



State of West Virginia
 Department of Administration
 Purchasing Division
 2019 Washington Street East
 Post Office Box 50130
 Charleston, WV 25305-0130

Request for Quotation

RFQ NUMBER
0311C345

PAGE
1

ADDRESS CORRESPONDENCE TO ATTENTION OF
BUYER 33
304-558-2402

VENDOR

RFQ COPY
 TYPE NAME/ADDRESS HERE

SHIP TO

DIVISION OF HIGHWAYS
 I-77/WV 47
 MILL RUN ROAD
 PARKERSBURG, WV
 26101

DATE PRINTED	TERMS OF SALE	SHIP VIA	F.O.B.	FREIGHT TERMS
03/30/2011				

BID OPENING DATE: **05/04/2011** BID OPENING TIME **01:30PM**

LINE	QUANTITY	UOP	CAT NO	ITEM NUMBER	UNIT PRICE	AMOUNT
0001	1	GL		745-67		
<p>QPR PERMANENT PAVEMENT REPAIR MATERIAL</p> <p>REQUEST FOR QUOTATION (RFQ) OPEN END CONTRACT</p> <p>THE WEST VIRGINIA STATE PURCHASING DIVISION FOR THE AGENCY, THE WEST VIRGINIA DIVISION OF HIGHWAYS, IS SOLICITING BIDS FOR AN OPEN END CONTRACT TO PROVIDE QPR PERMANENT PAVEMENT REPAIR MATERIAL OR EQUAL PER THE ATTACHED SPECIFICATIONS.</p> <p>TECHNICAL QUESTIONS CONCERNING THIS SOLICITATION MUST BE SUBMITTED IN WRITING TO SHERI SLONE IN THE WEST VIRGINIA STATE PURCHASING DIVISION VIA MAIL AT THE ADDRESS SHOWN IN THE BODY OF THIS RFQ, VIA FAX AT 304-558-4115, OR VIA EMAIL AT SHERI.D.SLONE@WV.GOV. A WORD DOCUMENT HAS BEEN ATTACHED FOR YOUR CONVENIENCE TO SUBMIT TECHNICAL QUESTIONS. DEADLINE FOR ALL TECHNICAL QUESTIONS IS 04/08/2011 AT THE CLOSE OF BUSINESS. ANY TECHNICAL QUESTIONS RECEIVED WILL BE ANSWERED BY FORMAL ADDENDUM TO BE ISSUED BY THE PURCHASING DIVISION AFTER THE DEADLINE HAS LAPSED.</p> <p>EXHIBIT 10</p> <p>REQUISITION NO.:</p>						

SEE REVERSE SIDE FOR TERMS AND CONDITIONS

SIGNATURE	TELEPHONE	DATE
TITLE	FEIN	ADDRESS CHANGES TO BE NOTED ABOVE

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GENERAL TERMS & CONDITIONS REQUEST FOR QUOTATION (RFQ) AND REQUEST FOR PROPOSAL (RFP)

