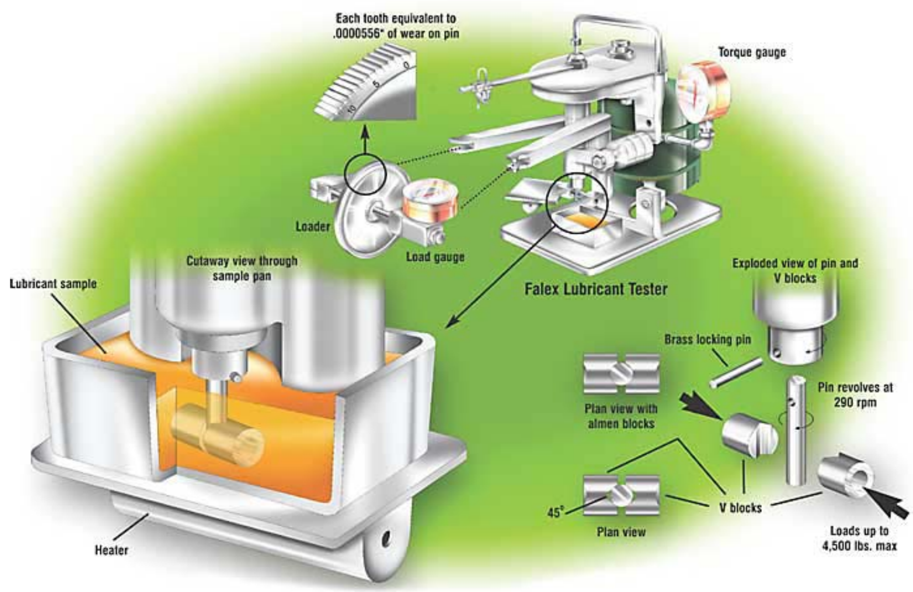
Comments:

Amos Mwangi

CHEMIST

Date issued:
02/11/2008

ASTM D3233 Test Procedures



Falex Continuous Load ASTM D3233

The Falex Continuous Load Test ASTM D3233 is used to measure the load-carrying properties of fluid-based lubricants.

This test method has been found to have some direct correlation to field service conditions, where high-load conditions are encountered. The test utilizes two procedures to evaluate the industrial gear lubricant's load-carrying properties and its ability to protect against scuffing. Procedure A is known as the Falex Run-Up Test Method, while Procedure B is referred to as the Falex One-Step Test Method.

Both test procedures consist of running a rotating steel pin at 290 rpm driven by a 1/3 horsepower motor against two stationary V-blocks that are immersed in 60 ml of the industrial gear lubricant being tested. The load applied to the V-blocks against the rotating pin is applied through the use of a ratchet mechanism. In Procedure A, increasing loads starting at 300 pounds of force (lbf) (1,334.4 newtons) are applied continuously. In Procedure B, the loads are applied at a starting point of 250 lbf (1,112 newtons) with the load being maintained for one-minute intervals at increasing loads. In both test methods, the test ends when the industrial gear lubricant being tested can no longer support the applied load, resulting in either breakage of the pin or seizure of the pin to the V-blocks. The highest load that can be applied in this test is 4,500 lbf (20,016 newtons). An industrial gear lubricant that exhibits a 1,500 lbf (6,672 newtons) is considered to provide sufficient protection against gear wear and scuffing under heavily loaded conditions.

An American National Standard

Standard Test Methods for Measurement of Extreme Pressure Properties of Fluid Lubricants (Falex Pin and Vee Block Methods)[1]

This standard is issued under the fixed designation D 3233; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover two procedures for making a preliminary evaluation of the load-carrying properties of fluid lubricants by means of the Falex Pin and Vee Block Test Machine.

NOTE 1—Additional information can be found in Appendix X1 regarding coefficient of friction, load gage conversions, and load gage cablibration curve.

1.2 The values stated in SI units are to be regarded as standard. Because the equipment used in these test methods is available only in inch-pound units, the SI units are omitted when referring to the equipment and the test specimens.

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

2. Referenced Documents

2.1 *ASTM Standards:*

B 16/B 16M Specification for Free-Cutting Brass, Rod, Bar and Shapes for Use in Screw Machines[2]

D 2670 Test Method for Measuring Wear Properties of Fluid Lubricants (Falex Pen and Vee Block Method)[3]

D 2783 Test Method for Measurement of Extreme-Pressure Properties of Lubricating Fluids (Four-Ball Method)[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *actual gage load*, *n*—the value obtained from the gage while running the test and before any corrections are made.

3.1.1.1 *Discussion*—This gage reading is irrespective of the particular gage used, and corrections are made by comparison to a standard reference.

3.1.2 *direct load*, *n*—that which is applied linearly, bisecting the angle of the vee block corrected to either the 800 or 3000-lbf gage reference.

3.1.2.1 *Discussion*—This load is equivalent to the true load times the cos 42°.

3.1.3 *true load*, *n*—the sum of the applied forces normal to the tangents of contact between the faces of one vee block and the journal pin corrected to the 4500 lbf gage reference line.

3.1.4 *true load failure value*, *n*—the true load at which the lubricant tested can no longer support the applied load resulting in either test pin or shear pin breakage, or inability to maintain or increase load.

3.1.4.1 *Discussion*—This value is also referred to as the limit of extreme pressure.

4. Summary of Test Methods

4.1 Both test methods consist of running a rotating steel journal at 290 ± 10 rpm against two stationary V-blocks immersed in the lubricant sample. Load is applied to the V-blocks by a ratchet mechanism. In Test Method A (Note 1), increasing load is applied continuously. In Test Method B (Note 1), load is applied in 250-lbf (1112-N) increments with load maintained constant for 1 min at each load increment. In both methods the load-fail value obtained is the criteria for level of load-carrying properties. Both methods require calibration of the load gage and reporting of test results as true (corrected) loads rather than actual gage loads.

NOTE 2—Test Method A is referred to as the Falex Run-Up Test. Test Method B is referred to as the Falex One-Minute Step Test.

5. Significance and Use

5.1 Evaluations by both test methods differentiate between fluids having low, medium, and high levels of extreme-pressure properties. The user should establish any correlation between results by either method and service performance.

NOTE 3—Relative ratings by both test methods on the reference fluids covered in Table 1 and Table 2 are in good general agreement with

[1] These test methods are under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.L0 on Industrial Lubricants.

Current edition approved Aug. 10, 2003. Published September 2003. Originally approved in 1986. Last previous edition approved in 1998 as D 3233–93(1998).

[2] *Annual Book of ASTM Standards*, Vol 02.01.

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

TABLE 1 Results of Cooperative Tests on Reference Fluids L-XI-1-2-A, B, C, D, E[A] (TEST METHOD A)

Laboratory	Test	L-XI-1-2-A Fail Load, lbf		L-XI-1-2-B Fail Load, lbf		L-XI-1-2-C Fail Load, lbf		L-XI-1-2-D Fail Load, lbf		L-XI-1-2-E Fail Load, lbf	
		Gage	True	Gage	True	Gage	True	Gage	True	Gage	True
A	1	1200	840	1200	840	4500 +	4100 +	4300	3950	2600	2100
	2	1275	920	1275	920	4500 +	4100 +	4500 +	4100 +	2400	1925
B	1	800	860	1000	1050	4500 +	4250 +	4100	3900	2050	2050
	2	850	900	950	1025	4500 +	4250 +	4300	4100	1950	1950
C	1	725	990	775	1020	4500 +	3200 +	3950	2900	1350	1460
	2	650	910	750	980	4500 +	3200 +	4100	3000	1300	1430
D	1	1400	1050	1100	770	4500 +	3500 +	4500 +	3500 +	2900	2150
	2	1400	1050	1250	900	4500 +	3500 +	4500 +	3500 +	2650	1975
E	1	825	900	1000	1060	4450	4500 +	4100	4475	1825	1970
	2	750	820	925	1000	4450	4500 +	4150	4500	1825	1970
F	1	1000[B]	920[B]	1000	800	4500 +	4500 +	3500	4500	1850	1900
	2	990[B]	910[B]	1050	850	4500 +	4500 +	2900	3510	1720	1720
G	1	800	900	690	800	4000	4275	3325	3625	1430	1600
	2	700	800	660	750	3750	4000	3150	3450	1500	1675
H	1	700	700	1000	1000	4500 +	4500 +	3750	3750	1900	1900
	2	700	700	1000	1000	4500 +	4500 +	4000	4000	1650	1650
I	1	750	600	1250	1000	4500 +	3750 +	4500 +	3750 +	1750	1450
	2	750	600	1000	800	4500 +	3750 +	4500 +	3750 +	1750	1450
Min Avg			600		775				2950[C]		1445
Max Avg			1050		1037				4488[C]		2063
Grand Avg			854		920				3809[C]		1796
		Repeatability $s = 0.0624$ $r = 0.179$ (TL)[D]						Reproducibility $S = 0.140$ $R = 0.402$ (TL)[D]			

[A] Reference fluids used and described in Test Method D 2783.
[B] Calibration curves shifted.
[C] Six laboratories.
[D] TL = average true load, lbf, of sample tested.

four-ball weld-point relative ratings obtained on these same reference fluids, covered in Test Method D 2783.

6. Apparatus

6.1 *Falex Pin and Vee Block Test Machine,*[4] illustrated in Fig. 1, Fig. 2, and Fig. 3, fitted with 4500-lbf (20 000-N) gage or 3000-lbf (13 350-N) gage.

6.2 *Required for Calibration*:

6.2.1 *Allen Screw*, with attached 10-mm Brinnell ball.[5]

6.2.2 *Back-Up Plug.*[5]

6.2.3 *Standard Test Coupon,*[5] soft, annealed copper, Hb 37–39.

6.2.4 *Brinnell Microscope*, or equivalent.

6.2.5 *Timer*, graduated in seconds and minutes.

6.2.6 *Rule*, steel, 6-in. (approximately 150-mm) long.

7. Reagents and Materials

7.1 *Standard Coined-Blocks,*[5] 96 ± 1° angle, AISI C-1137 steel, HRC 20 to 24, surface finish 5 to 10 µin. (1.3×10^{-7} to 2.5×10^{-7}m), rms.

7.2 *Standard Test Journals,*[5] ¼ in. (6.35 mm) outside diameter by 1¼ in. (31.75 mm) long, AISI 3135 steel, HRB 87 to 91 on a ground flat surface, surface finish 5 to 10 µin. (1.3×10^{-7} to 2.5×10^{-7} m) rms.

7.3 *Locking Pins,*[5] ½ H brass, conforming to Specification B 16/B 16M.

7.4 *Solvent*, safe, nonfilming, nonchlorinated.

NOTE 4—Petroleum distillate and benzene, formerly used as solvents in this method, have been eliminated due to possible toxic effects. Each user should select a solvent that can meet applicable safety standards and still thoroughly clean the parts.

8. Preparation of Apparatus

8.1 *Cleaning*:

8.1.1 Thoroughly clean the V-blocks, test journals, lubricant cup, and supports for V-blocks and test journals by washing, successively, with solvent selected in 7.4. Dry the V-blocks, test journals, lubricant cup, and supports by allowing the final solvent to evaporate in air.

[4] The Falex Pin and Vee Block Test Machine, available from the Falex Corp., 1020 Airpark Dr., Sugar Grove, IL 60554 has been found satisfactory for this purpose. A new model of this machine has been available since 1983. Certain operating procedures are different for this new model. Consult instruction manual of machine for this information.

[5] Available from Falex Corp., 1020 Airpark Dr., Sugar Grove, IL 60554.

TABLE 2 Results of Cooperative Tests on Reference Fluids L-XI-1-2-A, B, C, D, E[A] (TEST METHOD B)

Labora-tory	Test	L-XI-1-2-A Fail Load, lbf		L-XI-1-2-B Fail Load, lbf		L-XI-1-2-C Fail Load, lbf		L-XI-1-2-D Fail Load, lbf		L-XI-1-2-E Fail Load, lbf	
		Gage	True	Gage	True	Gage	True	Gage	True	Gage	True
A	1	1100	750	1400	1000	4150	3750	4350	4000	2750	2250
	2	1100	750	1400	1000	4350	4000	4150	3750	2200	1750
B	1	670	750	940	1000	4200	4000	3900	3750	2000	2000
	2	670	750	670	750	3900	3750	4200	4000	1750	1750
C	1	520	750	520	750	4100 +	3000 +	4100	3000	1750	1750
	2	520	750	790	1000	4100 +	3000 +	4100 +	3000 +	1750	1750
D	1	1600	1250	1080	750	4500 +	3500 +	4500 +	3500 +	3000	2250
	2	1600	1250	1080	750	4500 +	3500 +	4500 +	3500 +	3300	2500
E	1	700	750	925	1000	3850	4250	3850	4250	1380	1500
	2	700	750	925	1000	4150	4500	3650	4000	1850	2000
F	1	1075[B]	1000[B]	950	750	3350	4250	3350	4250	1925	2000
	2	1075[B]	1000[B]	950	750	3500	4500	3050	3750	1560	1500
G	1	660	750	660	750	3500	3750	3000	3250	1550	1750
	2	660	750	800	1000	3200	3500	2800	3000	1350	1500
H	1	750	750	1000	1000	3500	3500	4250	4250	1500	1500
	2	750	750	1000	1000	4000	4000	4000	4000	1750	1750
I	1	930	750	910	750	4400	3750	4400 +	3750 +	1800	1500
	2	930	750	910	750	4400	3750	4400 +	3750 +	1800	1500
Min Avg			750		750		3625[C]		3125[D]		1500
Max Avg			1250		1000		4375[C]		4125[D]		2375
Grand Avg			833		875		3932[C]		3837[D]		1846

Repeatability	Reproducibility
$s = 0.0624$	$S = 0.137$
$r = 0.179$ (TL)[E]	$R = 0.391$ (TL)[E]

[A] Reference fluids used and described in Test Method D 2783.
[B] Calibration curves shifted.
[C] Seven laboratories.
[D] Six laboratories.
[E] TL = average true load, lbf, of sample tested.

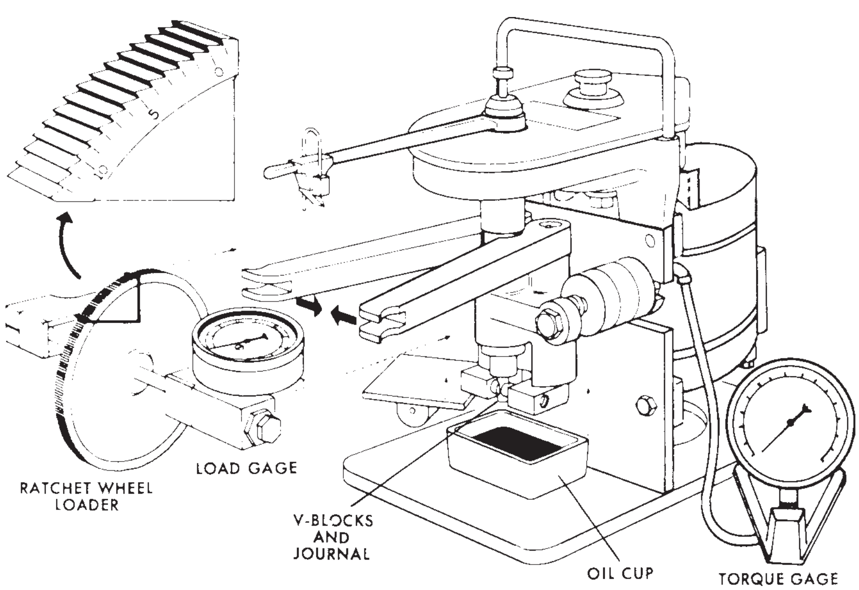


FIG. 1 Schematic Diagram of Falex Standard Pin and Vee Block Test Machine

FIG. 2 Falex Digital Pin and Vee Block Test Machine

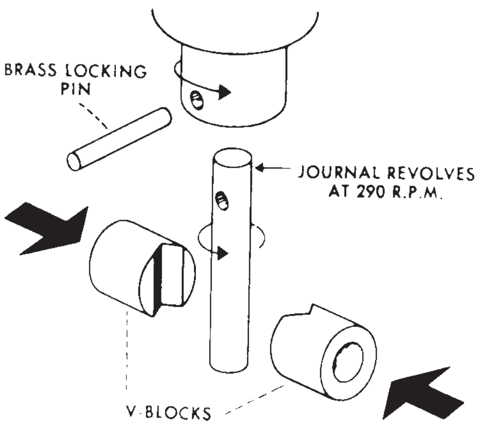


FIG. 3 Exploded View of V-Blocks and Journal Arrangement, Falex Pin and Vee Block Test Machines

8.1.2 After cleaning, handle the test pieces with care to prevent contamination. Particularly, avoid contact of fingers with mating surfaces of V-blocks and test journals.

8.2 *Assembly*:

8.2.1 Insert the test journal into the test shaft and secure with a new brass locking pin, as shown in Fig. 1 and Fig. 3.

8.2.2 Insert the V-blocks into the recesses of the loading device and swing the V-blocks inward to contact the journal so that the V-grooves are aligned with the journal major axis, as shown in Fig. 1 and Fig. 3.

8.2.3 Place 60 mL of test lubricant in the lubricant cup and raise the cup so that the V-blocks are immersed in the test lubricant. With highly viscous fluids, open the jaws slightly to ensure that the wear surfaces are covered with the lubricant.

8.2.4 Place the automatic loading device, with attached gage, on the jaw arms.

9. Preparation of True Load Calibration Curve

9.1 On log-log paper (K & E 467080 or equivalent) draw a straight-line plot of load, pounds-force (newtons) (ordinate), versus indentation diameter, millimetres (abscissa) using the data points shown below. Label this curve "True Load" (Note 5).

Load, lbf (N) (Ordinate)	Diameter, mm (Abscissa)
500 (2224)	2.62
1000 (4450)	3.42
1500 (6672)	4.00
2000 (8896)	4.47

NOTE 5—Fig. 3 shows the true-load calibration curve for the prescribed 4500-lbf (20 000-N) gage, prepared as covered in 9.1. Copies of Fig. 4, 8 by 11 in., are available at a nominal cost from ASTM. Although not originally used in development of these test methods, the 3000-lb direct reading load gage should be satisfactory providing results are corrected and reported with respect to the true load (4500-lbf) reference line. Refer to Test Method D 2670 for calibration of 3000-lb load gage.

10. Calibration of Load Gage 4500 lbf (20 000 N)

10.1 Remove the Allen set screw and ½-in. (12.70-mm) ball from the left jaw socket (Fig. 5).

10.2 Insert the special Allen screw with the attached 10-mm Brinnell ball into the working face of the left jaw. Adjust so that the ball projects about 5⁄32 in. (approximately 4 mm) from the face of the jaw.

10.3 Insert the back-up plug in the counterbore of the right-hand jaw. Adjust so that the plug projects about 1⁄32 in. (approximately 0.8 mm) from the face.

10.4 Support the standard test coupon so that the upper edge of the coupon is about 3⁄32 in. (approximately 2.5 mm) below the upper surface of the jaws. Place a steel rule across the face of the jaws. Adjust the Allen screw with the attached 10-mm ball until the face of the jaws are parallel to the steel rule with the test coupon in position for indentation.

10.5 With the test coupon in position for the first impression, place the load gage assembly on the level arms. Remove the slack from the assembly by moving the ratchet wheel by hand.

10.6 Place the loading lever on the ratchet wheel and actuate the motor. Allow the motor to run until the load gage indicates a load of 500 lbf (2224 N). A slight take-up on the ratchet wheel is required to hold the load due to the ball sinking into the test coupon. After a 500-lbf (2224-N) load is obtained, hold for 1 min for the indentation to form.