1. Awards will be made in the best interest of the State of West Virginia.
 2. The State may accept or reject in part, or in whole, any bid.
 3. Prior to any award, the apparent successful vendor must be properly registered with the Purchasing Division and have paid the required \$125 fee.
 4. All services performed or goods delivered under State Purchase Order/Contracts are to be continued for the term of the Purchase Order/Contracts, contingent upon funds being appropriated by the Legislature or otherwise being made available. In the event funds are not appropriated or otherwise available for these services or goods this Purchase Order/Contract becomes void and of no effect after June 30.
 5. Payment may only be made after the delivery and acceptance of goods or services.
 6. Interest may be paid for late payment in accordance with the *West Virginia Code*.
 7. Vendor preference will be granted upon written request in accordance with the *West Virginia Code*.
 8. The State of West Virginia is exempt from federal and state taxes and will not pay or reimburse such taxes.
 9. The Director of Purchasing may cancel any Purchase Order/Contract upon 30 days written notice to the seller.
 10. The laws of the State of West Virginia and the *Legislative Rules* of the Purchasing Division shall govern the purchasing process.
 11. Any reference to automatic renewal is hereby deleted. The Contract may be renewed only upon mutual written agreement of the parties.
 12. **BANKRUPTCY:** In the event the vendor/contractor files for bankruptcy protection, the State may deem this contract null and void, and terminate such contract without further order.
 13. **HIPAA BUSINESS ASSOCIATE ADDENDUM:** The West Virginia State Government HIPAA Business Associate Addendum (BAA), approved by the Attorney General, is available online at www.state.wv.us/admin/purchase/vrc/hipaa.htm and is hereby made part of the agreement. Provided that the Agency meets the definition of a Cover Entity (45 CFR §160.103) and will be disclosing Protected Health Information (45 CFR §160.103) to the vendor.
 14. **CONFIDENTIALITY:** The vendor agrees that he or she will not disclose to anyone, directly or indirectly, any such personally identifiable information or other confidential information gained from the agency, unless the individual who is the subject of the information consents to the disclosure in writing or the disclosure is made pursuant to the agency's policies, procedures, and rules. Vendor further agrees to comply with the Confidentiality Policies and Information Security Accountability Requirements, set forth in <http://www.state.wv.us/admin/purchase/privacy/noticeConfidentiality.pdf>.
 15. **LICENSING:** Vendors must be licensed and in good standing in accordance with any and all state and local laws and requirements by any state or local agency of West Virginia, including, but not limited to, the West Virginia Secretary of State's Office, the West Virginia Tax Department, and the West Virginia Insurance Commission. The vendor must provide all necessary releases to obtain information to enable the director or spending unit to verify that the vendor is licensed and in good standing with the above entities.
 16. **ANTITRUST:** In submitting a bid to any agency for the State of West Virginia, the bidder offers and agrees that if the bid is accepted the bidder will convey, sell, assign or transfer to the State of West Virginia all rights, title and interest in and to all causes of action it may now or hereafter acquire under the antitrust laws of the United States and the State of West Virginia for price fixing and/or unreasonable restraints of trade relating to the particular commodities or services purchased or acquired by the State of West Virginia. Such assignment shall be made and become effective at the time the purchasing agency tenders the initial payment to the bidder.
- I certify that this bid is made without prior understanding, agreement, or connection with any corporation, firm, limited liability company, partnership, or person or entity submitting a bid for the same material, supplies, equipment or services and is in all respects fair and without collusion or Fraud. I further certify that I am authorized to sign the certification on behalf of the bidder or this bid.

INSTRUCTIONS TO BIDDERS

1. Use the quotation forms provided by the Purchasing Division. Complete all sections of the quotation form.
2. Items offered must be in compliance with the specifications. Any deviation from the specifications must be clearly indicated by the bidder. Alternates offered by the bidder as **EQUAL** to the specifications must be clearly defined. A bidder offering an alternate should attach complete specifications and literature to the bid. The Purchasing Division may waive minor deviations to specifications.
3. Unit prices shall prevail in case of discrepancy. All quotations are considered F.O.B. destination unless alternate shipping terms are clearly identified in the quotation.
4. All quotations must be delivered by the bidder to the office listed below prior to the date and time of the bid opening. Failure of the bidder to deliver the quotations on time will result in bid disqualifications: Department of Administration, Purchasing Division, 2019 Washington Street East, P.O. Box 50130, Charleston, WV 25305-0130
5. Communication during the solicitation, bid, evaluation or award periods, except through the Purchasing Division, is strictly prohibited (W.Va. C.S.R. §148-1-6.6).



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ADDENDUM ACKNOWLEDGEMENT						
I HEREBY ACKNOWLEDGE RECEIPT OF THE FOLLOWING CHECKED ADDENDUM(S) AND HAVE MADE THE NECESSARY REVISIONS TO MY PROPOSAL, PLANS AND/OR SPECIFICATION, ETC.						
ADDENDUM NO.'S:						
NO. 1						
NO. 2						
NO. 3						
NO. 4						
NO. 5						
I UNDERSTAND THAT FAILURE TO CONFIRM THE RECEIPT OF THE ADDENDUM(S) MAY BE CAUSE FOR REJECTION OF BIDS.						
VENDOR MUST CLEARLY UNDERSTAND THAT ANY VERBAL REPRESENTATION MADE OR ASSUMED TO BE MADE DURING ANY ORAL DISCUSSION HELD BETWEEN VENDOR'S REPRESENTATIVES AND ANY STATE PERSONNEL IS NOT BINDING. ONLY THE INFORMATION ISSUED IN WRITING AND ADDED TO THE SPECIFICATIONS BY AN OFFICIAL ADDENDUM IS BINDING.						
				 SIGNATURE	
				 COMPANY	
					