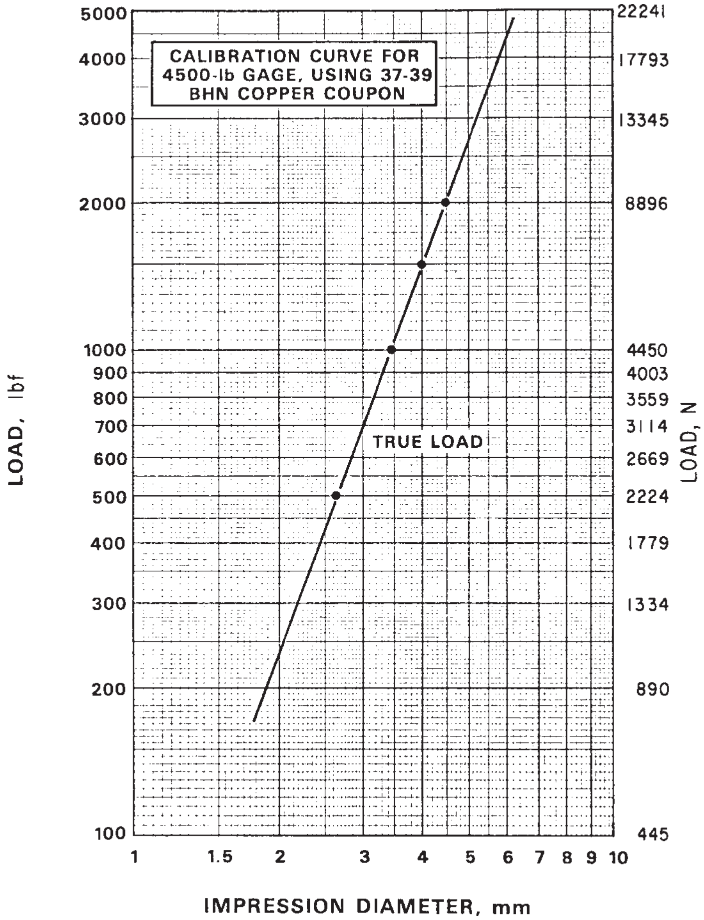


FIG. 4 Calibration Curve for 4500-lb Gage, Using 37-39 HB Copper Coupon

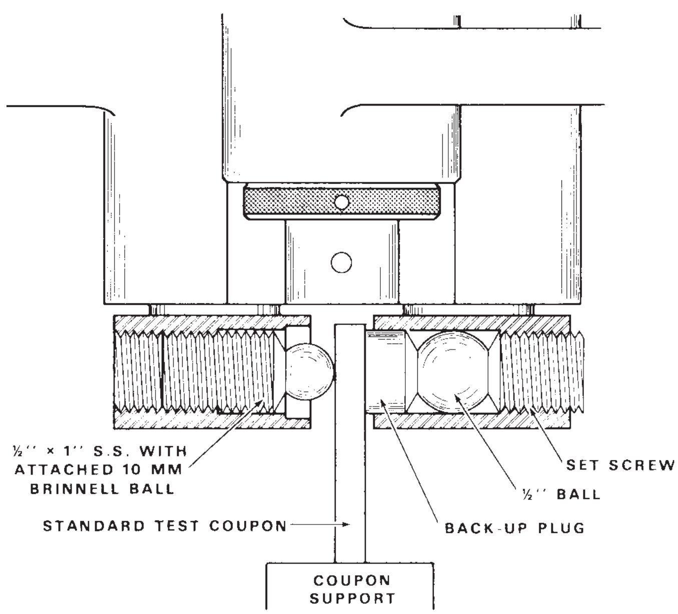


FIG. 5 Schematic Drawing of Calibration Accessories for Falex Pin and Vee Block Test Machines

10.7 Turn off the machine and back off the load until the test coupon is free of the jaws. Advance the test coupon approximately ⅜ in. (approximately 9.5 mm). Additional indentations should be separated by a minimum distance of 2.5 times the diameter of the initial indentation. Check the alignment of the jaws, and repeat the procedure described in 10.6 at gage loads of 1000, 1500, and 2000 lbf (4448, 6672, and 8896 N).

10.8 Remove the load gage assembly and test coupon and measure the diameter of each indentation to 0.01 mm with a microscope. Make three measurements of the indentation diameter, rotating the test coupon to ensure that no two measurements represent the same points. Average the three measurements of each impression and record.

10.9 Plot the four impression readings on the same log-log plot of true load prepared as prescribed in 9.1 and shown as Fig. 4. Draw a straight line through the four impression readings and label the line "Actual Gage Load."

NOTE 6—Currently, load gages are calibrated at the factory such that the actual 4500-lb gage load is equivalent to true load. Periodic calibrations should be made to ensure correct values are being reported for true load.

TEST METHOD A

11. Determination of Actual Gage Load for Run-In

11.1 The procedure, Section 12, requires a run-in at an actual gage load equivalent to 300-lbf (1334-N) true load (264-lbf direct load). This actual gage load is obtained as follows from the plot of actual gage load and true load prepared in Sections 9 and 10: Locate 300 lbf (1334 N) on the true load curve (264-lbf direct load). Through this point draw a vertical line to intersect the actual gage load curve. Through this point of intersection draw a horizontal line to the left-hand or right-hand load scale and read the actual gage load value. Record this actual gage load for run-in on a suitable reporting form.

NOTE 7—A suitable reporting form for Test Methods A and B, and data obtained on one of the reference fluids by one of the cooperating laboratories, is shown in Table 3. Fig. 6 shows the calibration curves used by the laboratory reporting the data in Table 3.

12. Procedure

12.1 *Run-In*:

12.1.1 Turn on "Heat Control" switch and heat test lubricant to 120 ± 5°F (51.7 ± 3°C); then turn off the switch.

12.1.2 Remove slack from assembly by moving the ratchet wheel by hand. At this setting the torque gage should read zero, or be adjusted to zero.

12.1.3 Actuate the motor, engage the automatic loading ratchet, and increase the load to a gage load equivalent to 300-lbf (1334-N) true load (264-lbf direct load), as determined in 8.1. Disengage the loading ratchet, start the timer, and allow the machine to run at this loading for a 5-min run-in period.

NOTE 8—Maintain load at near constant by taking up the load manually or automatically by means of the ratchet wheel if necessary.

12.2 *Test*:

12.2.1 Re-engage the automatic loading ratchet and leave it engaged until failure (Note 9) or until the highest indicated actual gage reading is reached. Stop the motor at failure or at the highest indicated actual gage load when no failure is obtained. Record the gage load at failure. Record 4500 lbf (20 000 N) if no failure is obtained.

NOTE 9—Failure is indicated by (*a*) breakage of shear pin or test pin, or (*b*) inability to take up the load automatically by means of the ratchet wheel.

12.2.2 Using the calibration curves prepared in Sections 9 and 10, determine and record the true load failure equivalent to the actual gage load failure, or, if no failure, the true load equivalent to the highest indicated actual gage load, with a plus (+) sign after the true load value.

NOTE 10—To convert actual gage load to true load, locate the gage load on the actual gage load curve. Through this point draw a vertical line to intersect the true load curve. Through this point of intersection draw a horizontal line to the left-hand or right-hand load scale and read the true load value.

TEST METHOD B

13. Determination of Actual Gage Load for Run-In and 250-lbf (1112-N) True Load Increments

13.1 The procedure, Section 14, requires a run-in at an actual gage load equivalent to 300-lbf (1334-N) true load (264-lbf direct load), and testing at incremental gage loadings equivalent to 250-lbf (1112-N) true load (224-lbf direct load) over the range from 500 to 4500-lbf (2224 to 20 000-N) true load (412 to 2885-lbf direct load). Determine the equivalent actual gage loads as prescribed in Section 8, and Test Method A. Record on a suitable reporting form, such as shown in Table 3.

TABLE 3 Suggested Report Form, Test Methods A and B, Showing Data

Operator: Laboratory A
Test Sample: L-XI-1-2-E
Gage Type:_______
Calibration Indentation Results: 500 lbf = 2.13 mm; 1000 lbf = 2.90 mm; 1500 lbf = 3.53 mm; 2000 lbf = 4.06 mm

TEST METHOD A
Run-In: 5 min at 520 lbf actual gage load, equivalent to 300 lbf true load.

	Actual Gate Load, lbf, at Failure	Equivalent True Load, lbf at Failure
Test No. 1	2600	2100
Test No. 2	2400	1925

TEST METHOD B
Run-In: 5 min at 520 lbf actual gage load, equivalent to 300 lbf true load.

True Load, lbf	Equivalent Actual Gage Load, lbf	Test No. 1	Test No. 2
500	800	pass	pass
750	1100	pass	pass
1000	1400	pass	pass
1250	1650	pass	pass
1500	1950	pass	pass
1750	2200	pass	fail
2000	2450	pass	
2250	2750	fail	
2500	3000		
2750	3200		
3000	3400		
3250	3700		
3500	3900		
3750	4150		
4000	4350		
4250			
4500			

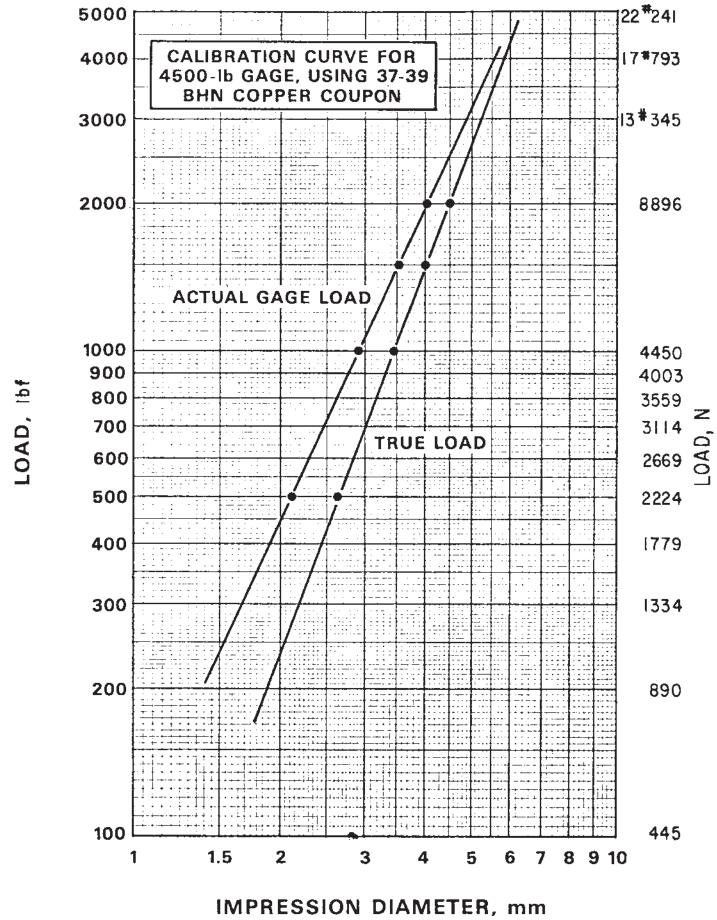


FIG. 6 Calibration Curves Used by Laboratory Reporting Data Shown in Table 1

14. Procedure

14.1 *Run-In*—Use the same procedure as prescribed in 12.1, Test Method A.

14.2 *Test*:

14.2.1 Re-engage the automatic loading ratchet and leave it engaged until the actual gage reading is equivalent to 500-lbf (2224-N) true load (412-lbf direct load), as determined in Section 13. Run for 1 min at this loading.

NOTE 11—Maintain load at near constant at this load and at subsequent incremental loading by taking up the load by means of the ratchet wheel when necessary.

NOTE 12—Keep the motor running after run-in and throughout subsequent loadings.

14.2.2 Increase actual gage loads in the increments equivalent to 250-lbf (1112-N) true load (224-lbf direct load), as determined in Section 13, running for 1 min at each increment loading. Record the load at which failure occurs (Note 14, Note 15). If no failure is obtained, record the last load run with a plus (+) sign after the value.

NOTE 13—Failure is indicated by (*a*) breakage of shear pin or test pin, or (*b*) inability to increase or maintain load by means of the ratchet wheel.

NOTE 14—If failure occurs during run-up between load increments, record the higher increment load as fail.

14.2.3 Determine and record the true load failure equivalent as described in 12.2.2.

TEST METHODS A AND B

15. Report

15.1 Report the true load value at which failure occurred.

15.2 If no failure is obtained, report the last true load run, with a plus (+) sign after the value.

16. Precision and Bias

16.1 The precision of these test methods as determined by statistical examination of interlaboratory results is as follows (see Note 15):

16.1.1 *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:

Test Method A, 27 % of the mean
Test Method B, 24 % of the mean

16.1.2 *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:

Test Method A, 40 % of the mean
Test Method B, 43 % of the mean

16.2 *Bias*—The procedure in this test method has no bias because the value of the Falex Extreme Pressure Failure Load can be defined only in terms of a test method.

NOTE 15—The precision data were derived from results of cooperative tests on L-XI-1-2-A, B, C, D, and E, covered in Table 1 and Table 2. These are the same reference fluids used and described in Test Method D 2783.

17. Keywords

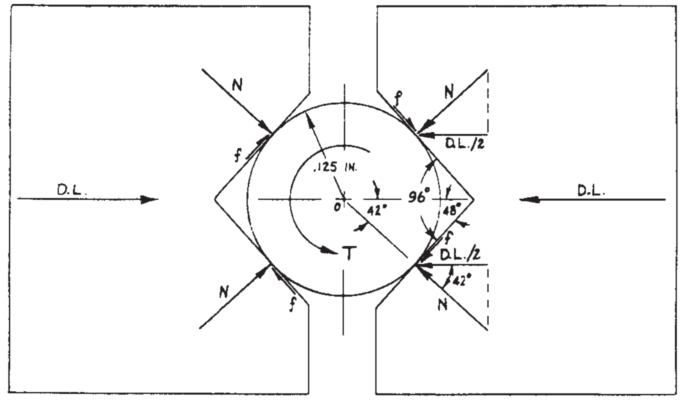
17.1 extreme pressure; Falex Pin and Vee Block; load-carrying; lubricant; wear

APPENDIX

(Nonmandatory Information)

X1. COEFFICIENT OF FRICTION CALCULATION AND LOAD GAGE CONVERSIONS

X1.1 See Figs. X1.1-X1.3.



D.L. = DIRECT LOAD, LBS.
T = TORQUE, IN. LBS.
N = NORMAL LOAD PER FACE, LBS.
u = COEFFICIENT OF FRICTION
M_o = SUMMATION OF MOMENTS ABOUT POINT "O"
f = FRICTION FORCE

$M_o = 0 = T\text{-}(4F \times 0.125)$
$f = T/(4 \times 0.125)$
$f = 2T$

$D.L./2 = N \times COS\ 42°$
$N = D.L./(2 \times COS\ 42°)$
$N = 0.672816\ D.L.$

$u = f/N = 2T/(0.672816 \times D.L.)$
$u = 2.9726\ T/D.L.$

FIG. X1.1 Falex #0 Pin and Vee Block Coefficient of Friction Calculation

(From Load Gage Calibration Curve 12/15/68)

FROM 800# OR 3000# GAGES TO 4500# GAGE

Direct Load 800# or 3000# Gage Reading	Equivalent Load on 4500# Reference Gage	Contact Load (Normal) per Vee Block Face	Brinell Impression Diameter (mm)
200	220	134.56	1.92
250	283	168.20	2.12
300	350	201.85	2.28
400	482	269.13	2.58
500	620	336.41	2.85
600	765	403.69	3.09
700	900	470.97	3.29
705	910	474.34	3.30
750	975	504.61	3.39
800	1050	538.25	3.49
1000	1355	672.82	3.85
1100	1500	740.10	4.00
1250	1750	841.02	4.24
1500	2140	1009.22	4.59
1700	2465	1143.79	4.84
1750	2540	1177.43	4.90
2000	2970	1345.63	5.20
2250	3400	1513.84	5.48
2500	3825	1682.04	5.73
2750	4280	1850.25	5.98
3000	4700	2018.45	6.20

4500# Reference Gage Reading	Equivalent Load on 800# or 3000# Gage	Contact Load (Normal) per Vee Block Face	Brinell Impression Diameter (mm)
250	224	150.71	2.01
300	264	177.62	2.16
350	300	201.85	2.28
500	412	277.20	2.62
750	590	396.96	3.06
910	705	474.34	3.30
1000	765	514.71	3.42
1250	930	625.72	3.73
1500	1100	740.10	4.00
1750	1250	841.02	4.24
2000	1410	948.67	4.47
2250	1555	1046.23	4.68
2500	1720	1157.24	4.88
2750	1870	1258.17	5.04
3000	2015	1355.73	5.22
3250	2155	1449.92	5.39
3500	2315	1557.57	5.53
3750	2450	1648.40	5.69
4000	2600	1749.32	5.82
4250	2740	1843.52	5.96
4500	2885	1941.08	6.09

FIG. X1.2 Contact Load (Normal) per Vee Block Face and Typical Falex Load Gage Conversions

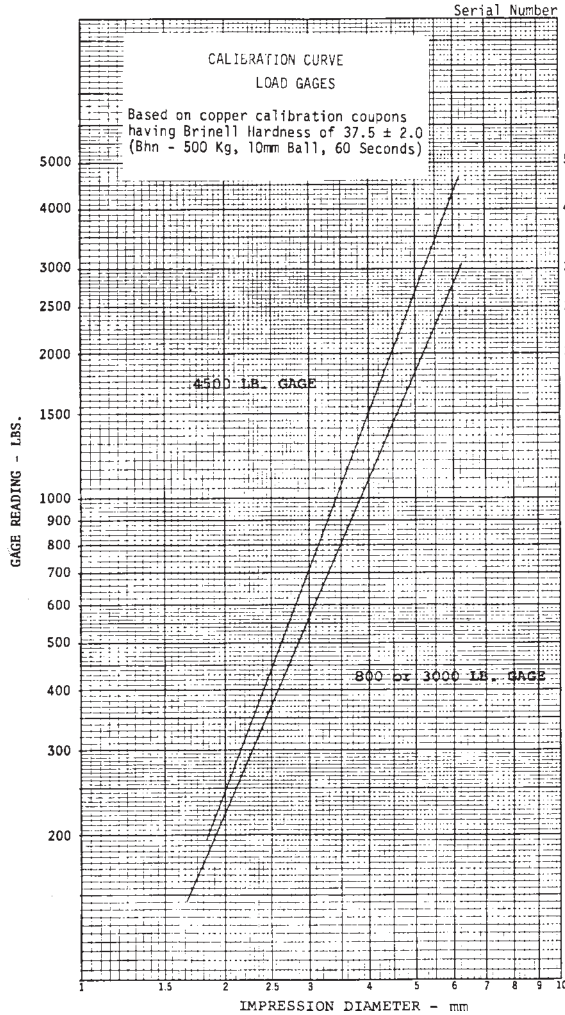


FIG. X1.3 Calibration Curve Falex Load Gages

Harris Testing Laboratories, Inc.

CERTIFICATE OF ANALYSIS

CLIENT:
Megatrol Inc.
9469 S 500 W
Sandy, UT 84070

PRODUCT: **FE**^{26} Premier
MARKS: NONE
DATE RECEIVED: 02/11/2008
LAB NO: HH0410-1202-P
SUBMITTED BY: Jon Rose

			SPECIFICATIONS	
METHOD	**TEST**	**RESULT**	**MIN.**	**MAX.**
D-4172	**Four Ball-Wear Test, mm**	**0.64**	**XXX**	**XXX**
		XXX	**XXX**	**XXX**

Comments: **See attached testing procedures for this product. Test was performed at lower RPM range 1250 rpm, not 1850 rpm.**

Amos Mwangi

Date issued:
02/11/2008

CHEMIST

Designation: D 4172 – 94 (Reapproved 1999)

An American National Standard

Standard Test Method for Wear Preventive Characteristics of Lubricating Fluid (Four-Ball Method)[1]

This standard is issued under the fixed designation D 4172; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers a procedure for making a preliminary evaluation of the anti-wear properties of fluid lubricants in sliding contact by means of the Four-Ball Wear Test Machine. Evaluation of lubricating grease using the same machine is detailed in Test Method D 2266.

1.2 The values stated in either inch-pound units or SI units are to be regarded separately as standard. Within the test the inch-pound units are shown in brackets. The values stated in each system are not exact equivalents, therefore each system must be used independently of the other. Combining values of the two systems may result in nonconformance with the specification.

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

2. Referenced Documents

2.1 *ASTM Standards:*

D 2266 Test Method for Wear Preventive Characteristics of Lubricating Grease (Four-Ball Method)[2]

2.2 *ANSI Standard:*

B3.12 Specification for Metal Balls[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *lubricant*, *n*—any material interposed between two surfaces that reduces the friction or wear between them.

3.1.2 *wear*, *n*—damage to a solid surface, generally involving progressive loss of material due to relative motion between that surface and a contacting substance or surface.

4. Summary of Test Method

4.1 Three 12.7-mm [½-in.] diameter steel balls are clamped together and covered with the lubricant to be evaluated. A fourth 12.7-mm diameter steel ball, referred to as the top ball, is pressed with a force of 147 or 392 N [15 or 40 kgf] into the cavity formed by the three clamped balls for three-point contact. The temperature of the test lubricant is regulated at 75°C [167°F] and then the top ball is rotated at 1200 rpm for 60 min. Lubricants are compared by using the average size of the scar diameters worn on the three lower clamped balls.

NOTE 1—Because of differences in the construction of the various machines on which the four-ball test can be made, the manufacturer's instructions should be consulted for proper machine set up and operation.

NOTE 2—Although the test can be run under other parameters, the precision noted in Section 10 may vary. No aqueous fluid was included in the round-robin to establish the precision limits.

5. Significance and Use

5.1 This test method can be used to determine the relative wear preventive properties of lubricating fluids in sliding contact under the prescribed test conditions. No attempt has been made to correlate this test with balls in rolling contact. The user of this test method should determine to his own satisfaction whether results of this test procedure correlate with field performance or other bench test machines.