SEE REVERSE SIDE FOR TERMS AND CONDITIONS

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DATE						
NOTE: THIS ADDENDUM ACKNOWLEDGEMENT SHOULD BE SUBMITTED WITH THE BID.						
REV. 09/21/2009						
EXHIBIT 3						
LIFE OF CONTRACT: THIS CONTRACT BECOMES EFFECTIVE UPON AWARD AND EXTENDS FOR A PERIOD OF ONE (1) YEAR OR UNTIL SUCH "REASONABLE TIME" THEREAFTER AS IS NECESSARY TO OBTAIN A NEW CONTRACT OR RENEW THE ORIGINAL CONTRACT. THE "REASONABLE TIME" PERIOD SHALL NOT EXCEED TWELVE (12) MONTHS. DURING THIS "REASONABLE TIME" THE VENDOR MAY TERMINATE THIS CONTRACT FOR ANY REASON UPON GIVING THE DIRECTOR OF PURCHASING 30 DAYS WRITTEN NOTICE.						
UNLESS SPECIFIC PROVISIONS ARE STIPULATED ELSEWHERE IN THIS CONTRACT DOCUMENT, THE TERMS, CONDITIONS AND PRICING SET HEREIN ARE FIRM FOR THE LIFE OF THE CONTRACT.						
RENEWAL: THIS CONTRACT MAY BE RENEWED UPON THE MUTUAL WRITTEN CONSENT OF THE SPENDING UNIT AND VENDOR, SUBMITTED TO THE DIRECTOR OF PURCHASING THIRTY (30) DAYS PRIOR TO THE EXPIRATION DATE. SUCH RENEWAL SHALL BE IN ACCORDANCE WITH THE TERMS AND CONDITIONS OF THE ORIGINAL CONTRACT AND SHALL BE LIMITED TO TWO (2) ONE (1) YEAR PERIODS.						
CANCELLATION: THE DIRECTOR OF PURCHASING RESERVES THE RIGHT TO CANCEL THIS CONTRACT IMMEDIATELY UPON WRITTEN						

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<p>NOTICE TO THE VENDOR IF THE COMMODITIES AND/OR SERVICES SUPPLIED ARE OF AN INFERIOR QUALITY OR DO NOT CONFORM TO THE SPECIFICATIONS OF THE BID AND CONTRACT HEREIN.</p> <p>OPEN MARKET CLAUSE: THE DIRECTOR OF PURCHASING MAY AUTHORIZE A SPENDING UNIT TO PURCHASE ON THE OPEN MARKET, WITHOUT THE FILING OF A REQUISITION OR COST ESTIMATE, ITEMS SPECIFIED ON THIS CONTRACT FOR IMMEDIATE DELIVERY IN EMERGENCIES DUE TO UNFORESEEN CAUSES (INCLUDING BUT NOT LIMITED TO DELAYS IN TRANSPORTATION OR AN UNANTICIPATED INCREASE IN THE VOLUME OF WORK.)</p> <p>QUANTITIES: QUANTITIES LISTED IN THE REQUISITION ARE APPROXIMATIONS ONLY, BASED ON ESTIMATES SUPPLIED BY THE STATE SPENDING UNIT. IT IS UNDERSTOOD AND AGREED THAT THE CONTRACT SHALL COVER THE QUANTITIES ACTUALLY ORDERED FOR DELIVERY DURING THE TERM OF THE CONTRACT, WHETHER MORE OR LESS THAN THE QUANTITIES SHOWN.</p> <p>ORDERING PROCEDURE: SPENDING UNIT(S) SHALL ISSUE A WRITTEN STATE CONTRACT ORDER (FORM NUMBER WV-39) TO THE VENDOR FOR COMMODITIES COVERED BY THIS CONTRACT. THE ORIGINAL COPY OF THE WV-39 SHALL BE MAILED TO THE VENDOR AS AUTHORIZATION FOR SHIPMENT, A SECOND COPY MAILED TO THE PURCHASING DIVISION, AND A THIRD COPY RETAINED BY THE SPENDING UNIT.</p> <p>BANKRUPTCY: IN THE EVENT THE VENDOR/CONTRACTOR FILES FOR BANKRUPTCY PROTECTION, THE STATE MAY DEEM THE CONTRACT NULL AND VOID, AND TERMINATE SUCH CONTRACT WITHOUT FURTHER ORDER.</p> <p>THE TERMS AND CONDITIONS CONTAINED IN THIS CONTRACT SHALL SUPERSEDE ANY AND ALL SUBSEQUENT TERMS AND CONDITIONS WHICH MAY APPEAR ON ANY ATTACHED PRINTED</p>						

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				DOCUMENTS SUCH AS PRICE LISTS, ORDER FORMS, SALES AGREEMENTS OR MAINTENANCE AGREEMENTS, INCLUDING ANY ELECTRONIC MEDIUM SUCH AS CD-ROM.		
				REV. 05/26/2009		
				PURCHASING CARD ACCEPTANCE: THE STATE OF WEST VIRGINIA CURRENTLY UTILIZES A VISA PURCHASING CARD PROGRAM WHICH IS ISSUED THROUGH A BANK. THE SUCCESSFUL VENDOR MUST ACCEPT THE STATE OF WEST VIRGINIA VISA PURCHASING CARD FOR PAYMENT OF ALL ORDERS PLACED BY ANY STATE AGENCY AS A CONDITION OF AWARD.		
				EXHIBIT 4		
				LOCAL GOVERNMENT BODIES: UNLESS THE VENDOR INDICATES IN THE BID HIS REFUSAL TO EXTEND THE PRICES, TERMS, AND CONDITIONS OF THE BID TO COUNTY, SCHOOL, MUNICIPAL AND OTHER LOCAL GOVERNMENT BODIES, THE BID SHALL EXTEND TO POLITICAL SUBDIVISIONS OF THE STATE OF WEST VIRGINIA. IF THE VENDOR DOES NOT WISH TO EXTEND THE PRICES, TERMS, AND CONDITIONS OF THE BID TO ALL POLITICAL SUBDIVISIONS OF THE STATE, THE VENDOR MUST CLEARLY INDICATE SUCH REFUSAL IN HIS BID. SUCH REFUSAL SHALL NOT PREJUDICE THE AWARD OF THIS CONTRACT IN ANY MANNER.		
				REV. 3/88		
				NOTICE		
				A SIGNED BID MUST BE SUBMITTED TO:		

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DEPARTMENT OF ADMINISTRATION PURCHASING DIVISION BUILDING 15 2019 WASHINGTON STREET, EAST CHARLESTON, WV 25305-0130						
THE BID SHOULD CONTAIN THIS INFORMATION ON THE FACE OF THE ENVELOPE OR THE BID MAY NOT BE CONSIDERED:						
SEALED BID						
BUYER: SHERI SLONE - FILE 33						
RFQ. NO.: 0311C345						
BID OPENING DATE: 05/04/2011						
BID OPENING TIME: 1:30 PM						
PLEASE PROVIDE A FAX NUMBER IN CASE IT IS NECESSARY TO CONTACT YOU REGARDING YOUR BID:						

CONTACT PERSON (PLEASE PRINT CLEARLY):						

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***** THIS IS THE END OF RFQ 0311C345 ***** TOTAL:						

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Specifications

Purpose: To establish a contract to provide the District Three West Virginia Division of Highways with QPR or equal.

1.0 DEFINITIONS

- A. The "Agency" shall be defined as the West Virginia Division of Highways
- B. The "Vendor" shall be defined as the successful bidder.
- C. The "Purchase Order" shall be defined as the binding agreement that is entered into between the State of West Virginia and the Vendor to provide the services as herein specified.
- D. "Mandatory Requirements: The terms "must", "will", "shall", "minimum", "maximum", or "is/are required" identify a mandatory item or factor. Decisions regarding compliance with any mandatory requirements shall be at the sole discretion of the State.

2.0 SCOPE OF WORK

The successful Vendor shall provide QPR, or equal, permanent pavement repair material for District Three.

2.1 Technical Requirements:

Material supplied under this contract shall be a plant or pug mill mixed high performance pavement patching material capable of storage in an uncovered outdoor stockpile for a minimum of 12 months. It shall be composed of laboratory approved mineral aggregates and modified bituminous liquid oil blend capable of coating wet aggregates without stripping and have stripping resistance of retained coating of not less than 95%. The permanent asphalt repair shall be uniform, remain flexible and cohesive to -15°F and be capable of retaining adhesive qualities in wet applications. The patching material shall be able to repair asphalt, concrete, surface treated roads and shall not require removal and replacement if ever the pavement repair area is overlaid.