6. Apparatus

6.1 *Four-Ball Wear Test Machine*[4]—See Figs. 1-3.

NOTE 3—It is important to distinguish between the Four-Ball E.P. and the Four-Ball Wear Test Machines. The Four-Ball E.P. Test Machine is designed for testing under heavier loads and lacks the sensitivity necessary for wear tests.

6.2 *Microscope*, [4] capable of measuring the diameters of the scars produced on the three stationary balls to an accuracy of

[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.L on Industrial Lubricants (Joint ASTM/ASLE).

Current edition approved Feb. 15, 1994. Published April 1994. Originally published as D 4172 – 82. Last previous edition D 4172 – 88.

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

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

[4] The Four-Ball Wear Test Machine and the Falex Model #6, Multi-Specimen Friction and Wear Test Machine, both made by Falex Corp., 1020 Airpark Drive, Sugar Grove, IL 60554, have been found satisfactory for this purpose. This company can also furnish a microscope with a special base to measure the wear scars without removing the balls from the test-oil cup. Discontinued models of the Four-Ball Wear Test Machine made by Precision Scientific Co. and Roxana Machine Works are also satisfactory.

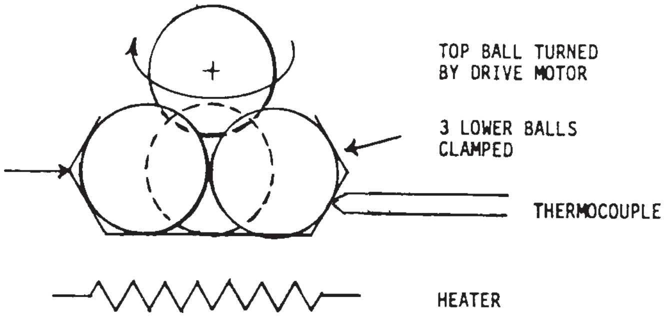


FIG. 1 Schematic of a Four-Ball Wear Test Machine

0.01 mm. It is more efficient to measure the scars without removing the three balls from the holder.

7. Materials

7.1 *Test Balls*, [5] chrome alloy steel, made from AISI standard steel No. E-52100, with diameter of 12.7 mm [0.5 in.] Grade 25 EP (Extra Polish). Such balls are described in ANSI B3.12. The extra-polish finish is not described in that specification. The Rockwell C hardness shall be 64 to 66, a closer limit than is found in the ANSI requirement.

7.2 *Cleaning Fluids* for preparing balls and apparatus for the test should be those approved as nontoxic, capable of removing antirust coatings from the balls, eliminating test-oil carryover from one test to the next, and not contribute to wear or antiwear of the test lubricant. When the fluid(s) is flammable, appropriate precautions should be taken (see Note 1). In the round-robin tests to determine repeatability and reproducibility no specific directions were given for cleaning balls and machine parts. Operators reported using various solvents with and without a sonic cleaning bath. Cleaning techniques reported by some cooperators are included in Research Report RR: D02-1152, see Note 4.

8. Test Conditions

8.1 The test conditions used to develop the precision data as stated in Section 10 were:

	A	*B*
Temperature	75 ± 2°C [167 ± 4°C]	75 ± 2°C [167 ± 4°C]
Speed	1200 ± 60 rpm	1200 ± 60 rpm
Duration	60 ± 1 min	60 ± 1 min
Load	147 ± 2 N [15 ± 0.2 kgf]	392 ± 2 N [40 ± 0.2 kgf]

9. Preparation of Apparatus

9.1 Set up the drive of the machine to obtain a spindle speed of 1200 ± 60 rpm.

9.2 Set temperature regulator to produce a test-oil temperature of 75 ± 2°C [167 ± 4°F].

9.3 When an automatic timer is used to terminate a test, it should be checked for the required ±1 min accuracy at 60 min elapsed time.

[5] Steel balls meeting this description were used in developing the precision of the test. They are available from the manufacturer of the test machine and some ball manufacturers. Some operators prefer to check a new box of balls by running an oil with a known result.

9.4 The loading mechanism must be balanced to a zero reading with all parts and test oil in place. To demonstrate proper precision an addition or subtraction of 19.6 N [0.2 kgf] should be detectable in imbalance. Determination of accuracy of loading at 147 and 392 N [15 and 40 kgf] is difficult and generally limited to careful measurement of lever-arm ratios and weights or piston diameter and pressure gage calibration.

10. Procedure

10.1 Thoroughly clean four test balls, clamping parts for upper and lower balls and the oil cup using solvent or solvents with precautions indicated in 6.2. The parts can be final wiped using a fresh (unused) lint free industrial wipe. After cleaning, all parts are only to be handled using a fresh wipe. No trace of solvent should remain when the test oil is introduced and the machine assembled.

10.2 Tighten one of the clean balls into the spindle of the test machine.

10.3 Assemble three of the clean test balls in the test-oil cup and hand tighten using the wrench supplied by the equipment manufacture which has been found to be approximately 2.8 to 5.6 N·m [25 to 50 lb·in.].

10.4 Pour the oil to be evaluated into the test-oil cup to a level at least 3 mm [⅛ in.] above the top of the balls. Observe that this oil level still exists after the test-oil fills all of the voids in the test-oil cup assembly. In the round-robin to establish this test method the effect of oil level on wear was not determined.

10.5 Install the test-oil cup/three balls in the machine and avoid shock loading by slowly applying the test load (147 or 392 N) [15 or 40 kgf].

10.6 Turn on the heaters and set controls to obtain 75 ± 2°C [167 ± 4°F]. Heater voltage or offset on proportional controllers should be capable of bringing stabilized temperature within the prescribed limits.

10.7 When the test temperature is reached, start the drive motor which was previously set to drive the top ball at 1200 ± 60 rpm. Machines with automatic start using a proportional controller will start below the set temperature. The proportional band should be set narrow enough to limit the "under temperature'' at start to near 2°C [4°F].

10.8 After the drive motor has been on for 60 ± 1 min, turn off the heaters and drive motor and remove the test-oil cup and three-ball assembly.

10.9 Measure the wear scars on the three lower balls to an accuracy of± 0.01 mm by one of the following methods:

10.9.1 *Option A*—Drain the test oil from three-ball assembly and wipe the scar area with a tissue. Leave the three balls clamped and set the assembly on a special base of a microscope that has been designed for the purpose.[4] Make two measurements on each of the wear scars. Take one measurement of the scar along a radial line from the center of the holder. Take the second measurement along a line 90° from the first measurement. Report the arithmetic average of the six measurements as scar diameter in millimetres.

10.9.2 *Option B*—Remove the three lower balls from their clamped position. Wipe the scar area. Make two measurements of each of the three scars. Make the two measurements at 90° to each other. If a scar is elliptical take one measurement with the striations and the other across the striations. Take care to

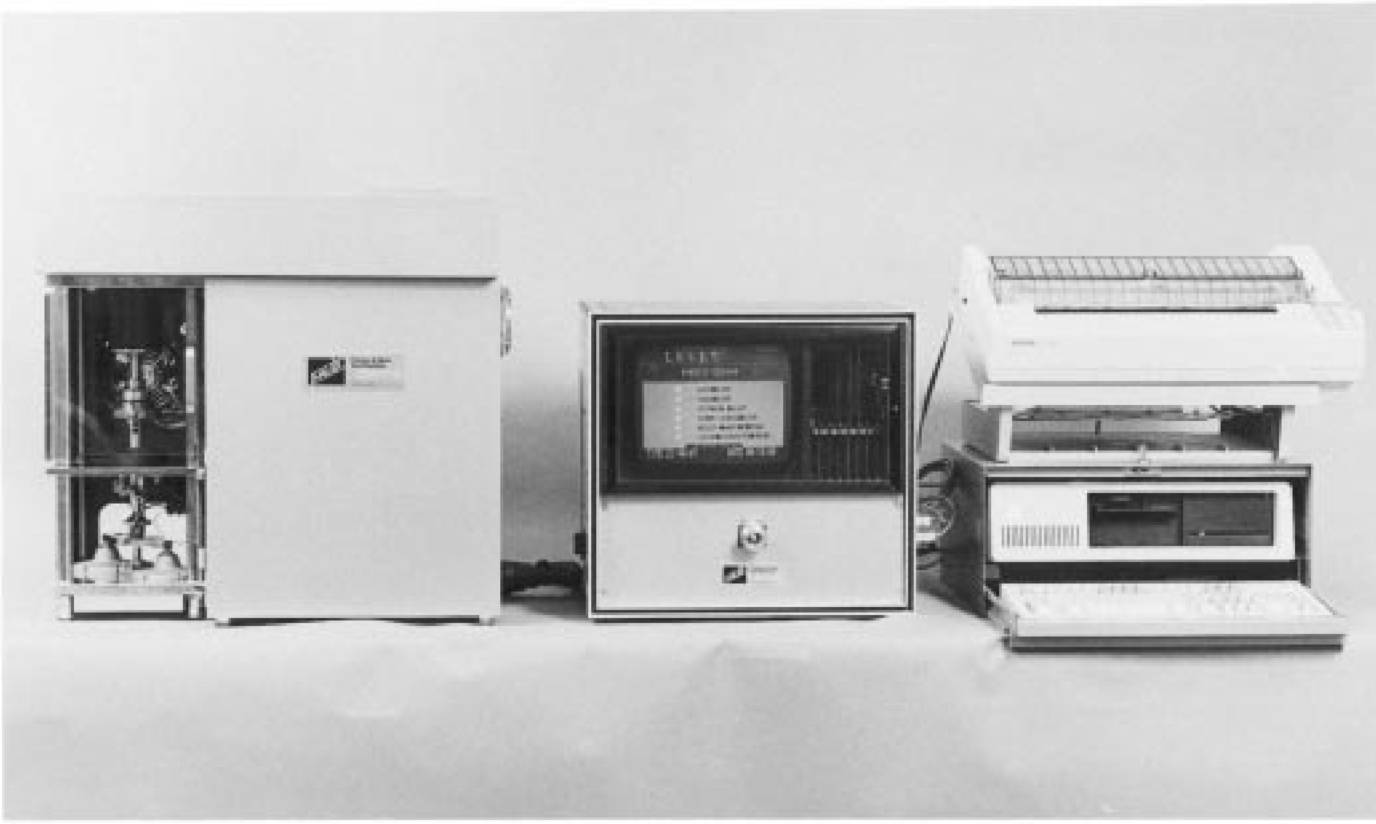


FIG. 2 Falex Model #6, Multi-Specimen Friction and Wear Test Machine

ensure that the line of sight is perpendicular to the surface being measured. As in Option A, average the six readings and report as scar diameter in millimetres.

10.9.3 If the average of the two measurements on one ball varies from the average of all six readings by more than 0.04 mm, investigate the alignment of the three lower balls with the top ball.

11. Precision and Bias [6]

NOTE 4—The precision data[6] were derived from cooperative testing by 13 laboratories on 5 oils under the conditions listed in Section 7. A description of the oils and the average of wear scars obtained at each of the two testing conditions on each of the oils are shown in the Appendix.

11.1 The precision of this test method as determined by the statistical examination of interlaboratory test results is as follows.

11.1.1 *Repeatability*—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 the test method, exceed the following value only in one case in twenty:

$$\text{Repeatability} = 0.12 \text{ mm scar diameter difference} \quad (1)$$

11.1.2 *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, exceed the following value only in one case in twenty:

$$\text{Reproducibility} = 0.28 \text{ mm scar diameter difference} \quad (2)$$

11.2 *Bias*—The procedure in this test method has no bias because the value of ball scar width can only be defined in terms of a test method.

12. Keywords

12.1 lubricant; wear

[6] The complete results of the cooperative round-robin test program are filed at ASTM Headquarters. Request RR: D02-1152.

FIG. 3 Falex Variable-Speed Four-Ball Wear Test Machine

APPENDIX

(Nonmandatory Information)

X1. SUMMARY OF COOPERATIVE TESTING

X1.1 Table X1.1 is a summary of cooperative testing.

TABLE X1.1 Summary of Cooperative Testing

Sample		Scar Diameter, mm	
Number	Description	147 N	392 N
LXI2-1	Mineral Oil, 46 cSt at 40°C	0.56	0.72
LXI2-2	LXI2-1 plus 1 % wt ZDT[A]	0.27	0.42
LXI2-3	LXI2-1 plus 2 % wt S/P[B]	0.28	0.35
LXI2-4	Synthetic hydrocarbon	0.53	0.76
LXI2-5	Tricresyl phosphate	0.54	0.59

[A]ZDT = zinc O, O-dialkylphosphorodithioate.
[B]S/P = additive containing sulfur and phosphorus.

Harris Testing Laboratories, Inc.

CERTIFICATE OF ANALYSIS

CLIENT:
Megatrol Inc.
9469 S 500 W
Sandy, UT 84070

PRODUCT: **FE**^{26} Premier
MARKS: NONE
DATE RECEIVED: 02/11/2008
LAB NO: HH0410-1202-P
SUBMITTED BY: Jon Rose

			SPECIFICATIONS	
METHOD	**TEST**	**RESULT**	**MIN.**	**MAX.**
D-5800	**Noack Volatility, wt %**	**6.2**	**XXX**	**XXX**

Comments: **Good Product**

Amos Mwangi

Date issued:
02/11/2008

CHEMIST

Designation: D 5800 – 04

An American National Standard

Standard Test Method for Evaporation Loss of Lubricating Oils by the Noack Method[1]

This standard is issued under the fixed designation D 5800; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method covers three procedures for determining the evaporation loss of lubricating oils (particularly engine oils). Procedure A uses the Noack evaporative tester equipment; Procedure B uses the automated non-Woods metal Noack evaporative apparatus; and Procedure C uses Selby-Noack volatility test equipment. The test method relates to one set of operating conditions but may be readily adapted to other conditions when required.

1.2 Noack results determined using Procedures A and B show consistent differences. Procedure A gives slightly lower results versus Procedure B on formulated engine oils, while Procedure A gives higher results versus Procedure B on basestocks.

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

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

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance

D 6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

2.2 *DIN Standards:*[3]

DIN 1725 Specification for Aluminum Alloys

DIN 12785 Specifications for Glass Thermometers

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *evaporation loss—of a lubricating oil by the Noack method*, that mass of volatile oil vapors lost when the oil is heated in a test crucible through which a constant flow of air is drawn.

3.1.2 *volatility*, *n*—the tendency of a liquid to form a vapor.

4. Summary of Test Method

4.1 A measured quantity of sample is placed in an evaporation crucible or reaction flask that is then heated to 250°C with a constant flow of air drawn through it for 60 min. The loss in mass of the oil is determined.

4.2 Interlaboratory tests have shown that Procedure A, Procedure B, and Procedure C yield essentially equivalent results, with a correlation coefficient of $R^2 = 0.996$. See the research report[4] for the Selby-Noack interlaboratory study.

5. Significance and Use

5.1 The evaporation loss is of particular importance in engine lubrication. Where high temperatures occur, portions of an oil can evaporate.

5.2 Evaporation may contribute to oil consumption in an engine and can lead to a change in the properties of an oil.

5.3 Many engine manufacturers specify a maximum allowable evaporation loss.

5.4 Some engine manufacturers, when specifying a maximum allowable evaporation loss, quote this test method along with the specifications.

5.5 Procedure C, using the Selby-Noack apparatus, also permits collection of the volatile oil vapors for determination of their physical and chemical properties. Elemental analysis of the collected volatiles may be helpful in identifying components such as phosphorous, which has been linked to premature degradation of the emission system catalyst.

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.06 on Analysis of Lubricants.

Current edition approved May 1, 2004. Published June 2004. Originally approved in 1995. Last previous edition approved in 2003 as D 5800–03a.

[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 Deutsches Institut für Normunge, Beuth Verlag GmbH, Burggrafen Strasse 6, 1000 Berlin 30, Germany.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1462.

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

Procedure A

6. Apparatus

6.1 *Noack Evaporative Tester*, comprising the following:

6.1.1 *Electrically Heated Block Unit*, made from a malleable aluminum alloy (see DIN 1725, Sheet 1), insulated at the jacket and base against loss of heat. (**Warning—**This block is heated to 250°C.) The block is heated electrically by a base and jacket heater, having a total power consumption of 1 to 1.2 kW. In this respect the difference between both individual power consumption should not exceed 0.15 kW. In the center of the heating block, there is a circular recess to insert the evaporating crucible, the space between block and crucible being filled with Woods alloy or a suitable equivalent. Two catches on the block prevent the crucible from rising in the liquid metal bath. Two additional circular recesses at equal intervals from the center of the block are provided for the thermometers (see Fig. 1).

6.1.2 *Evaporating Crucible*, with screw cover. The crucible is made of stainless steel (see Fig. 2). Above the support ring is the thread for the cover. The nickel-plated brass cover is hermetically sealed to the crucible by an internal conical sealing surface (see Fig. 3). Three nozzles of hardened steel permit the air stream to pass through the cover. The extraction tube, which slopes downward, leads from a threaded and sealed connection in the center of the cover.

6.2 *Balance*, capable of weighing at least 200 g to the nearest 0.01 g.

6.3 *Crucible Clamp and Spanner*.

6.4 *Reamer*, 2-mm diameter.

6.5 *Ball Bearing*, 3.5-mm diameter.

6.6 *Thermometer*, M260 (see DIN 12785) or temperature sensing device capable of reading temperature to 0.1°C. The thermometer should be calibrated with appropriate procedure at appropriate frequency (generally every six months).

6.7 *Contact Type Control Thermometer* (for manual).

6.8 *Glass Y-piece*, an internal diameter of 4 mm. The upright arms, each 45-mm long, should form an angle such that the arm connected to the crucible extraction tube and the Y-piece form a straight line. The vertical arm is 60-mm long and beveled at 45°.

6.9 *Glass Delivery Tubes*, an internal diameter of 4 mm, each arm length 100 mm, beveled at 45° at ends entering and leaving the bottles.

6.9.1 Bent at an angle of approximately 80°.

6.9.2 Bent at an angle of approximately 100°, length to 20 mm of bottle base.

6.9.3 Bent at an angle of approximately 90°.

6.10 *Two Glass Bottles*, approximately 2-L capacity, fitted with rubber bungs bored to receive inlet and outlet tubes (see Fig. 4).

6.11 *Manometer*, inclined form, water-filled, precision 0.2 mm H_2O or suitable pressure sensor capable of measuring 20 $\pm$ 0.2 mm of H_2O (a 0 to 50-mm H_2O pressure transducer has been found to be satisfactory).

NOTE 1—Some manometers use water as the reference fluid, others may use a lower density fluid correlated to read in millimetres of water. Users should ensure that the manometer is filled with the correct density reference fluid.

6.12 *Glass T-Piece*, with bleed valve attached.

6.13 *Vacuum Pump*.

6.14 *Timer*, with accuracy of 0.2 s.

6.15 *Silicone Rubber Tubing*, cut to size, with an internal diameter of 4 mm.

6.15.1 40-mm long; three pieces required,

6.15.2 300-mm long, and

6.15.3 100-mm long.

NOTE 2—The use of automated equipment is permissible as long as it gives equivalent results specified in this test method. All hardware dimensions, make-up of the block, crucible, heat capacity, and so forth, and glassware must conform to the specifications given in this test method.

7. Reagents and Materials

7.1 *Cleaning Solvent*—A mixture of naphtha and toluene is recommended for the cleaning of the crucible. (**Warning—**Flammable, vapor harmful.) Overnight soaking may be necessary.

7.2 *Datum Oil RL 172*, (formerly RL-N) Reference Oil.

7.3 *Insulated Gloves*.

7.4 *Paint Brush*, such as a tinnerps acid brush (15 to 25-mm width).

7.5 *Woods Metal*[5] *or suitable heat transfer material*—(**Warning—**Woods metal contains lead (25 %), bismuth (50 %), antimony (12.5 %), and cadmium (12.5 %); these have been found to be health hazardous. Avoid contact with skin at all times.)

8. Hazards

8.1 *Safety Hazards*—It is assumed that anyone using this test method will either be fully trained and familiar with all normal laboratory practices, or will be under the direct supervision of such a person. It is the responsibility of the operator to ensure that all local legislative and statutory requirements are met.

8.2 (**Warning—**Though the test method calls for a draft free area, the exhaust fumes from the evaporating oil must be ventilated to an outside source. Precaution shall be taken to avoid any possibility of fire or explosion.)