2.2 MATERIALS

The aggregate shall consist of 100% crushed stone or a laboratory approved equivalent under ASTM C-136. All aggregate is to be from approved sources, and representative samples of both fine and coarse aggregate shall be from the plant site and laboratory tested.

Recommended gradation analyses are as follows:

<u>SCREEN SIZES</u>	<u>PERCENTAGE PASSING</u>
1/4"	100
#4	40-90
#8	5-40
#16	0-10
#50	0-6
#200	0-2

2.2.1 Bituminous Material

The modified bituminous liquid oil blend must meet the following requirements:

ASTM D-1310 Flashpoint (TOC):	200°F (94° C) minimum
ASTM D-2170 Kinematic Viscosity at 60° C (140° F):	300-400
ASTM D-95 Water:	0.2% maximum
ASTM D-402 Distillate Test (Volume of original sample):	
To 437° F (225° C)	None
To 500° F (260° C)	0-5%
To 600° F (315° C)	0-25%
Residue from distillate at 680 ° F (360° C)	72-95%

2.3 PLANT MIX

The preferred mixing ratio shall be 4.5% to 6% liquid oil blend per finished ton (2000 lbs) of mixed material.

2.4 Freight

2.4.1 All shipments are to be FOB Destination.

2.4.2 The vendor shall be responsible for all costs associated with delivery to the job site.

2.5 Delivery

2.5.1 The above material is to be delivered to the West Virginia Division of Highways, District Three lot, located at 1423 Mill Run Road, I-77 and WV 47 interchange, Parkersburg, WV (Quonset hut) 26104. Requested deliveries will be by complete pallets.

2.5.2 Vendor to notify District Three Storeroom 1 day prior to delivery at (304-420-4669)

3.0 INVOICING

3.1 Payment

The Vendor must send an invoice to:

West Virginia Division of Highways
624 Depot Street
Parkersburg, WV 26101
Attn: Beverly Myers

4.0 AWARD

Purchase order will be awarded complete and not split.

COST SHEET

Item #	Est Quantity	Description*	Unit Cost	Amount
1	2646	QPR Permanent Pavement Repair Material - or equal High Performance pavement Patching Material for Repairs - Bags * "Or Equal" merchandise-vendor must provide complete manufacturer's literature demonstrating adherence to the mandatory requirements contained herein.		
		Total Amount		



Designation: D2170/D2170M – 10



Designation: 319/84 (89)

Standard Test Method for Kinematic Viscosity of Asphalts (Bitumens)¹

This standard is issued under the fixed designation D2170/D2170M; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

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

1. Scope

1.1 This test method covers procedures for the determination of kinematic viscosity of liquid asphalts (bitumens), road oils and distillation residues of liquid asphalts (bitumens) all at 60°C [140°F] and of asphalt cements at 135°C [275°F] (Note 1) in the range from 6 to 100 000 mm²/s [cSt].

1.2 Results of this test method can be used to calculate viscosity when the density of the test material at the test temperature is known or can be determined. See Annex A1 for the method of calculation.

NOTE 1—This test method is suitable for use at other temperatures and at lower kinematic viscosities, but the precision is based on determinations on liquid asphalts and road oils at 60°C [140°F] and on asphalt cements at 135°C [275°F] only in the viscosity range from 30 to 6000 mm²/s [cSt].

1.3 **Warning**—Mercury has been designated by United States Environmental Protection Agency (EPA) and many state agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—<http://www.epa.gov/mercury/faq.htm>—for additional information. Users should be aware that selling mercury, mercury containing products, or both, in your state may be prohibited by state law.

1.4 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

¹ This test method is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.44 on Rheological Tests. In the IP this test method is under the jurisdiction of the Standardization Committee.

Current edition approved June 1, 2010. Published August 2010. Originally approved in 1963. Last previous edition approved in 2007 as D2170 – 07. DOI: 10.1520/D2170-10.