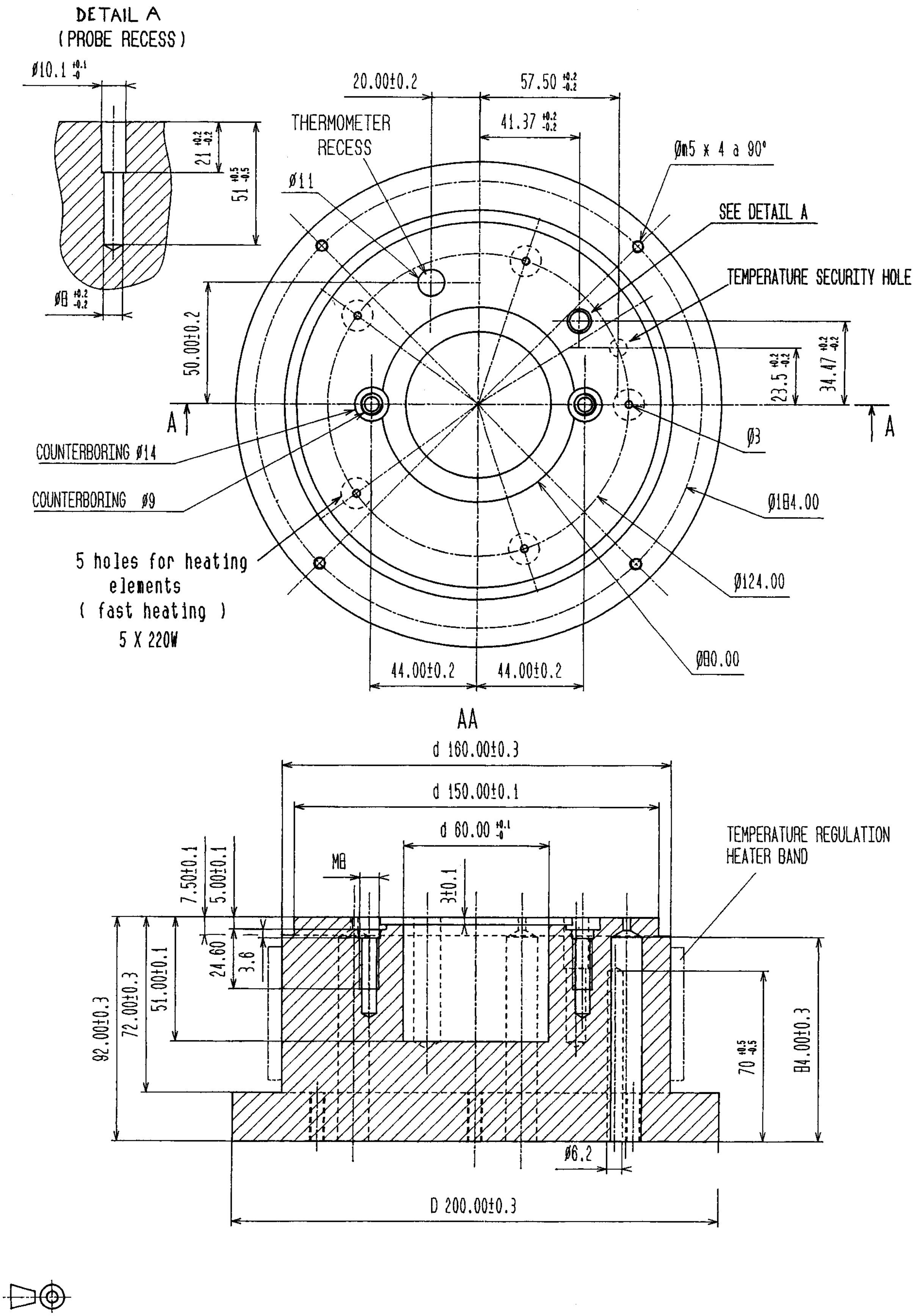
9. Preparation of Apparatus

9.1 A standard assembly of the apparatus is shown in Fig. 5. To avoid disturbing the thermal equilibrium, the apparatus shall be assembled in a draft free area and comply with Fig. 5 in dimensions and apparatus. (See 8.2.)

9.2 Add sufficient Woods metal or equivalent material to the recesses of the heating block so that, with the crucible and thermometer in place, the remaining spaces will be filled with the molten metal.

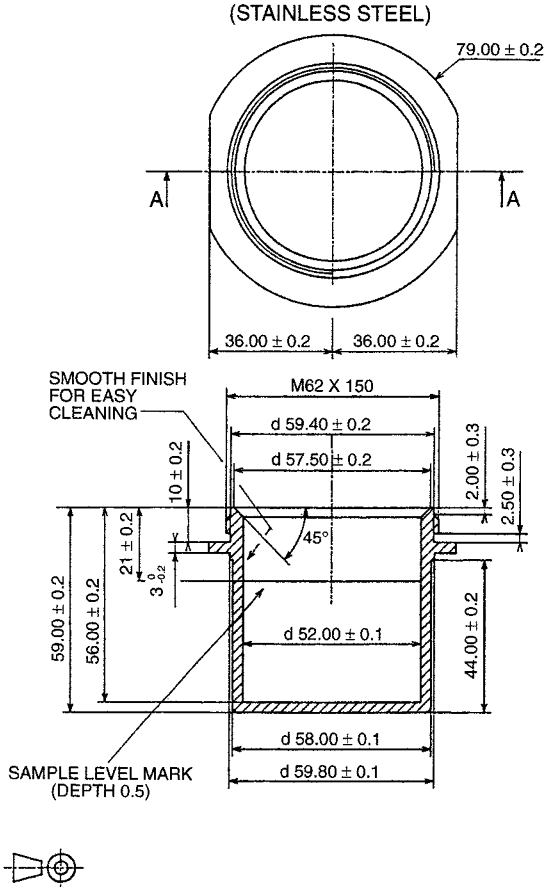
9.3 Using the highest heating rate possible, raise the temperature of the heating block until the Woods metal is molten. Insert the thermometers with their bulbs touching the bottom of the recesses, and ensure that the contact thermometer is

[5] Woods metal, available from Aldrich Chemical Co., has been found satisfactory for this purpose. An equivalent may be used. Subcommittee D02.06 is evaluating less toxic alloys as alternatives.



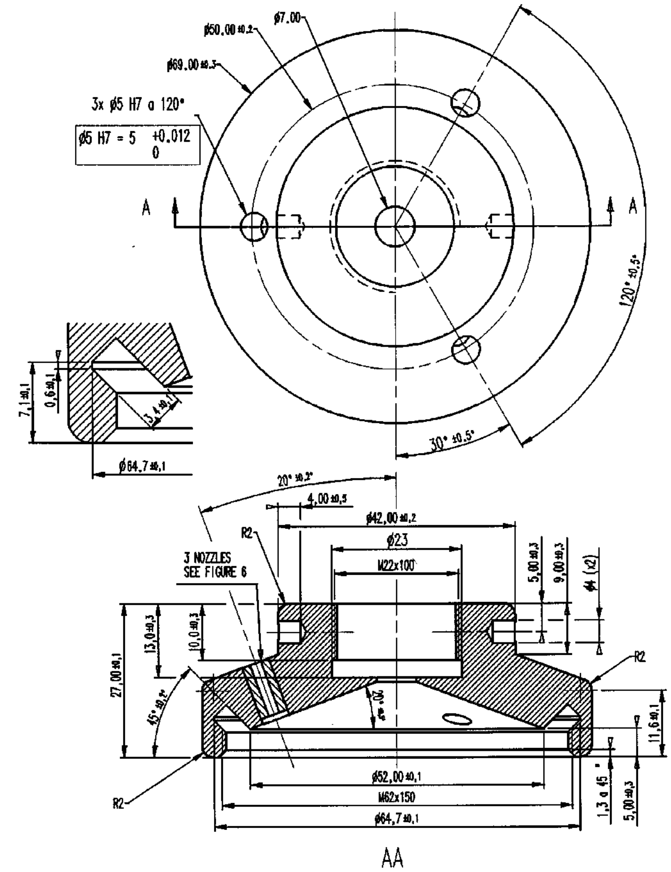
NOTE—All dimensions in millilitres.

FIG. 1 Heating Block



NOTE—All dimensions in millilitres.

FIG. 2 Crucible



NOTE—All dimensions in millilitres.

FIG. 3 Cover

plugged in the back of the heating block. Adjust the power supplied to the heating block so that the temperature can be maintained at 250 ± 0.5°C.

9.4 Assemble the remaining apparatus, less the crucible, as shown in Fig. 5.

9.5 Place an empty crucible in the heating block, securing the flange under the screw heads against the buoyancy of the Woods metal. The level of the molten metal should be such that a trace of it can be seen at the flange of the crucible and the top of the heating block.

9.6 Check that the readings can be obtained on the manometer scale, or other measuring device, by connecting the crucible to the assembled apparatus. A reading of 20.0 ± 0.2 mm shall be obtained.

9.7 Disconnect and remove the crucible from the assembled apparatus.

9.8 Switch off the pump and the heating block and raise the crucible and the thermometers from the molten Woods metal. Using the brush, return any Woods metal clinging to the crucible to the heating block.

9.9 Clean the Y-piece and glass tubing to prevent a build up of condensate.

10. Verification

10.1 Switch on the pump and the heating block and ensure that the apparatus is assembled, minus the crucible, as shown in Fig. 5.

10.2 Check that the crucible and cover are free from lacquer.

10.2.1 After every test, clean the crucible and cover with solvent and allow to dry. Stubborn lacquer can be cleaned by abrasion from a glass beader under pressure.

10.3 Pass the reamer through each of the three nozzles in the cover to ensure that they are clear. (**Warning**—Using a reamer with a diameter larger than 2 mm can enlarge the nozzles. This can lead to higher losses because of increased air flow.)

10.4 Run the ball bearing through the extraction tube to ensure that it is clear of dirt.

10.5 Weigh the empty crucible without its cover to the nearest 0.01 g.

10.6 Weigh into the crucible 65.0 ± 0.1 g of the Reference Oil.

10.7 Screw on the cover using the clamp and spanner.

10.8 Ensure the temperature of the heating block is at 250 ± 0.5°C. Place the crucible in its recess in the heating block, securing the flange under the screw heads against the buoyancy

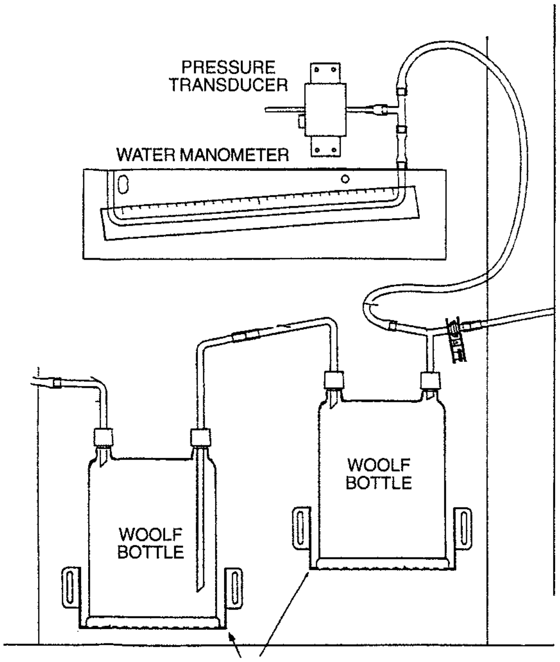


FIG. 4 Glassware

of the Woods metal. Switch the control of the heating block to compensate for the heat capacity of the crucible. Immediately (in less than 5 s), connect the extraction tube of the crucible to the arm of the glass Y-piece, making a butt joint. Simultaneously, start the pump and the stopwatch and adjust the bleed valve to give a pressure differential of 20 ± 0.2 mm.

NOTE 3—When the crucible is in the test position, its flange should be flush with the top of the heating block. Any protrusion of the crucible flange above the heating block may suggest a buildup of Woods metal slag at the bottom of the heating block recess. The heating block and the thermometer recesses should be cleaned and the Woods metal replaced on a regular basis to avoid the accumulation of slag. Oxidized Woods metal will affect the heat transfer to the crucible and hence may have a deleterious effect on the results obtained.

10.9 Adjust the control on the heating block to maintain the block temperature approximately 5°C below the test temperature. Readjust the temperature control so that the test temperature is reestablished within 3 min of the start of the test.

NOTE 4—Temperature and pressure will be controlled automatically when automated equipment is used.

10.10 At the start of the test, constant attention shall be paid to maintaining the correct pressure. Once this becomes steady, usually within 10 to 15 min, check periodically that the temperature and pressure differential remain constant throughout the period of the test.

10.11 After 60 min ± 5 s, lift the crucible from the heating block, remove any adhering alloy, and place the crucible in a warm water bath to a depth of at least 30 mm. The time period from the end of the test to immersion of the crucible shall not exceed 60 s.

10.12 After 30 min, remove the crucible from the water, dry the outside, and carefully remove the lid.

10.13 Reweigh the crucible without the lid to the nearest 0.01 g.

10.14 Calculate to the nearest 0.1 % mass/mass (M/M) the evaporation loss of the reference oil.

10.15 Compare the result obtained against the given value for the reference oil. If the result is within 6 % of the value, repeat the procedure from 11.1, using the test sample.

10.16 If the result is not within 6 % of the given value, check that the apparatus complies with that shown in Fig. 5, and that the procedure has been adhered to. Check the calibration of the thermometer and pressure sensing device.

10.17 Re-check the evaporation loss of the reference oil.

NOTE 5—Condensate should not be allowed to build up in the 2-L glass bottles. These should be washed out with solvent before a maximum 1 cm of condensate collects.

NOTE 6—The equipment should be referenced approximately every ten tests if the test is used frequently. If the testing is infrequent, the equipment should be referenced before the first sample is run.

11. Procedure

11.1 Weigh into a tarred crucible 65 ± 0.1 g representative of the test sample to a precision of 0.01 g.

NOTE 7—Sample in accordance with Practice D 4057 or Practice D 4177.

11.2 Proceed as described in 10.7 to 10.12.

11.3 Calculate to the nearest 0.1 % M/M the evaporation loss of the sample.

12. Calculations and Results

12.1 Evaporation loss is obtained from the difference in weight before and after 1 h at 250°C.

$$\text{evaporation loss} = \frac{(B - A) - (C - A)}{B - A} \times 100 \tag{1}$$

where:

A = empty crucible weight,
B = crucible plus sample weight, and
C = crucible plus sample after 1 h of heating.

13. Report

13.1 Report the following information:

13.1.1 The nearest 0.1 % M/M as evaporation loss (Test Method D 5800).

14. Precision and Bias

14.1 The interlaboratory round robin used manual, semi-automated, and automated equipment. The precision values were calculated on the statistical examinations of interlaboratory test results as follows.

14.1.1 *Repeatability*—A quantitative measurement of precision associated with single results obtained by the same operator with the same equipment in the same laboratory within a short interval of time. In the normal and correct operation of the test method, the following values were exceeded in only one case in twenty.

$$\text{Repeatability} = 5.8\ \% \times \text{average M/M evaporation loss} \tag{2}$$

14.1.2 *Reproducibility*—A quantitative measure of precision with single results obtained in different laboratories on identical test material. In the normal and correct operation of the test, the following values were exceeded in only one case in twenty.

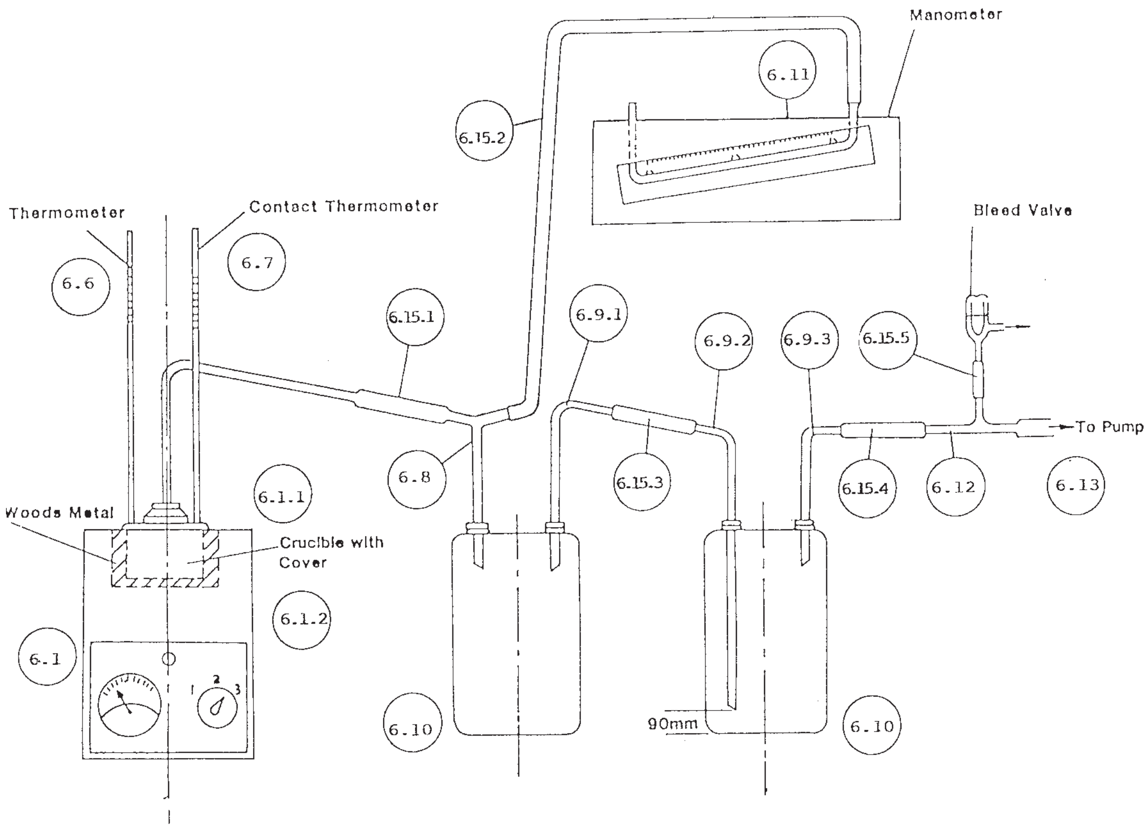


FIG. 5 Test Apparatus

$$\text{Reproducibility} = 18.3\ \% \times \text{average M/M evaporation loss} \quad (3)$$

14.2 The procedure in this test method has no bias because the value of the volatility is defined only in terms of this test method.

Procedure B Non-Woods Metal Apparatus

15. Introduction

15.1 The following procedure describes an automated test method that uses the same principle, and the same crucible as Procedure A. Only the heat transfer to the sample is different. It does not use Woods alloy, and the sample temperature is directly monitored.

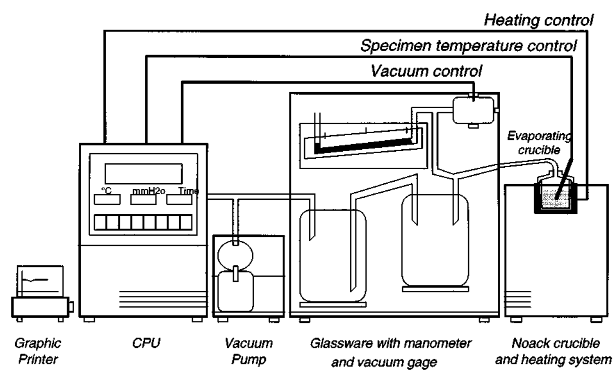


FIG. 6 Automated Non-Woods Metal Noack Evaporative Apparatus

16. Apparatus

16.1 *Noack Evaporative Tester* (see Fig. 6), comprising the following:

16.1.1 *Heating Block Unit*, electrically heated by base and jacket heaters, having a total power consumption sufficient to ensure a specimen temperature profile similar to the one recorded in the specimen when heated with the Woods metal heater block. In the center of the heating block, there is a circular recess to insert the evaporative crucible. The jacket heater is configured to ensure a direct contact with the crucible. A mechanism is provided to open the jaws for crucible insertion. Two catches on the block prevent the crucible from rising, and the base heater is spring loaded to ensure a direct contact with the crucible.

16.1.2 *Evaporative Crucible*, with screw cover (see Fig. 7). The crucible is made of stainless steel (see Fig. 8). Above the support ring is the thread for the cover. The nickel-plated brass cover (see Fig. 9) is hermetically sealed to the crucible by an internal conical sealing surface. Three nozzles of hardened steel (see Fig. 10) permit the air stream to pass through the cover. The extraction tube (see Figs. 11 and 12), which slopes downward, leads from a threaded and sealed connection in the center of the cover.

16.1.3 *Temperature Probe*—The specimen temperature measuring device shall have an accuracy of 0.5°C, or better, and a resolution of 0.1°C, or better. The probe is provided with a calibration certificate of 250.0°C with a precision of ±0.1°C. Its diameter is 4 mm, and its position is as indicated in Fig. 8.