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

- C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials
- D92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester
- D341 Practice for Viscosity-Temperature Charts for Liquid Petroleum Products
- D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
- D446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers
- D2162 Practice for Basic Calibration of Master Viscometers and Viscosity Oil Standards
- D2493 Standard Viscosity-Temperature Chart for Asphalts
- E1 Specification for ASTM Liquid-in-Glass Thermometers
- E77 Test Method for Inspection and Verification of Thermometers

3. Terminology

3.1 Definitions:

3.1.1 *density*—the mass per unit volume of liquid. The SI unit of density is 1 kg/m³. The cgs unit of density is 1 g/cm³.

3.1.2 *kinematic viscosity*—the ratio of the viscosity to the density of a liquid. It is a measure of the resistance to flow of a liquid under gravity. The SI unit of kinematic viscosity is m²/s; for practical use, a submultiple (mm²/s) is more convenient. The cgs unit of kinematic viscosity is 1 cm²/s and is

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.

called a stoke (symbol St). The centistoke (1 cSt = 10^{-2} St) is $1 \text{ mm}^2/\text{s}$ and is often used.

3.1.3 *Newtonian liquid*—a liquid in which the rate of shear is proportional to the shearing stress. The constant ratio of the shearing stress to the rate of shear is the viscosity of the liquid. If the ratio is not constant, the liquid is non-Newtonian.

3.1.4 *viscosity*—the ratio between the applied shear stress and rate of shear is called the coefficient of viscosity. This coefficient is a measure of the resistance to flow of a liquid. It is commonly called the viscosity of the liquid. The cgs unit of viscosity is $1 \text{ g/cm}\cdot\text{s}$ ($1 \text{ dyne}\cdot\text{s/cm}^2$) and is called a poise (P). The SI unit of viscosity is $1 \text{ Pa}\cdot\text{s}$ ($1 \text{ N}\cdot\text{s/m}^2$) and is equivalent to 10 P.

4. Summary of Test Method

4.1 The time is measured for a fixed volume of the liquid to flow through the capillary of a calibrated glass capillary viscometer under an accurately reproducible head and at a closely controlled temperature. The kinematic viscosity is then calculated by multiplying the efflux time in seconds by the viscometer calibration factor.

5. Significance and Use

5.1 The kinematic viscosity characterizes flow behavior. The method is used to determine the consistency of bitumen as one element in establishing the uniformity of shipments or sources of supply. The specifications are usually at temperatures of 60 and 135°C .

6. Apparatus

6.1 *Viscometers*, capillary-type, made of borosilicate glass, annealed, suitable for this test are described in Annex A2 and include the following:

- 6.1.1 *Cannon-Fenske viscometer for opaque liquids*,
- 6.1.2 *Zeitfuchs cross-arm viscometer*,
- 6.1.3 *Lantz-Zeitfuchs viscometer*,
- 6.1.4 *BS U-Tube modified reverse flow viscometer*.

6.1.5 Calibrated viscometers are available from commercial suppliers. Details regarding calibration of viscometers are given in Annex A3.

6.2 *Thermometers*—Calibrated liquid-in-glass thermometers of an accuracy after correction of 0.02°C [0.04°F] can be used or any other thermometric device of equal accuracy. ASTM Kinematic Viscosity Thermometers 47C and 47F conforming to Specification E1, and IP 35C and 35F are suitable for use at 60°C [140°F] and ASTM Kinematic Viscosity Thermometers 110C and 110F are suitable for use at 135°C [275°F].

6.2.1 The specified thermometers are standardized at “total immersion,” which means immersion to the top of the liquid column with the remainder of the stem and the expansion chamber at the top of the thermometer exposed to room temperature. The practice of completely submerging the thermometer is not recommended. When thermometers are completely submerged, corrections for each individual thermometer based on calibration under conditions of complete submergence must be determined and applied. If the thermometer is completely submerged in the bath during use, the

TABLE 1 Acceptability of Test Results for Kinematic Viscosity

NOTE 1—The values given in column 2 are the coefficients of variation that have been found to be appropriate for the materials and conditions of test described in column 1. The values given in column 3 are the limits that should not be exceeded by the difference between the results of two properly conducted tests.