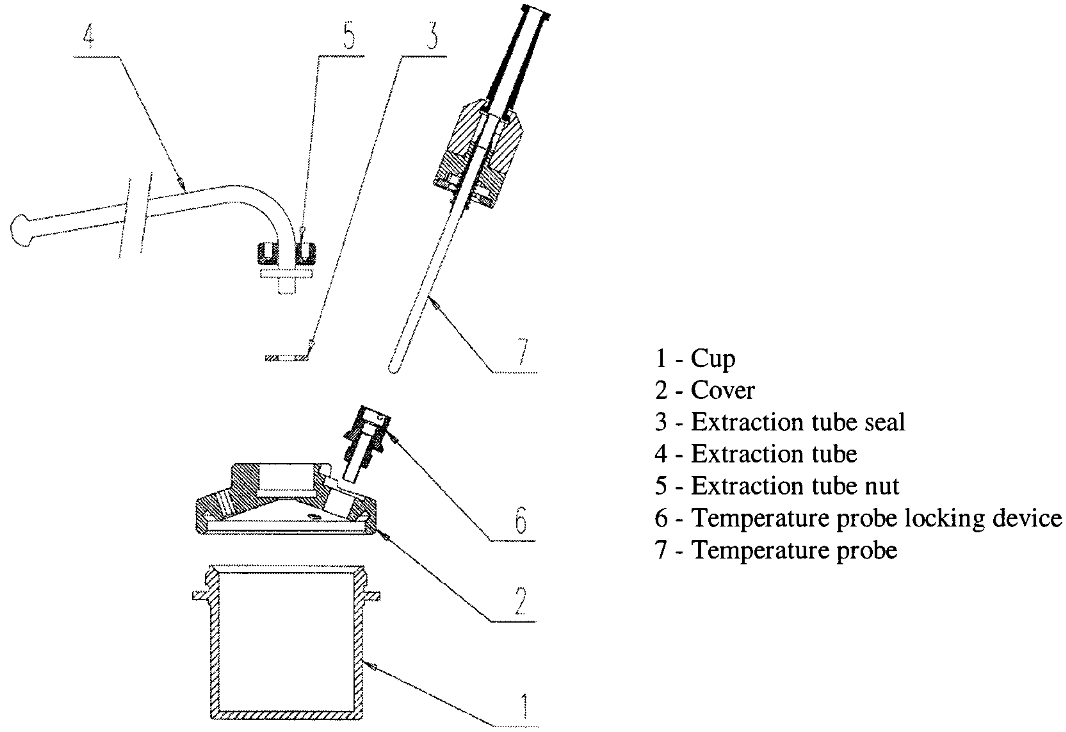


FIG. 7 Crucible with Temperature Probe

It should be calibrated with appropriate procedure at appropriate frequency (minimum once a year).

16.2 *Balance*, capable of weighing at least 500 g to the nearest 0.01 g.

16.3 *Crucible Clamp and Spanner*.

16.4 *Reamer*, 2-mm diameter.

16.5 *Ball Bearing*, 3 to 5-mm diameter.

16.6 *Glassware Assembly*, strictly identical to the description in 6.6 to 6.12 and 6.15 of Procedure A.

16.7 *Vacuum Pump*.

16.8 *Central Processing Unit* (CPU), capable of controlling the specimen temperature, the vacuum, the time, the heating, and the printing. The specimen is heated to 245.2 ± 0.5°C with the temperature profile recorded in the specimen when tested with a Woods metal apparatus (1 h at 250°C) with automatic test duration compensation. The automatic test duration compensation is used because a test may be started with a heating block at room temperature or at hot temperature when several tests are carried without cooling phase. The CPU automatically adjusts the pressure differential of 20 ± 0.2 mm. These conditions can be checked with the printed report.

16.9 *Printer*, to print the graphs of the specimen temperature and the vacuum recorded during the test.

17. Reagents and Materials

17.1 *Cleaning Solvent*—A mixture of naphtha and toluene is recommended for cleaning the crucible. (**Warning**—Flammable, vapor harmful.) Overnight soaking may be necessary.

17.2 *Noack Reference Fluid*—Oil having a known evaporative loss, the value of which is provided by the manufacturer.

17.3 *Insulated Gloves*.

17.4 *Drying Paper*.

18. Hazards

18.1 *Safety Hazards*—It is assumed that anyone using this test method will either be fully trained and familiar with all normal laboratory practices, or will be under the direct supervision of such a person. It is the responsibility of the operator to ensure that all local legislative and statutory requirements are met.

18.2 (**Warning**—Though the test method calls for a draft free area, the exhaust fumes from the evaporating oil must be ventilated to an outside source. Precaution shall be taken to avoid any possibility of fire or explosion.)

19. Preparation of Apparatus

19.1 A standard assembly of the apparatus is shown in Fig. 6. To avoid disturbing the thermal equilibrium, the apparatus shall be assembled in a draft free area and shall comply with Fig. 6 dimensions and apparatus. (See 18.2.)

19.2 Prepare the automated apparatus for operation in accordance with the manufacturer's instructions for calibrating, checking, and operating the equipment.

19.3 Clean the glass bottles, the glass tubing, and the Y-piece to prevent a build up of condensate.

NOTE 8—Condensate should not be allowed to build up in the 2-L glass bottles. These should be washed out with solvent and dried before a maximum 2 cm of condensate collects.

20. Verification

20.1 Switch the instrument on a minimum of 30 min before running the test to allow temperature stabilization of measurement circuitry.

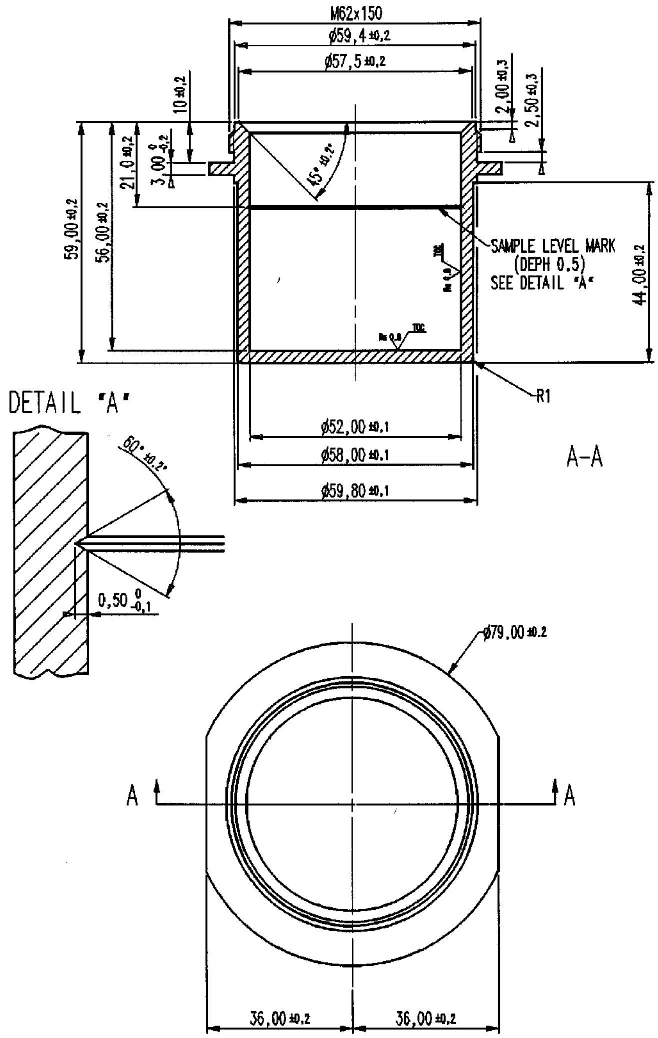


FIG. 8 Noack Cup (Detail 1 of Fig. 7)

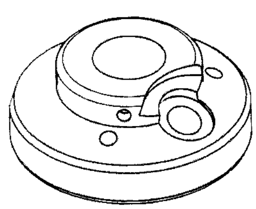


FIG. 9 Crucible Cover (Detail 2 of Fig. 7)

20.2 Make sure that the glassware assembly and the vacuum pump are cleaned and all the connections are sealed.

20.3 Thoroughly clean and dry all parts of the test cup and its accessories before starting the test. Check that the crucible and cover are free from lacquer. Stubborn lacquer should be removed by light abrasion with fine carborundum powder on a pad of cotton wool soaked in solvent or with a fibrous abrasive pad, followed by a rinse with solvent.

20.4 Pass the reamer through each of the three nozzles in the cover to ensure that they are clear. (**Warning**—Using a reamer with a diameter larger than 2 mm can enlarge the nozzles. This can lead to a wrong losses result due to increased air flow.)

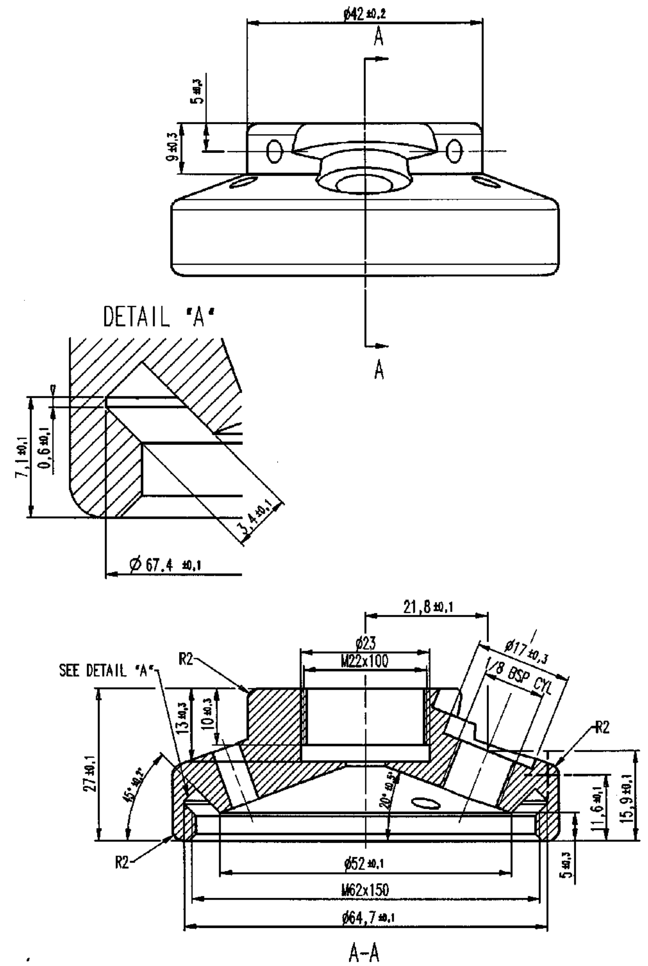


FIG. 9 Crucible Cover *(continued)*

20.5 Run the ball bearing through the extraction tube to ensure that it is clear of contaminants.

20.6 After the 30 min stabilization period, calibrate the temperature measuring device in accordance with the manufacturer's instructions.

20.7 Calibrate the vacuum measuring device in accordance with the manufacturer's instructions.

20.8 Weigh the empty cup without its cover to the nearest 0.01 g.

20.9 Weigh into the tared crucible 65.0 ± 0.1 g of reference fluid to a precision of 0.01 g. This mass is called M_1.

20.10 Screw on the cover using the clamp and the spanner. During this phase, make sure that the specimen will never splash on the inside part of the cover. If this occurs, even only one time, the test shall be repeated from 20.3.

20.11 Connect the specimen temperature probe to the instrument.

20.12 Press down on the locking lever located on the front of the heating block. Place the crucible in the heating block. Rotate the crucible, securing the flange under the screw heads. Adjust the final position of the extraction tube so that it is located in front of the arm of the glass Y-piece, and release the locking lever.

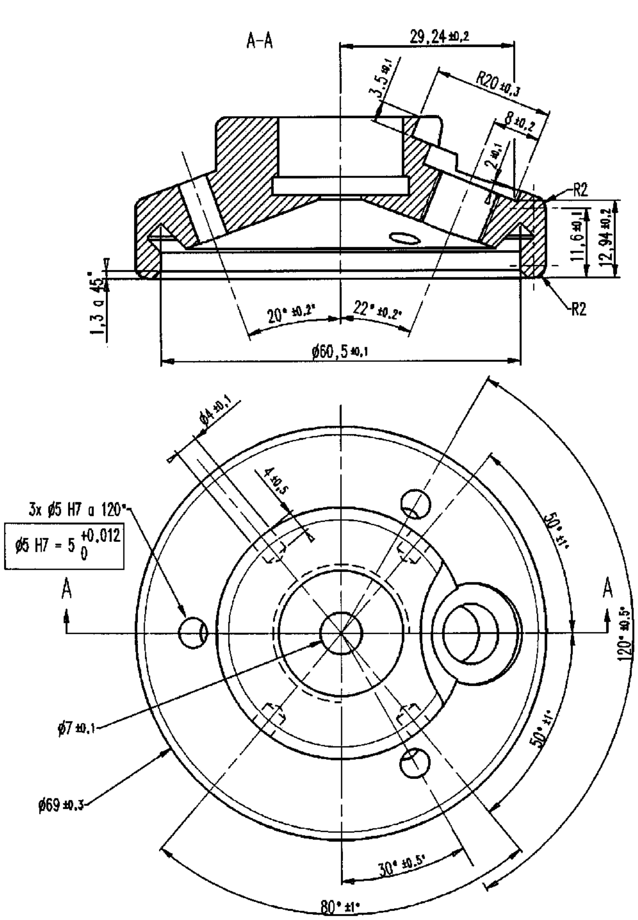


FIG. 9 Crucible Cover *(continued)*

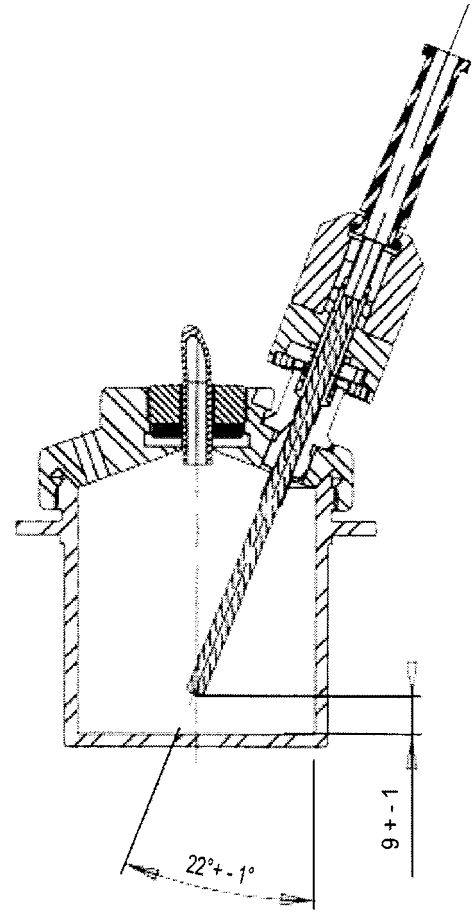


FIG. 10 Specimen Temperature Probe Positioning

20.13 Connect the extraction tube to the arm of the glass Y-piece, and secure the connection with the clamp. Be sure that the stainless extraction tube, the male connection, and the right arm of the Y-piece are properly aligned.

20.14 Start the test by pressing the ON key of the CPU. In default configuration, the printer is activated. If not, refer to the instruction manual to activate the real time printing of the specimen temperature and the vacuum curves.

20.15 When the audible alarm signals the last 3 min of the test, stop the audible alarm by pressing on the OFF key. Stay in front of the equipment, and be prepared to disconnect the extraction tube.

20.16 After 60 min, the test is automatically stopped, and the end of the test alarm sounds. Remove the specimen temperature probe. Disconnect the extraction tube within 15 s maximum. Press down the locking lever. Remove the crucible. Stop the audible alarm by pressing on the OFF key.

20.17 Stand the crucible in a cold water bath to a minimum depth of 30 mm.

20.18 Check the printed report to ensure that the specimen temperature and the vacuum plotted curves stayed within the indicated limits. If one of the graph is not within the specified limits, check that the apparatus complies with the manufacturer's instruction and that the procedure has been adhered to. After these checks, rerun the test from 20.2.

20.19 After 30 min, remove the crucible from the water bath, dry the outside, and carefully remove the lid. This phase is very critical. Make sure that the sample is never in contact with the inside part of the lid.

NOTE 9—It is very important during the manipulation of the crucible, at the start and the end of the test, to not splash the internal face of the cover with the specimen in the cup. When this occurs, it leads to higher losses and *the test must be rerun*.

20.20 Reweigh the crucible without the lid to the nearest 0.01 g.

20.21 Calculate the M_2 mass by subtracting the empty cup mass from the mass measured in 20.19.

20.22 Calculate to the nearest 0.1 % M/M the evaporation loss of the reference fluid, using the following equation:

$$[(M_1 - M_2)/M_1] \times 100 \tag{4}$$

where:

M_1 = specimen mass before the test, and
M_2 = specimen mass after the test at 245.2°C.

20.23 Compare the result obtained against the given value for the reference fluid. If the result is within limits, proceed to Section 21.

20.24 If the result is not within the limits, check that the apparatus complies with the manufacturer's instruction and that the procedure has been adhered to.

20.25 Recheck the evaporation loss of the reference oil. To do so, proceed as described in 20.2.

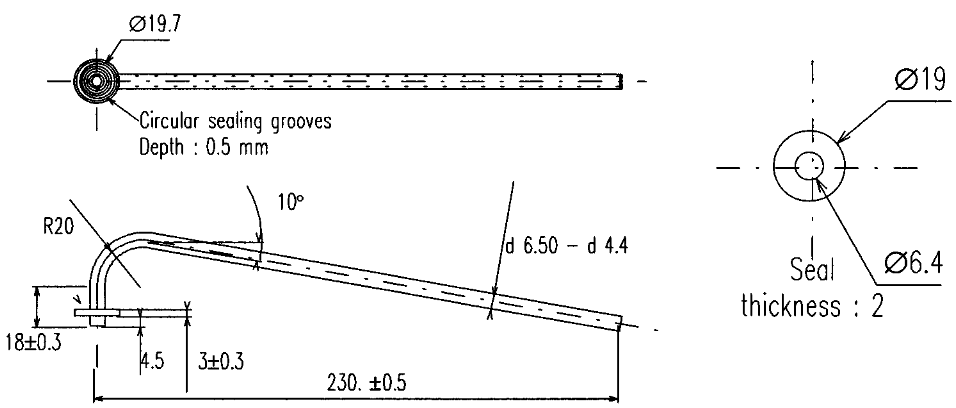


FIG. 11 Extraction Tube (Stainless Steel) with Its Seal (Details 3 and 4 of Fig. 7)

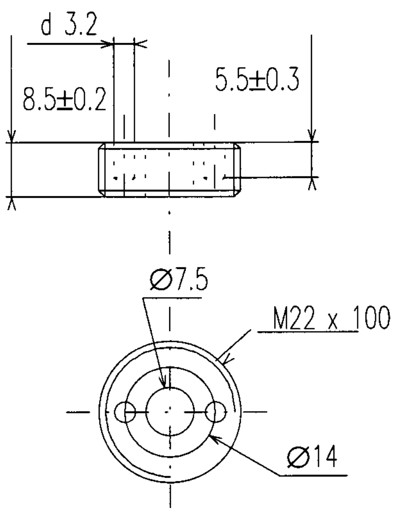


FIG. 12 Extraction Tube Nut (Stainless Steel) (Detail 5 of Fig. 7)

21. Procedure

21.1 Weigh into a tared crucible 65 ± 0.1 g representative of test specimen to a precision of 0.01 g.

NOTE 10—Sample in accordance with Practice D 4057 or Practice D 4177.

21.2 Proceed as described in 20.3-20.20.

21.3 Calculate to the nearest 0.1 % M/M the evaporation loss of the specimen, using Eq 4.

22. Calculation

22.1 Evaporation loss is obtained from the difference in weight before and after test. The specimen is heated in accordance with the temperature profile recorded in the specimen when tested with a Woods metal apparatus (1 h at 250°C) with automatic test duration compensation. The automatic test duration compensation is used because a test may be started with a heating block at room temperature or at hot temperature when several tests are carried without cooling phase. The checking of these conditions can be done with the printed report. Calculate evaporation loss, using the following equation:

$$[(M_1 - M_2)/M_1] \times 100 \quad (5)$$

where:

M_1 = $B - A$,
M_2 = $C - A$,
A = empty crucible weight,
B = crucible plus specimen weight, and
C = crucible plus specimen after the test.

22.2 Some consistent differences in results determined using Procedures A and B have been observed depending on the type of sample tested. A test result obtained using one of the procedures can be transformed to an estimated result on the basis of the other procedure as follows:

22.2.1 *Formulated Engine Oils*—The following relationships are based on the round robin test results on formulated engine oils with volatilities in the range of 10 % to 22 % Noack:

$$\text{Value by Noack Procedure B} = 1.027 \times \text{Value by Noack Procedure A} \quad (6)$$

$$\text{Value by Noack Procedure A} = 0.974 \times \text{Value by Noack Procedure B} \quad (7)$$

The 95 % confidence limits for the regression coefficient in Eq 6 are 1.021 to 1.033; those for the coefficient in Eq 7 are 0.968 to 0.980.

22.2.2 The following relationships are based on round robin test results on basestocks with volatilities in the range of 4 % to 15 % Noack:

$$\text{Value by Noack Procedure B} = 0.955 \times \text{Value by Noack Procedure A} \quad (8)$$

$$\text{Value by Noack Procedure A} = 1.048 \times \text{Value by Noack Procedure B} \quad (9)$$

The 95 % confidence limits for the regression coefficient in Eq 8 are 0.950 to 0.959; those for the coefficient in Eq 9 are 1.043 to 1.053.

23. Report

23.1 Report the following information:

23.1.1 The nearest 0.1 % M/M as evaporation loss (Test Method D 5800, Procedure B).

23.2 Conversion of values from either D 5800 A or D 5800 B to the other:

23.2.1 Only if the nature of the test specimen is known with certainty, in other words, it is known to be either a basestock or a formulated engine oil, the evaporation loss calculated in 22.2.1 on the basis of either Procedure A or Procedure B may

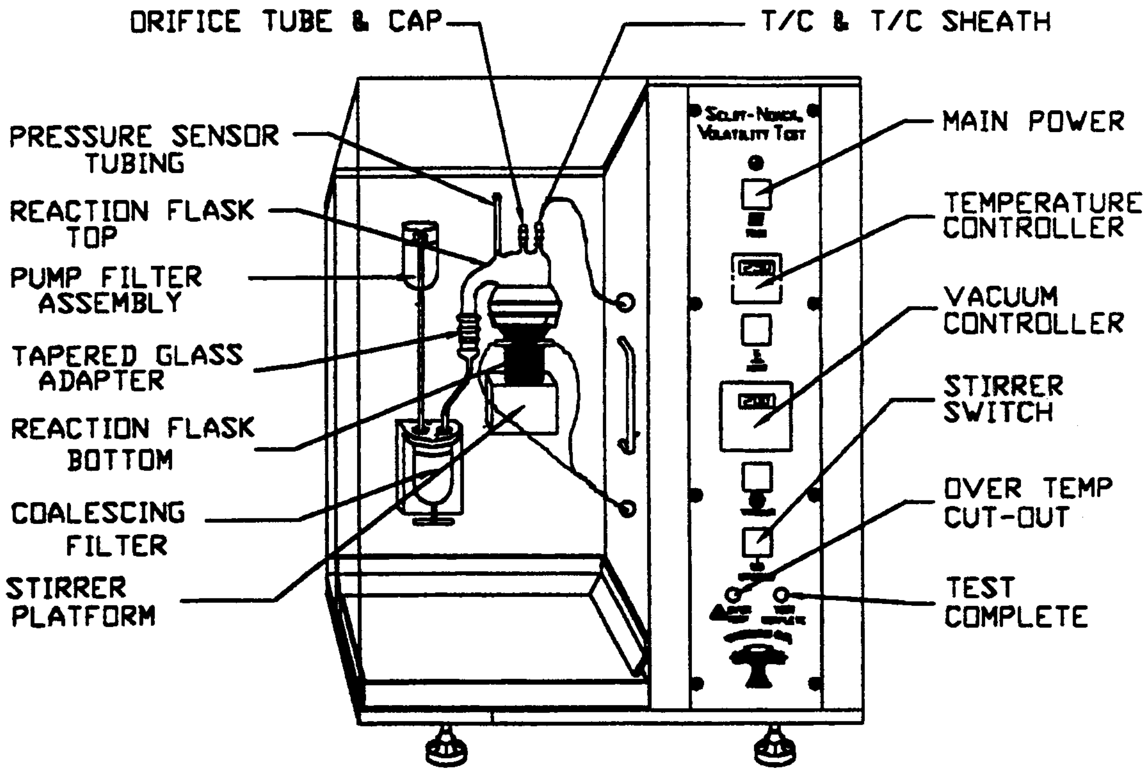


FIG. 13 Selby-Noack Test Equipment

be converted to an equivalent result on the basis of the other procedure. The appropriate equation from those above should be selected and applied according to the type of the sample tested (formulated engine oil or basestock).

23.3 Report the converted result from Procedure A to B, or Procedure B to A to the nearest 0.1 m % as evaporation loss of the test sample as converted from the original procedure to the calculated basis procedure.

23.4 If the nature of the test specimen is not known as being either a basestock or a formulated engine oil, then the results of the test using D 5800 B must be identified as being run under D 5800 B and the value of percent evaporation so obtained will require additional information on the nature of the test specimen for calculations to be made to generate the standard value produced by D 5800 A.

23.4.1 Converted results should be reported as D 5800 A (converted from the results obtained by D 5800 B) or as D 5800 B (converted from the results obtained by D 5800 A).

24. Precision and Bias

NOTE 11—Equipment available from ISL, BP 40, 14790 VERSON - France was used to develop the precision statement for Procedure B.[4]

24.1 To estimate the precision of Procedure B, the test results from the interlaboratory study were analyzed following Practice D 6300.

24.2 The interlaboratory study included eight oils, two base oils, and six finished oils, tested in twelve laboratories.

24.3 The precision of this test method, as determined by the statistical examination of the interlaboratory study test results, is as follows:

24.3.1 *Repeatability*—The difference between two tests results obtained by same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, exceed the following value in only one case in twenty.

$$\text{Repeatability} = 0.095X^{0.5} \quad (10)$$

where

X = average of the two determinations under consideration.

24.3.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test materials would, in the long run, exceed the following value in only one case in twenty.

$$\text{Reproducibility} = 0.26X^{0.5} \quad (11)$$

where

X = average of the two determinations under consideration.

24.4 *Bias*—Since there is no accepted reference suitable for determining the bias for this procedure, no statement on bias is being made.

Procedure C Selby-Noack Volatility Test

25. Apparatus [6]

25.1 Selby-Noack Instrument, (see Fig. 13) including glassware and other parts as follows:

25.1.1 Reaction Flask Bottom, capable of heating a sample quickly to test temperatures of 250°C.

25.1.2 Reaction Flask Top.

25.1.3 Coalescing Filter.

25.1.4 Pump Filter.

25.1.5 Orifice Tube.

25.1.6 Orifice Cap, of a set of incremental sizes.

25.1.7 Thermocouple Sheath.

25.1.8 Magnetic Stir Bar.

25.1.9 Thermocouple.

25.1.10 Measuring Rod, of known length.

25.1.11 Viton O-rings.

25.1.12 Coalescing Filter Cartridges.

25.1.13 Pump Filter Cartridges.

25.2 Balance, capable of weighing at least 300 g to the nearest 0.01 g.

25.3 Beaker, 600 mL.

25.4 Cork Ring, capable of supporting collection flask during weighing.

25.5 Digital Timer.

25.6 Tapered Glass Adapter.

26. Reagents and Materials

26.1 Cleaning Solvent, such as VarCleen, capable of removing varnish from glassware

26.2 Hydrocarbon Solvent, such as hexane.

26.3 Average Volatility Reference Oil.

26.4 High Volatility Reference Oil.

26.5 Pump Oil, suitable for vacuum pump installed in instrument.

27. Preparation of Apparatus

27.1 Locate the instrument where there is minimal pressure fluctuation or draft. Vent the vacuum pump outlet to a suitable exhaust.

27.2 Set the height of the outlet of the orifice tube 11.4 cm above the inside bottom of the reaction flask.

27.2.1 Measure the length of the measuring rod and the length of the orifice tube (without the cap) in centimetres.

27.2.2 Add the measured length of the orifice tube without the orifice cap to 11.4 cm.

27.2.3 Use the value obtained in 27.2.2 to set the length of the measuring rod by affixing the collar at that height. This will give the desired value from the lower tip of the measuring rod up to the bottom of the collar.

27.2.4 Turn the tapered plastic piece on the orifice tube toward the top of threaded section and be sure the orifice cap is removed.

27.2.5 Temporarily assemble the top and bottom pieces of the reactor and put the orifice tube in position.

27.2.6 Insert the measuring rod through the orifice tube. When the lower end of the rod is in contact with the reactor bottom, the lower surface of the collar should be above the upper end of the orifice tube.

27.2.7 Adjust the tapered plastic piece on the orifice down to achieve very light contact between the upper end of the orifice tube and the lower surface of the collar on the measuring rod to establish the proper position.

27.3 Insert the two thermocouple connectors into the thermocouple receptacles on the cabinet.

27.4 Insert the heater cable into the heater connection on the cabinet.

27.5 Be sure that the inside of the reactor bottom is clean (see 30.5) and that the other glassware, hardware, and tubing are free of any oil residue.

27.6 If collection of the volatilized oil is desired, clean the coalescing filter housing with a hydrocarbon solvent, dry, and install new filter cartridge.

27.7 Turn on the main power switch located on the front panel.

27.8 Before operating the instrument close the shield door.

28. Calibration

28.1 Calibrate the thermocouple at 100°C or higher against a certified thermometer or other standard temperature measuring device and, if necessary, adjust the calibration offset on the temperature controller according to the manufacturer's instructions.

28.2 Preheat reaction flask bottom by placing the clean flask (with magnetic stirrer inside) on the reaction flask platform. Place the thermocouple inside the flask and be sure that the tip is firmly touching the inside wall. Set the temperature controller for 100°C and turn on the heater switch. When the temperature reaches 100°C, start the timer for 5 min.

28.3 After the 5 min has expired, turn off the heater switch and wait for the flask to cool below 50°C. After cooling, check the dry weight of the reaction flask bottom against the weight of previous weighings.

NOTE 12—The sequential weighings should be reasonably constant (that is, within ±0.02 g) although the weight will decrease in a regular way over days and months of use. Any sudden increase in weight or erratic fluctuation is an indication that the outside insulating coating of the bottom reaction flask has been contaminated and the flask should be repeatedly baked according to 28.2 until constant weight is reestablished.

28.4 Weigh the flask with the stirring bar and record the value to the nearest 0.01 g.

28.5 Pour 65 g ± 0.02 g of reference oil into the reaction flask. Record the mass of the oil to the nearest 0.01 g.

28.6 Insert the appropriate orifice cap size into orifice tube making certain there is a good seal. Insert the orifice tube (with the orifice cap) into the center glass tapered joint on the reaction flask top and make certain it has seated properly.

[6] The sole source of supply of the apparatus known to the committee at this time is Tannas Co., 4800 James Savage Rd., Midland, MI 48642. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee[1], which you may attend.

28.7 Insert the thermocouple sheath, without the thermocouple, into the thermocouple connection on the reaction flask top with the bend facing away from the glass sidearm.

28.8 Join the reaction flask top and bottom and insert the assembly into the flask clip provided.

28.9 Raise the stirrer platform to stabilize the reaction flask bottom.

28.10 If collection of volatiles is desired, weigh the clean coalescing filter assembly including the filter cartridge and record the results to the nearest 0.01 g.

28.11 Mount to bracket with the arrow pointing in the direction of air flow (toward the vacuum pump).

28.12 Connect the inlet coalescer tubing to the barbed end of the tapered glass adapter (which will be later connected to the reaction flask top sidearm).

28.13 Connect the tubing to the input side of the coalescing filter and connect the tapered glass adapter to the reaction flask top sidearm and secure with a glass joint clamp.

28.14 Connect the vacuum hose from the pump filter (above the coalescing filter) to the output connection on the coalescing filter.

28.15 Connect the hose from the pressure sensor to the barbed connection on the reaction flask top next to the sidearm. After connections are made, check that the reaction flask top is horizontal with the reaction flask bottom and that the O-ring seal between them is in proper position.

28.16 Slide the thermocouple into the thermocouple sheath in such a manner that the plastic tubing seals the joint between the thermocouple and the thermocouple sheath. This will ensure no leaks.

28.17 Make certain that the thermocouple contacts the wall. This can be viewed by looking down through the top of the reaction flask top. It is important that the thermocouple touches the inside wall of the reaction flask at least 0.5 in below the surface of the oil for proper results.

28.18 With the vacuum control Open/Close switch set to the middle or Flow position, turn on the Vacuum On/Off switch on the upper console.

28.19 Press down the Gauge Factor/Flow/Set Pt. switch and, using the Set Point dial, adjust the vacuum to 2.00 cm (20 mm) of water on the meter readout. When at a reading of 2.00, release the Gauge Factor/Flow/Set Pt. switch.

28.20 Press one finger tightly over the hole in the orifice cap and toggle the vacuum control Open/Close switch to the closed position. The vacuum reading should increase to a stable value (this must be below 19.00 cm of water). Once stabilized and with finger still in place, turn the vacuum pump off and monitor the vacuum to observe how well it maintains the vacuum. Remove finger from the orifice cap and again monitor the vacuum; it should quickly decrease to a zero reading.

28.21 If there is a leak, recheck all of the glass fittings, the orifice tube and the thermocouple sheath for proper seating. Repeat step 28.20. If there is no leak then continue to 28.22.

NOTE 13—High temperature vacuum grease may be used to seal leaks, if necessary. Vacuum grease should not be used on components that are weighed during the test procedure.

28.22 With the vacuum switch already on and controlling at 2.00 cm of water, turn on the stirrer. Visually verify the stirring.

28.23 Set the temperature controller to 250°C and turn on the heater switch. An automatic timer will activate when the heater switch is turned on. The timer will turn off the heater, vacuum, and stirring motor after 1 h.

28.24 Allow the unit to cool down to a temperature safe for handling (approximately 20 min). Gloves may be used to handle while hot.

28.25 Weigh the volatilized oil (if desired).

28.25.1 Remove the tubing from the coalescing filter assembly and remove the assembly from the bracket.

28.25.2 Weigh the entire coalescing filter assembly and record the results to the nearest 0.01 g. Subtract previous dry assembly weight from 28.10 to obtain the weight of volatilized oil.

28.25.3 Save the volatilized oil, if desired, by draining out through the petcock in the bottom of the coalescing filter housing.

28.26 Remove the pressure sensor tube and the thermocouple, and then remove the reaction flask top. The orifice tube and thermocouple sheath may be left in position during cleaning and reassembly. (If any oil is clinging to the sheath tip, touch it to the side wall of the reactor bottom to return it to the reaction flask.)

28.27 Weigh and record the reaction flask bottom plus remaining residual oil to the nearest 0.01 g and either discard or save the residual oil for further analysis.

28.28 Clean all glassware and hardware as described in 30.1-30.7.

28.29 Calculate the percent volatility loss of the reference fluid to the nearest 0.1 % (see Section 31).

28.30 Compare the result to the given value of the reference fluid. If results are within limits, proceed to the Sample Procedure.

28.31 If the result is not within the limits of the reference fluid check that the procedure has been followed and that the apparatus is set up properly with no leaks. Check the calibration of the temperature controller and pressure sensing device.

NOTE 14—Procedures for calibration of the temperature controller and pressure controller can be found in the Operations Manual for the Selby-Noack Volatility test.

28.32 If no errors in setup or procedure are identified, proceed to changing the size of the orifice cap. The size should be changed in increments of 0.001, with each change corresponding to a directly related change of 0.3 % evaporation loss. The orifice chosen should be of the smallest size giving accurate results. Rerun the test on the reference fluid after making any changes.

NOTE 15—If using new glassware, or approximate orifice size is unknown, begin with an 0.084 orifice and increase until good results are achieved.

28.33 If problems persist, contact the instrument manufacturer.

29. Sample Procedure

29.1 Perform steps 28.4-28.28 substituting a test sample for the reference fluid.

29.2 Calculate the percent evaporation loss of the test sample to the nearest 0.1 %.

29.3 Collect the volatilized oil (if desired for further analysis) from the coalescing filter by placing a small container under the filter petcock and opening it to release the oil.

NOTE 16—It is recommended that a reference fluid be tested to confirm calibration at the beginning of each series of sample tests and at the beginning of every other day of continuous testing. If testing is not conducted on a daily basis, test the reference fluid at the beginning of each test day. If the percent evaporation loss of the reference fluid is not within limits, check the instrument for operating precision or re-calibrate before samples are tested, or both.

30. Cleaning

30.1 Clean all of the glassware and hardware, with the exception of the reaction flask bottom, with a suitable hydrocarbon solvent (for example, hexane, heptane, cyclo-hexane).

30.2 If the coalescing filter is to be used for collecting volatilized oil, it can be cleaned while disassembled. Unscrew the collection cup, remove the filter cartridge, and clean with a suitable hydrocarbon solvent (for example, hexane, heptane, cyclo-hexane). Upon reassembly, the filter cartridge can be replaced with a new, clean cartridge. The filter can also be removed from the bracket, if necessary, for cleaning.

30.3 In order to prevent oil from coming into contact with the outside of the reaction flask bottom, wrap the outside lip of the reaction flask bottom with clean toweling and quickly pour out the contents into a beaker in order to catch the stirrer bar. While still holding the flask upside down remove the towel, wipe the lip dry, and then carefully rinse the inside of the flask with a hydrocarbon solvent. When the unit is clean, wipe it dry with another towel.

NOTE 17—If the insulating covering of the reaction flask bottom becomes contaminated, follow the directions in Note 12.

30.4 If oil is spilled on the outside insulation of the reaction flask bottom, immediately rinse the oil off with a suitable hydrocarbon solvent. Exercise care not to spread the oil to other areas of the insulation. When all oil has been removed, dry the reaction flask bottom with an air source. The flask should be allowed to completely; dry at room temperature before continuing with the test procedure.

30.5 Place 10 mL of a varnish removing solvent into the reaction flask bottom. Insert clean paper towel into the solvent and wipe inside of flask thoroughly, removing any varnish that may be present on the wall. Rinse carefully with hot water and dry.

30.6 If other glass parts develop a varnish film, clean these with the same procedure as indicated in 30.5 or put the parts into a half/half water or full-strength solution of varnish remover overnight.

30.7 With a towel dampened with varnish removing solvent, clean the end of the thermocouple. Wipe with towel dampened with hot water and dry to remove any remaining cleaning solvent.

31. Calculation

31.1 Percent volatility is determined by mass loss found by subtracting the combined weight of the flask bottom and oil after testing (see 28.27) from their combined weight before the test (see 28.4 and 28.5).

NOTE 18—The percent volatility is obtained by taking the mass loss of the reaction flask and dividing that by the exact mass of the test oil sample recorded earlier: that is if weight of the oil sample is 65.1 g and weight of the oil lost is 10.2 g, then (10.2 g/65.1 g) × 100 = 15.67 %.

31.2 The mass of volatiles collected is obtained by subtracting the coalescing filter assembly weight before the test (see 28.10) from its weight at the end of the test (see 28.26).

NOTE 19—The percent of volatiles collected is obtained by dividing the mass of volatiles collected by the mass loss: that is, If the weight of the empty coalescing filter assembly is 163.2 g and the weight of the filter assembly after volatilization gained 9.8 g, and if the weight loss shown by 31.1 is 10.2 g, then (9.8 g/10.2 g) × 100 = 96.08 % of the volatiles were collected.

32. Report

32.1 Report the evaporation loss to the nearest 0.1 %.

32.2 Limited amount of data available shows that Procedures A and C give similar results for formulated engine oils. However, no comparative data is available for the basestocks. Further work is necessary to quantitate the relationship.

33. Precision

NOTE 20—The equipment listed in the research report,[4] was used to develop this precision statement. This is not an endorsement or certification by ASTM International.

33.1 The interlaboratory study included six test oils tested on eight apparatus with eight different operators. The samples measured in the study were engine oils covering the range of 11.84 % loss to 20.18 % loss.

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

$$\text{Repeatability, \% evaporation loss} = 0.81 \quad (12)$$

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

$$\text{Reproducibility, \% evaporation loss} = 1.62 \quad (13)$$

34. Quality Control for Procedures A, B, and C

34.1 Procedures A, B, and C require confirmation of the performance of the apparatus by analyzing a quality control (QC) sample.

34.2 Prior to monitoring the measurement process, the user of the test method needs to determine the average value and control limits of the QC sample. (See Practice D 6299 and MNL 7.[7])

34.3 Record the QC results and analyze by control charts or other statistically equivalent techniques to ascertain the statistical control status of the total testing process. (See Practice

[7] ASTM MNL7, *Manual on Presentation of Data Control Chart Analysis*, 6th ed., Section 3, Control Chart for Individuals, ASTM International, W. Conshohocken, PA 19428.

D 6299 and MNL 7.[7]) Any out-of-control data should trigger investigation for root cause(s). The results of this investigation may, but not necessarily, result in instrument recalibration.

34.4 The frequency of QC testing is dependent on the demonstrated stability of the testing process, customer requirements and the recommendations of the equipment manufacturer. The QC sample precision should be periodically checked against the ASTM test method precision to ensure data quality.

34.5 It is recommended that, if possible, the type of QC sample that is regularly tested be representative of the samples routinely analyzed. An ample supply of QC sample material should be available for the intended period of use and must be homogeneous and stable under the anticipated storage conditions.

35. Keywords

35.1 evaporation loss of lubricants; Noack volatility; volatility of lubricants

APPENDIXES

(Nonmandatory Information)

X1. HELPFUL HINTS FOR NOACK VOLATILITY (PROCEDURES A and B)

X1.1 Be sure to use the correct manometer fluid to fill the manometer. The density of the fluid is critical and must be of the type designed for the manometer (see 6.11, Note 1). Millimetres of water (implicitly at 1 G) is a unit of pressure. Not every manometer that gives readings in millimetres of water is made for use with water as the manometer fluid. Consult the manual or manufacturer for the correct manometer fluid properties.

X1.2 Be sure that the manometer reservoir is filled so that the manometer is reading exactly zero with no external vacuum or pressure. This should be checked before each run. Evaporation may require occasional refilling of the manometer reservoir. It is also important that the unit be properly leveled.

X1.3 For inclined manometers, be sure to read the meniscus at the same position at both 1 and 20 mm of water.

X1.4 Rubber tubes used for connections should be changed periodically because oil-mist causes rubber swelling after extended period of service.

X1.5 Cleaning at regular intervals of foreign material adhering on exhaust system parts, particularly on Y-glass piece, is necessary.

X1.6 There is a drop in metal bath temperature when inserting the sample. Monitor that the temperature recovers in approximately 3 min.

X1.7 Strong air drafts or turbulence around the pressure transducer or the heated crucible may adversely affect the test precision and accuracy. Do not place the apparatus in a draft area; however, the exhaust fumes from the evaporating oil shall be ventilated to an outside source.

X1.8 Clean the crucible and the cover thoroughly with solvent between tests and allow to dry. Remove stubborn lacquer by immersing in hot detergent solution, by light abrasion with fine carborundum powder, or a fine abrasive pad.

X1.9 Vacuum must be accurately set and maintained or the Noack values can be greatly altered. Run the pump for 30 min before testing. Vacuum pump should be cleaned out daily using a hydrocarbon solvent (consult manufacturer for a compatible solvent recommendation). Run a pressure test daily; let the vacuum run until the pressure stabilizes.

X1.10 Condensed liquid collecting in tubes and at junctions is also a common source of problem.

X1.11 The CEC Reference Oil, RL-172, should be analyzed each day the samples are analyzed. Make sure the correct reference oil performance certificate is being used. Some suppliers may not be correctly updating their reference oil performance certificates. Note that the evaporation loss values differ for Procedures A and B.

X1.12 Cleanliness of the extraction tube, glass and silicone tubing, and air jets should be assured prior to all tests.

X1.13 Possible contamination of the thermal sensor well with slag and Woods metal should be monitored. Slag should be checked and removed periodically after a series of runs.

X1.14 Check all connections to be sure they are tight before test. Alignment of all connections without any restrictions should be maintained. All tubing should allow all flow to travel downhill to the vacuum pump (no low points).

X1.15 Verify that the temperature probe holder spring is working properly to seat the probe correctly. The temperature probe should be cleaned to remove varnish.

X1.16 On automatic machines, if pressure reads different from zero before testing, recalibrate the pressure.

X1.17 If extraction tube is loose, tighten or check the gasket.

X1.18 Woods metal bath must be full and overflowing around crucible and thermal sensor wells.

X1.19 At all times while handling the sample crucible, be careful not to splash the test sample on the crucible lid, especially when removing the lid. Use of a table-mounted holder to hold the crucible may help prevent splashing while operating the crucible (see 20.10).

X1.19.1 Do not overtighten the crucible lid, and do not use the extraction tube as a handle to tighten or open the lid.

X1.20 *Timing*—Place the crucible in the bath, connect the vacuum and start timer in quick succession, as nearly simultaneously as possible.

X1.21 Start the pump before starting the stopwatch for the test. Instrument electronics must be on for at least 30 min prior to the start of the first test to warm up the vacuum transducer. Leaving the electronics on overnight satisfies this recommendation.

X1.22 Monitor calibration thermometer versus recorded temperature occasionally during run. The temperature circuit electronics should be verified at least monthly using a calibrated temperature probe simulator.

X1.23 *End of Test*—Disconnect vacuum at the end of the test time and place the crucible in cooling bath within 1 min.

X1.24 Be careful not to tilt the crucible in handling during the test, particularly at the end.

X1.25 Final weighing should not be done until the crucible is at room temperature. Do not use extra force (for example, hammers) to open or close the crucible.

X1.26 At the end of the test, check the pressure and temperature scans from automatic machines to see that proper parameters were maintained during the runs.

X2. HELPFUL HINTS FOR SELBY-NOACK VOLATILITY (PROCEDURE C)

X2.1 Be sure that the thermocouple is touching the side of the reaction flask bottom. This can be accomplished by looking down through the reaction flask top when positioning the thermocouple.

X2.2 To reduce potential flow problems, make certain that the hose from the unit to the exhaust hood or vent is not pinched anywhere.

X2.3 To prevent leaks, securely seat all glassware, the thermocouple sheath, and the orifice tube.

X2.4 For accurate results, make certain that the test runs as close to 1 h as possible (within 15 s).

X2.5 At the end of the test, the glassware may be removed for the 20-min cool-down period using thermal gloves. This permits immediate starting of another run with a second set of glassware.

X2.6 Alternatively, two sets of top and bottom flasks may be used each with its own orifice tube and cap.

SUMMARY OF CHANGES

Subcommittee D02.06 has identified the location of selected changes to this standard since the last issue (D 5800–03a) that may impact the use of this standard. (Approved May 1, 2004.)

(1) Updated Appendix X1.

Subcommittee D02.06 has identified the location of selected changes to this standard since the last issue (D 5800–03) that may impact the use of this standard. (Approved Dec. 10, 2003.)

(1) Revised Section 1 to mention differences in the results obtained by Procedures A and B.
(2) Added 22.2 to provide correlation equations between results by Procedures A and B.
(3) Updated Section 23 to include new reporting protocols.
(4) Added 32.2 to discuss the results obtained by Procedures A and C.

Harris Testing Laboratories, Inc.

CERTIFICATE OF ANALYSIS

CLIENT
Megatrol Inc.
9469 S 500 W
Sandy, UT 84070

PRODUCT: FE^{26} Premier
MARKS: NONE
DATE RECEIVED: 02/11/2008
LAB NO: HH0410-1202-P
SUBMITTED BY: Jon Rose

			SPECIFICATIONS	
METHOD	TEST	RESULT	MIN.	MAX.
Ethyl Corp.	Lead Corrosion		Clear	Clear
Lead Corrosion	Lead, ppm	13 ppm	XXX	XXX

Comments:

- 2% additive by weight in Drakeol 600 White Mineral Oil USP.
- Failure of the product is 30.0 ppm or greater.

Date issued:
02/11/2008

Sassan Badr

CHEMIST

Ethyl™
CORPORATION

Lead Corrosion Test Procedures: "This is an Ethyl Corp Test Procedure"

"Testing for EMD Engines for after market oil additives"

1. Conditions with which to test any potential component for medium speed diesel EMD Series Engines (additive applications)

2. Additive concentration: 2%w in solvent neutral mineral oil, viscosity range preferred is 12 - 15 cSt @ 100 C

3. Put 65 grams of the above additive solution in a 150 ml Pyrex beaker. Heat the oil @ 250 degrees Fahrenheit on a hotplate and stir magnetically at 200rpm.

4. Prepare a lead test coupon by cleaning a 1" square piece of sheet lead, 1/16" or 1/32" thick with steel wool to remove accumulated lead oxide. Drill small hole in one side of the test piece and suspend the test piece from an inert thin nichrome wire attached to a glass rod or wooden stick.

5. When the oil is at temperature, suspend the lead test piece so it is entirely immersed in the oil. Hold at temperature with stirring for 50 hours.

6. At the end of 50 hours, analyze the used oil for dissolved lead by atomic absorption or more preferably ICP.

Conclusion:

The lead content of the test oil should be less than 30 ppm.

Test Solution only for base line reading

Sample Code: TMC 1006 (NEAT)
Test Method: CEC L-39-T-96
Test Duration: 168 hrs.

Elastomer	Test Temperature, C	Volume Change, %	Points Hardness Change	Tensile Strength Change, %	Elongation Change, %
RE1 (08/03)	150	2.9	1	-41	-53
Fluoroelastomer		3.0	1	-39	-52
		3.0	0	-39	-52
Average		3.0	1	-40	-52
RE2 (04/03)	150	5.6	1	2	-34
Polyacrylate		5.4	1	3	-32
		5.4	0	8	-30
Average		5.5	1	4	-32
RE4 (05/03)	100	5.7	-2	-24	-46
Nitrile		5.7	-3	-26	-46
		5.7	-3	-25	-46
Average		5.7	-3	-25	-46

Rebecca D. Grinfield, Research Scientist

Test Solution mixed with FE26 Premier

FE26 Premier

Sample Code: FE26 Premier/TMC 1006
Test Method: CEC L-39-T-96
Test Duration: 168 hrs.

Elastomer	Test Temperature, C	Volume Change, %	Points Hardness Change	Tensile Strength Change, %	Elongation Change, %
RE1 (08/03)	150	1.8	-1	-24	-32
Fluoroelastomer		1.6	-1	-25	-36
		1.5	0	-26	-36
Average		1.6	-1	-25	-35
RE2 (04/03)	150	5.3	-1	14	-20
Polyacrylate		5.3	-2	7	-23
		5.3	-1	14	-19
Average		5.3	-1	12	-21
RE4 (05/03)	100	9.6	-6	-10	-38
Nitrile		9.7	-7	-14	-32
		9.5	-6	-11	-30
Average		9.6	-6	-12	-33

Rebecca D. Grinfield, Research Scientist

SAE International™ **SURFACE VEHICLE STANDARD**

SAE J2643

ISSUED Prop Dft MAY2003

Issued Proposed Draft 2003-05

Standard Reference Elastomers (SRE) for Characterizing the Effect of Liquids on Vulcanized Rubbers

Foreword—The development of these standard reference elastomers (SRE) is based on static and dynamic engine oil sealing applications for passenger cars and light duty trucks.

1. Scope

Scope—This SAE Standard specifies requirements for vulcanized rubbers in sheet form for use as standards in characterizing the effect of test liquids and service fluids. The annexes contain the standard reference elastomer formulas.

The property changes of the SRE in contact with the indicated fluid under specified test conditions are the responsibility of the user. See 7.3 and Table 1.

This standard is not designed to provide formulations of elastomeric product compositions for actual service.

1.1 Safety—This standard may involve hazardous materials, operations and equipment. It does not address the safety concerns which may be associated with its use. It is the responsibility of any user of this standard to consult and establish appropriate health and safety practices, and determine the applicability of regulatory limitations before use.

2. References

2.1 Applicable Publications—The following publications form a part of this standard to the extent specified herein. The latest issue of all standards shall apply.

2.1.1 ASTM PUBLICATIONS—Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

ASTM D 412—ASTM for Vulcanized Rubber and Thermoplastic Elastomers-Tension
ASTM D 471—STM for Rubber Property - Effect of Fluids
ASTM D 1349—Standard Practice for Rubber – Standard Temperatures for Testing
ASTM D 1418—Standard Practice for Rubber and Rubber Latices – Nomenclature
ASTM D 3182—Standard Practice for Rubber Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets
ASTM D 3187—Standard Test Methods for Rubber – Evaluation of NBR (Acrylonitrile-Butadiene Rubber)
ASTM D 3767—Rubber – Measurement of Dimensions
ASTM D 4678—Standard Practice for Rubber – Preparation, Testing Acceptance, Documentation, and Use of Reference Materials

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Tel: 724-776-4970 (outside USA)
Fax: 724-776-0790
Email: custsvc@sae.org
SAE WEB ADDRESS: http://www.sae.org

2.1.2 ISO PUBLICATIONS—Available from ANSI, 25 West 43rd St., New York, NY 10036-8002.

1629—Rubber & latices – Nomenclature
2230—Vulcanized rubber property – Guide to storage

3. SRE Composition

3.1 The recommended mixing procedures and specified properties are from the identified materials in the annexes.

3.2 The compounding and preparation ensure the property profile;

a. agrees sufficiently with the material group and applications it represents;
b. exhibits reasonable "sensitivity" to designated fluid additive and base stock changes;
c. is consistent for reliable reproducibility.

3.3 SRE compound materials must be readily available worldwide.

4. Summary of Preparation

4.1 Each compound shall be a homogeneous mix of all materials shown in the annexes, weighed to the accuracy required in ASTM D 3182.

4.2 The mixing of compounds and vulcanization of test sheets follow ASTM D 3182, modified to details given in each annex condition and procedure.

4.3 **Approved Mixing and Distribution Facilities—**See Appendix.

4.3.1 An SRE by definition is mixed only by approved designated facilities authorized to mix, and distribute these compounds. See 4.3.2.

4.3.2 APPROVED FACILITY—Akron Rubber Development Lab, 2887 Gilchrist Road, Akron, OH 44305.

4.3.3 Labs can mix approved SRE formulations for internal use only.

5. Requirements and Testing

5.1 **Sheet Dimensions—**The vulcanized sheets meet ASTM D 3182 figure 1 mold cavity dimensions measured to ASTM D 3767.

5.1.1 All sheets are tested for thickness. One test sheet from each lot (all sheets from a single batch vulcanized under the same conditions) is tested for the specified properties for compliance with the tolerances given in the annexes.

5.2 **Sheet Appearance—**The molded sheet must be free of any surface defects or internal voids observed with normally corrected vision.

5.3 **Identification—**Using mold marks or visible, durable ink, mark each sheet along one edge with the ASTM D 1418 / ISO 1629 nomenclature letters with SRE formulation number, mix number, lot number, and grain direction (arrow). Separate each item with a slash (/) mark (see Figure 1).

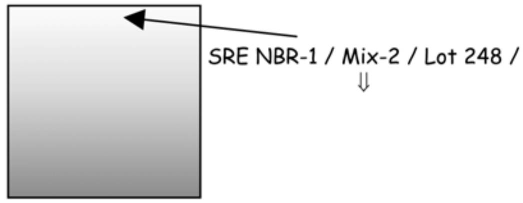

FIGURE 1—Identification Protocol of Test Slabs Example

5.4 Lot Testing—A sufficient number of molded test sheets from each lot (all sheets from a single batch vulcanized under the same conditions) is tested for compliance to specified properties using the appropriate test (see item 7.1).

5.4.1 Each shipped lot will include the approved lab(s) certification that the material complies with its annex requirements.

5.4.2 Test specimens are cut 15mm in from the sheet edge, with the longitudinal axis parallel to the sheet grain direction.

5.4.3 Test 5 specimens for each physical property, and 5 specimens for volume change.

6. Storage

6.1 Test sheets are stored in accordance to ISO 2230. After one year they are retested or discarded.

6.2 When the composition of any SRE is revised or changed, authorized lab(s) shall not discard stored test sheets for that SRE until new changes are approved by SAE Committee on Automotive Rubber Specifications (CARS).

6.3 In the event SRE changes are made, do not distribute the previous SRE formulation, except for comparative purposes. Authorized lab(s) will phase in the revised SRE when changes are approved.

7. Application

7.1 Development of SRE—Candidate selection (sensitivity) is based on percent change per ASTM D 412, die C, tensile strength, elongation at break, and tensile stress at 50%. ASTM D 471 percent volume change rounds out the requirements. Hardness measurements may be made but evidence from the Inter Lab Test program indicates that hardness is much less sensitive to differences in "rubber stiffness" compared to 50% modulus.

7.1.1 Test conditions reflect OEM engine oil system requirements and the material classification limits. Test temperature conditions follow ASTM D 1349 practice. To achieve equilibrium, immerse test specimens 168 h or longer (as designated).

7.1.2 For each material classification, the tester should be aware of the effect of aeration on a candidates physical properties during immersion testing. Use the fluid treatment (static or aerated) most representative of the intended use. Appendix tables are based on static immersion.

7.2 Test Fluid—Use ASTM Service Fluid 105 to evaluate SRE candidate selection and establish a property loss baseline for engine oil applications using the properties in 7.1. Service Fluid 105 is available from the ASTM Test Monitoring Center, 6555 Penn Ave., Pittsburgh, PA 15206.

7.3 Appendices A-J—Appendices A-J gives all formulations for the selected SRE's of this standard. Also included in the appendices is a Summary Table for SRE properties. Part 1 contains delta or percent change in property values for each SRE after immersion for 168 hr in ASTM Service Fluid 105 at temperatures as specified in Table 1. Part 2 contains the original properties for each SRE prior to immersion. The summary table results were obtained in an interlaboratory test program with all of the SREs as listed in the annex and table, using data from eight typical industry laboratories. Prior to the final analysis as indicated in the Summary Table, outlier values were deleted.

7.3.1 The interlaboratory test program did not contain a sufficiently large database to obtain interlaboratory standard deviations based on 20 or more degrees of freedom, DF. Thus the value equivalent to the classical or standard (±) 3 sigma limit (at the DF applicable to each SRE), is defined in the table as t*SDev, where t is the tabulated 95% confidence or p=0.05 level t value at the DF applicable to the standard deviation, SDev, as evaluated in the test program.

TABLE 1—SRE TEST TEMPERATURE

SRE	Temperature (°C)
NBR-1, NBR-2	100
All others	150

7.4 Appendix K—Appendix K provides protocol for creation, maintenance and control of formulations for the selected SRE's of this standard.

PREPARED BY THE SAE ENGINE OIL COMP. WITH RUBBER GASKETS AND SEALS SUBCOMMITTEE
OF THE SAE COMMITTEE ON AUTOMOTIVE RUBBER STANDARDS

APPENDIX A

A.1 Standard Reference Formulas and Procedures

A.1.1 Nitrile Rubber

A.1.1.1 Standard formulas for the following nitrile rubber compounds are given in Tables A2 and A3:

TABLE A1—

Number	Type
NBR-1	sulfur-cured
NBR-2	peroxide-cured

A.1.1.2 Mixing procedure shall follow the guidelines in Section 5 of ASTM D 3187.

A.1.1.3 RECOMMENDED STANDARD TENSILE SHEET CURES—Cure NBR-1 specimen 10 minutes at 168 °C [335 °F]. Cure NBR-2 specimen 10 minutes at 182 °C [360 °F].

A.1.1.4 COMPOSITION

TABLE A2—COMPOSITION OF SRE NBR-1 (28/S)

Ingredient	Parts by mass
NBR with 28 ± 0.5% by mass of acrylonitrile(1)	100.0
Zinc oxide(2)	5.0
Stearic acid, triple press(3)	2.0
STANGARD 500(4)	2.0
Carbon black N774	70.0
PLASTHALL 7050(5)	5.0
SPIDER Sulfur(6)	0.5
N-Cyclohexyl-2-benzothiazolesulphenamide (CBTS)	1.0
Tetramethyl thiuram disulfide (TMTD)	1.0
Tetraethyl thiuram disulfide (TETD)	1.0
Total	187.5

1. NIPOL DN2850, Zeon Chemicals, Inc. No equivalent known.
2. KADOX 920C, activator, Zinc Corp. of America. No equivalent.
3. C.P. Hall.
4. Blend from Harwick standard.
5. Monomeric plasticizer, C. P. Hall Co. No equivalent.
6. SPIDER S, C.P. Hall. No equivalent.

TABLE A3—COMPOSITION SRE NBR-2 (28/P)

Ingredient	Parts by mass
NBR with 28 ± 0.5% by mass of acrylonitrile(1)	100.0
Zinc oxide(2)	5.0
Stearic acid, triple press(3)	2.0
STANGARD 500(4)	2.0
Carbon black N774	70.0
PLASTHALL 7050(5)	
Dicumyl peroxide, 40% active MA carrier(6)	
	187.0

1. NIPOL DN2850, Zeon Chemicals, Inc. No equivalent known.
2. KADOX 920C, activator, Zinc Corp. of America. No equivalent.
3. C.P. Hall.
4. Blend from Harwick standard.
5. Monomeric plasticizer, C. P. Hall Co. No equivalent.
6. DICUP 40 KE Hercules. Burgess clay carrier. No equivalent.

APPENDIX B

B.1 Polyacrylate Rubber

B.1.1 Standard formulas for the following polyacrylate rubber compounds are given in Table B2:

TABLE B1—

Number	Type
ACM-1	sodium stearate-cured

B.1.2 Mixing procedure shall follow the guidelines in Section 5 of ASTM D 3187.

B.1.3 Recommended Standard Tensile Sheet Cures—Cure ACM-1 specimen 10 minutes at 168 °C [335 °F].

B.1.4 Composition

TABLE B2—COMPOSITION OF SRE ACM-1

Ingredient	Parts by mass
Acrylic rubber(1)	100.0
Stearic acid	1.0
NAUGARD 445(2)	2.0
Carbon black N550	80.0
Sodium stearate	4.0
conc. fatty acid ester(3)	2.0
NPC-25(4)	4.0
Total	191.0

1. HYTEMP 4052, Zeon Chemicals, Inc. No equivalent known.
2. Substituted diphenyl amine, antioxidant, Uniroyal Chemical Co., Inc. No equivalent.
3. Process aid, Strukol WB222. No equivalent.
4. Vulcanizing agent, Zeon Chemicals, Inc. No equivalent.

APPENDIX C

C.1 Epichlorohydrin Rubber

C.1.1 Standard formulas for the following epichlorohydrin rubber compounds are given in Table C2:

TABLE C1—

Number	Type
ECO-1	ETU-cured

C.1.2 Mixing procedure shall follow the guidelines in Section 5 of ASTM D 3187.

C.1.3 Recommended Standard Tensile Sheet Cures—Cure ECO-1 specimen 25 minutes at 191 °C [375 °F]. Post cured 4 h at 177 °C [350 °F].

C.1.4 Composition

TABLE C2—COMPOSITION SRE ECO-1

Ingredient	Parts by mass
Hydrin C2000L(1)	100.0
Carbon black N550	70.0
TP95(2)	10.0
Stearic acid	1.0
4- and 5-methylmercaptobenzimidazole(3)	0.5
NAUGARD 445(4)	1.0
MAGLITE D	3.0
GND-75 (ETU) (5)	1.0
Total	186.5

1. HYDRIN C2000L, Zeon Chemicals, Inc. No equivalent known.
2. Plasticizer, Rohm & Haas. No equivalent.
3. VULKANOX MB-2/MG antioxidant, Bayer AG. No equivalent.
4. Substituted diphenyl amine, antioxidant, Uniroyal Chemical Co., Inc. No equivalent.
5. Ethylene thiourea, accelerator, 75% active.

APPENDIX D

D.1 Ethylene Acrylic Rubber

D.1.1 Standard formulas for the following ethylene acrylic rubber compounds are given in Table D2:

TABLE D1—

Number	Type
AEM-1	DOTG- diamine cured

D.1.2 Mixing procedure shall follow the guidelines in Section 5 of ASTM D 3187.

D.1.3 Recommended Standard Tensile Sheet Cures—Cure AEM-1 specimen 10 minutes at 177 °C [350 °F] Post cure: 4h at 177 °C [350 °F].

D.1.4 Composition

TABLE D2—COMPOSITION SRE AEM-1

Ingredient	Parts by mass
VAMAC® G(1)	100.0
NAUGARD 445(2)	2.0
Stearic acid	1.5
ARMEEN 18D	0.5
VANFRE VAM(3)	1.0
FEF Carbon black N550	60.0
DIAK #1(4)	1.5
di-o-tolylguanidine (DOTG)	4.0
Total	170.5

1. VAMAC® a registered tradename for ethylene/acrylic rubber from DuPont. No equivalent known.
2. Substituted diphenyl amine, antioxidant, Uniroyal Chemical Co., Inc. No equivalent.
3. Organic phosphate ester free acid, processing aid, R. T. Vanderbilt Co., Inc. No equivalent.
4. Accelerator, DuPont. No equivalent.

APPENDIX E

E.1 Hydrogenated Nitrile Rubber

E.1.1 Standard formulas for the following hydrogenated nitrile rubber compounds are given in Table E2:

TABLE E1—

Number	Type
HNBR-1	peroxide-cured

E.1.2 Mixing procedure shall follow the guidelines in Section 5 of ASTM D 3187.

E.1.3 Recommended Standard Tensile Sheet Cures—Cure HNBR-1 specimen 10 minutes at 177 °C [350 °F]. Post-cure none.

E.1.4 Composition

TABLE E2—COMPOSITION OF SRE HNBR-1

Ingredient	Parts by mass
HNBR with 36 ± 0.5% by mass of nitrile groups and £5% of residual double bonds(1)	100.0
Zinc oxide(2)	5.0
Stearic acid	0.5
NAUGARD 445(3)	1.5
Zinc 2-mercapto-toluimidazole(4)	1.0
Carbon black N774	50.0
PLASTHALL TOTM(5)	5.0
40% a,a'-bis-(t-butyl peroxy) diisopropylbenzene on Burgess KE Clay(6)	8.0
Total	171.0

1. ZETPOL 2010, Zeon Chemicals, Inc. No equivalent known.
2. KADOX 911C, activator, Zinc Corp. of America. No equivalent.
3. Substituted diphenyl amine, antioxidant, Uniroyal Chemical Co., Inc. No equivalent.
4. VANOX ZMTI, antioxidant, R. T. Vanderbilt Co. No equivalent.
5. PLASTHALL, monomeric plasticizer, C. P. Hall Co. No equivalent.
6. VULCUP 40KE from Hercules. No equivalent.

APPENDIX F

F.1 Silicone Rubber

F.1.1 Standard formulas for the following silicone rubber compounds are given in Table F2:

TABLE F1—

Number	Type
VMQ-1	peroxide-cured

F.1.2 Mixing procedure shall follow the guidelines in Section 5 of ASTM D 3187.

F.1.3 Recommended Standard Tensile Sheet Cures—Cure VMQ-1 specimen 10 minutes at 177 °C [350 °F]. Post cure: none.

F.1.4 Composition

TABLE F2—COMPOSITION VMQ SRE-1

Ingredient	Parts by mass
vinyl methyl silicone base polymer(1)	40.0
vinyl methyl silicone base polymer(2)	60.0
HT-1(3)	1.0
Magnesium oxide	3.0
2,5-dimethyl-2,5-di (tertbutylperoxy) hexane(4)	1.0
Total	105

1. Dow Corning Q4-4758. Base. No equivalent known.
2. Dow Corning Q4-4768. Base. No equivalent known.
3. Thermal stabilizing additive, Dow Corning. No equivalent.
4. Vulcanizing agent, VAROX DBPH-50, R. T. Vanderbilt Co., Inc. or equivalent.

APPENDIX G

G.1 Fluorosilicone Rubber

G.1.1 Standard formulas for the following fluorosilicone rubber compounds are given in Table G2:

TABLE G1—

Number	Type
FVMQ-1	peroxide-cured

G.1.2 Mixing procedure shall follow the guidelines in Section 5 of ASTM D 3187.

G.1.3 Recommended Standard Tensile Sheet Cures—Cure FVMQ-1 specimen 10 minutes at 177 °C [350 °F]. Post cure: 4h at 200 °C [392 °F].

G.1.4 Composition

TABLE G2—COMPOSITION FVMQ SRE-1

Ingredient	Parts by mass
Fluoro-vinyl methyl silicone base polymer(1)	100.0
HT-1(2)	1.0
2,5-dimethyl-2,5-di (tertbutylperoxy) hexane(3)	1.0
Total	102

1. Dow Corning Silicones LS-2860. No equivalent known.
2. Thermal stabilizing additive, Dow Corning. No equivalent.
3. Vulcanizing agent, VAROX DBPH-50, R. T. Vanderbilt Co., Inc. or equivalent.

APPENDIX H

H.1 Fluorocarbon Rubber

H.1.1 Standard formulas for the following fluorocarbon rubber compounds are given in Table H2:

TABLE H1—

Number	Type
FKM-1	bisphenol-cured

H.1.2 Mixing procedure shall follow the guidelines in Section 5 of ASTM D 3187.

H.1.3 Recommended Standard Tensile Sheet Cures—Cure FKM-1 specimen 10 minutes at 177 °C [350 °F]. Post cure: 24h at 200 °C [392 °F].

H.1.4 Composition

TABLE H2—COMPOSITION OF SRE FKM-1

Ingredient	Parts by mass
Fluorocarbon base polymer(1)	97.5
Calcium hydroxide	3.0
Magnesium Oxide(2)	6.0
N990 MT Carbon Black(3)	25
VC 20(4)	0.5
VC 50(5)	2
VPA-1 (6)	1
Total	135.0

1. Viton B600 from DuPont Dow Elastomers. No equivalent.
2. Elastomag 170 from Akrochem. No equivalent.
3. Thermax Flowform from Cancarb, Ltd. No equivalent.
4. Curative from DuPont Dow Elastomers. No equivalent
5. Curative from DuPont Dow Elastomers. No equivalent.
6. Processing aid from DuPont Dow Elastomers. No equivalent.

APPENDIX I

I.1 Copolymer of Tetrafluoroethylene and Propylene Rubber

I.1.1 Standard formulas for the following fluorocarbon rubber compounds are given in Table I2:

TABLE I1—

Number	Type
FEPM-1	peroxide-cured

I.1.2 Mixing procedure shall follow the guidelines in Section 5 of ASTM D 3187.

I.1.3 **Recommended Standard Tensile Sheet Cures—**Cure FEPM-1 specimen 10 minutes at 177 °C [350 °F]. Post cure: 24 hrs at 232 °C [450 °F].

I.1.4 **Composition**

TABLE I2—COMPOSITION SRE FEPM-1

Ingredient	Parts by mass
Tetrafluoroethylene-propylene copolymer(1)	100.0
N990 MT Carbon Black(2)	15.0
Calcium metasilicate(3)	10.0
Sodium Stearate	1.0
Triallyl isocyanurate (TAIC)(4)	4.0
40% a,a'-bis-(t-butyl peroxy) diisopropylbenzene on Burgess KE Clay(5)	4.0
Total	134.0

1. AFLAS 100S Polymer from Asahi Glass, Japan, distributed by 3M/dyneon. No equivalent.
2. Thermax Flowform from Cancarb, Ltd. No equivalent.
3. Nyad 400 from Nyco Inc. No equivalent.
4. DIAK #7 from DuPont Dow Elastomers. No equivalent.
5. VULCUP 40KE from Hercules. No equivalent.

APPENDIX J

J.1 Summary Table

Delta Elongation, % for Lab Averages ; Outliers Deleted

			ACM	AEM	FKM	FEPM	NBR-1	NBR-2	HNBR	VMQ	FVMQ	ECO
		Avg	-6.9	-18.8	-50.6	-9.8	-54.1	-35.3	-14.9	-23.1	-25.5	-76.9
Min =	Avg - t*SDev		-13.9	-22.8	-59.2	-19.0	-59.8	-48.8	-21.8	-35.7	-32.3	-81.3
Max =	Avg + t*SDev		-0.04	-14.73	-41.97	-0.54	-48.35	-21.77	-8.01	-10.46	-18.80	-72.57
		Range	13.8	8.1	17.3	18.5	11.4	27.0	13.7	25.3	13.5	8.7

Delta 50% Modulus, % , for Lab Averages ; Outliers deleted

			ACM	AEM	FKM	FEPM	NBR-1	NBR-2	HNBR	VMQ	FVMQ	ECO
		Avg	-8.8	-15.7	25.5	-19.6	7.5	6.4	-4.9	-25.3	32.5	
Min =	Avg - t*SDev		-39.7	-23.0	11.5	-37.7	-13.7	-7.1	-9.6	-40.1	18.7	
Max =	Avg + t*SDev		22.1	-8.4	39.4	-1.5	28.7	19.9	-0.2	-10.4	46.4	
		Range	61.8	14.6	28.0	36.1	42.4	27.0	9.3	29.7	27.7	

Delta Tensile Strength, % , for Lab Averages - Outliers Deleted

			ACM	AEM	FKM	FEPM	NBR-1	NBR-2	HNBR	VMQ	FVMQ	ECO
		Avg	-2.78	-19.9	-56.4	-11.7	-33.1	-33.2	-4.00	-25.3	-20.6	-44.1
Min =	Avg - t*SDev		-12.5	-24.7	-63.2	-25.1	-59.0	-46.5	-15.9	-41.3	-33.4	-53.4
Max =	Avg + t*SDev		6.9	-15.1	-49.6	1.8	-7.1	-19.9	7.9	-9.2	-7.7	-34.8
		Range	19.4	9.6	13.5	26.9	52.0	26.7	23.9	32.1	25.7	18.6
		Avg	-2.8	-19.9	-56.4	-11.7	-33.1	-33.2	-4.0	-25.3	-20.6	-44.1

Delta Hardness; % for Lab Averages - Outliers Deleted

			ACM	AEM	FKM	FEPM	NBR-1	NBR-2	HNBR	VMQ	FVMQ	ECO
		Avg	-7.8	-16.1	3.2	-7.0	-1.8	-1.8	-5.0	-31.6	11.0	10.3
Min =	Avg - t*SDev		-16	-21	1	-9	-12	-7	-7	-36	3	7
Max =	Avg + t*SDev		1	-11	6	-5	9	4	-3	-28	19	14
		Range	17	11	5	4	21	11	3	8	16	6

Percent Volume Swell ; Lab Averages - Outliers deleted

			ACM	AEM	FKM	FEPM	NBR-1	NBR-2	HNBR	VMQ	FVMQ	ECO
		Avg	2.00	24.5	0.60	8.73	4.42	5.51	4.07	30.7	-0.18	-0.0
Min =	Avg - t*SDev		0.4	22.0	-0.0	8.0	2.3	3.7	3.5	26.5	-1.3	-2.0
Max =	Avg + t*SDev		3.4	26.6	1.1	9.4	6.4	7.2	4.6	34.0	1.0	2.0
		Range	2.9	4.6	1.2	1.4	4.1	3.5	1.1	7.5	2.3	4.1

FIGURE J1a—Summary Table Part 1: Change (Delta) in Properties after Immersion

Original Ultimate Elongation, % ; Lab Averages - Outliers Deleted

			ACM	AEM	FKM	FEPM	NBR-1	NBR-2	HNBR	VMQ	FVMQ	ECO
		Avg	169	261	320	309	400	225	412	371	364	154
Min =	Avg - t*SDev		*150*	*224*	*292*	*292*	*380*	*204*	*372*	*287*	*341*	*105.3*
Max =	Avg + t*SDev		*188*	*297*	*348*	*326*	*421*	*247*	*451*	*455*	*387*	*202.5*
		Range	38	72	56	35	41	43	78	167	47	97
	Range as % of Avg		22	28	17	11	10	19	19	45	13	63

Original 50% Modulus, psi Lab Averages - Outliers Deleted

			ACM	AEM	FKM	FEPM	NBR-1	NBR-2	HNBR	VMQ	FVMQ	ECO
		Avg	339	442	295	308	283	299	289	180	171	524
Min =	Avg - t*SDev		*254*	*335*	*278*	*245*	*255*	*266*	*272*	*157*	*146*	*313.0*
Max =	Avg + t*SDev		*423*	*550*	*312*	*372*	*311*	*333*	*307*	*203*	*196*	*735.9*
		Range	169	214	34	127	56	67	35	46	49	423
	Range as % of Avg		50	48	12	41	20	22	12	25	29	81

Original Tensile Strength, psi , Lab Averages - Outliers Deleted

			ACM	AEM	FKM	FEPM	NBR-1	NBR-2	HNBR	VMQ	FVMQ	ECO
		Avg	1737	2731	1617	2498	2516	2384	3831	1186	1392	1491
Min =	Avg - t*SDev		*1616*	*2607*	*1488*	*2243*	*2338*	*1994*	*3573*	*935*	*1280*	*1256*
Max =	Avg + t*SDev		*1857*	*2856*	*1745*	*2753*	*2694*	*2774*	*4089*	*1436*	*1505*	*1726*
		Range	241	249	257	510	356	780	517	501	226	470
	Range as % of Avg		14	9	16	20	14	33	13	42	16	32

Original Shore A Durometer, pts , Lab Averages - Outliers Deleted

			ACM	AEM	FKM	FEPM	NBR-1	NBR-2	HNBR	VMQ	FVMQ	ECO
		Avg	67	75	73	70	69	68	71	62	60	78
Min =	Avg - t*SDev		*60*	*70*	*68*	*61*	*63*	*64*	*65*	*58*	*53*	*74*
Max =	Avg + t*SDev		*74*	*81*	*79*	*78*	*75*	*73*	*77*	*66*	*67*	*81*
		Range	14	11	11	17	12	9	12	7	14	7
	Range as % of Avg		21	15	15	24	18	13	17	12	24	9

FIGURE J1b—Summary Table Part 2: Original Properties

APPENDIX K

K.1 Protocol for Creation of Standard Reference Elastomer (SRE) Formula

K.1.1 Sensitivity—A SRE formulation must be reasonably sensitive to oil formulation changes as measured by elastomer property changes using approved test methods.

K.1.2 Support—There must exist a sufficient demand to justify creation of a new SRE formulation.

K.1.3 Representation—A SRE formulation must represent significant current usage volume in kilos and number of units in the ground vehicle transportation market.

K.1.3.1 This requirement is waived for new formulations developed to replace existing ones.

K.1.3.2 To capture all usage requirements, more than one SRE formulation within a ISO 1629 or ASTM D 1418 material classification is acceptable. For example, a family with different cure systems, polymer content or multiple base polymers.

K.1.4 Materials—SREs and their compounding materials must be stable, have a reasonable shelf-life, be safe for use, and readily available world wide.

K.1.4.1 Each SRE formulation must be processable to prepare the required test specimens.

K.1.4.2 Select generic SRE materials (from the commercial formulation) that aid in the assessment of compatibility with engine oil. Exclude non-value added materials (e.g. coagents) to minimize test variation.

K.1.5 Any new SRE formula is approved based on the performance of a material type (or all) immersion data in ASTM SF105.

K.2 Protocol for Maintenance of Standard Reference Elastomer (SRE) Formula

K.2.1 Substitutions—No SRE material or reference fluid (other than ASTM SF105) shall be substituted without the knowledge and written approval of the responsible organization or committee controlling the SRE formulation.

K.2.1.1 Materials are substituted for reasons of commercial obsolesce or banned by recognized health and safety organizations.

K.2.2 Permanent Changes—Approved changes shall be communicated by the responsible committee chairperson to the approved manufacturing and distribution facilities (see 4.3.2).

K.2.3 Uniformity—Revised SRE formulations shall be subjected to testing in accordance to the procedures outlined in Annex A4 of ASTM D 4678. Prior to Annex A4 testing it is necessary to establish uniformity in the lot as prepared for the candidate formula. Separate lots shall be tested days or months apart. A test result is the median of five individual values.

K.2.4 Review Frequency—The approved manufacturing facility shall review the demand (for continuation) and relevancy of SRE formulations every five years or less from the time of the latest revision.

K.2.5 Batch Life—The approved manufacturing and distribution facilities should maintain a six month supply on single batch quantities of SRE formulation materials and test slabs. See Section 6.

K.2.6 Batch Change Notification—The approved manufacturing facility shall notify customers of transitions between batches.

K.2.7 Retain Test Slabs—The approved manufacturing facility shall keep a sufficient supply of ASTM D 3182 test slabs from retained lot(s) for verification and qualification activity.

K.3 Control of Standard Reference Elastomer (SRE) Formula

K.3.1 Supply—More than one approved manufacturing facility is permitted, provided it supplies SRE formulas satisfying item K2.3 uniformity requirements. Distribution may be by separate sources to meet global demand. Approved facilities must satisfy all document requirements.

K.3.2 SRE formulations are produced and controlled (production and storage controls, statistical data, monitoring, etc.) using ISO 9000 and QS 9000 protocol.

K.3.2.1 Each SRE formulation has a written and audited process control plan.

K.3.3 Approved SRE formulations shall be published or distributed on request.

Rationale—Not applicable.

Relationship of SAE Standard to ISO Standard—Not applicable.

Application—This SAE Standard specifies requirements for vulcanized rubbers in sheet form for use as standards in characterizing the effect of test liquids and service fluids. The annexes contain the standard reference elastomer formulas.

The property changes of the SRE in contact with the indicated fluid under specified test conditions are the responsibility of the user. See 7.3 and Table 1.

This standard is not designed to provide formulations of elastomeric product compositions for actual service.

Reference Section

ASTM D 412—ASTM for Vulcanized Rubber and Thermoplastic Elastomers-Tension

ASTM D 471—STM for Rubber Property - Effect of Fluids

ASTM D 1349—Standard Practice for Rubber – Standard Temperatures for Testing

ASTM D 1418—Standard Practice for Rubber and Rubber Latices – Nomenclature

ASTM D 3182—Standard Practice for Rubber Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

ASTM D 3187—Standard Test Methods for Rubber – Evaluation of NBR (Acrylonitrile-Butadiene Rubber)

ASTM D 3767—Rubber – Measurement of Dimensions

ASTM D 4678—Standard Practice for Rubber – Preparation, Testing Acceptance, Documentation, and Use of Reference Materials

1629—Rubber & latices – Nomenclature

2230—Vulcanized rubber property – Guide to storage

Developed by the SAE Engine Oil Comp. with Rubber Gaskets and Seals Subcommittee

Sponsored by the SAE Committee on Automotive Rubber Standards