Material and Type Index	Coefficient of Variation (% of mean) ^A	Acceptable Range of Two Results (% of mean) ^A
Single-operator precision:		
Asphalt cements at 135°C [275°F]	0.64	1.8
Liquid asphalts at 60°C [140°F]:		
below $3000 \text{ mm}^2/\text{s}$ [cSt]	0.53	1.5
3000 to $6000 \text{ mm}^2/\text{s}$ [cSt]	0.71	2.0
$6000 \text{ mm}^2/\text{s}$ [cSt] and above	3.2	8.9
Multilaboratory precision:		
Asphalt cements at 135°C [275°F]	3.1	8.8
Liquid asphalts at 60°C [140°F]:		
below $3000 \text{ mm}^2/\text{s}$ [cSt]	1.06	3.0
3000 to $6000 \text{ mm}^2/\text{s}$ [cSt] ^B	3.11	9.0
above $6000 \text{ mm}^2/\text{s}$ [cSt] ^B	3.6	10.0

^A These numbers represent, respectively, the (1s %) and (d2s %) limits as described in Practice C670.

^B Based on less than 30 degrees of freedom.

pressure of the gas in the expansion chamber will be higher or lower than during standardization, and may cause high or low readings on the thermometer. Thermometric devices for this test method must be standardized at least every 6 months.

6.2.2 It is essential that liquid-in-glass thermometers be calibrated periodically using the technique given in Test Method E77 (see Appendix X1).

6.3 *Bath*—A bath suitable for immersion of the viscometer so that the liquid reservoir or the top of the capillary, whichever is uppermost, is at least 20 mm below the upper bath level, and with provisions for visibility of the viscometer and the thermometer. Firm supports for the viscometer shall be provided, or the viscometer may be an integral part of the bath. The efficiency of the stirring and the balance between heat losses and heat input must be such that the temperature of the bath medium does not vary by more than $\pm 0.03^\circ\text{C}$ [$\pm 0.05^\circ\text{F}$] over the length of the viscometer, or from viscometer to viscometer in the various bath positions at 60°C [140°F].

NOTE 2—Distilled water is a suitable bath liquid for determinations at 60°C [140°F]. USP White Oil with a flash point above 215°C [420°F] has been found suitable for determinations at 135°C [275°F]. The flash point is determined in accordance with Test Method D92.

6.4 *Timer*—A stop watch or other timing device graduated in divisions of 0.1 s or less and accurate to within 0.05 % when tested over intervals of not less than 15 min. Timing devices for this test method must be calibrated at least every 6 months.

6.4.1 *Electrical Timing Devices* may be used only on electrical circuits the frequencies of which are controlled to an accuracy of 0.05 % or better.

6.4.1.1 Alternating currents, the frequencies of which are intermittently and not continuously controlled, as provided by some public power systems, can cause large errors, particularly over short timing intervals, when used to actuate electrical timing devices.

7. Preparation of Sample

7.1 To minimize loss of volatile constituents and to obtain reproducible results, proceed as follows:

7.1.1 Procedure for Liquid Asphalt (Bitumen) and Road Oil:

7.1.1.1 Allow sealed samples, as received, to reach room temperature.

7.1.1.2 Open the sample container and mix the sample thoroughly by stirring for 30 s taking care to avoid the entrapment of air. If the sample is too viscous for such stirring, place the sample in the tightly sealed container in a bath or oven maintained at $63 \pm 3^\circ\text{C}$ [$145 \pm 5^\circ\text{F}$] until it becomes sufficiently liquid for stirring.

7.1.1.3 Immediately charge the viscometer, or, if the test is to be made at a later time, pour approximately 20 mL into one or more clean, dry containers having a capacity of approximately 30 mL and immediately seal with an airtight closure.

7.1.1.4 For materials with kinematic viscosities at 60°C [140°F] above $800 \text{ mm}^2/\text{s}$ [cSt], heat the 20-mL sample in the sealed container in an oven or bath maintained at $63 \pm 3^\circ\text{C}$ [$145 \pm 5^\circ\text{F}$] until it is sufficiently liquid for a convenient transfer into the viscometer. Such heating should not exceed 30 min.

7.1.2 Procedure for Asphalt Cement:

7.1.2.1 Heat the sample with care to prevent local overheating until it has become sufficiently fluid to pour, occasionally stirring the sample to aid heat transfer and to assure uniformity.

7.1.2.2 Transfer a minimum of 20 mL into a suitable container and heat to $135 \pm 5.5^\circ\text{C}$ [$275 \pm 10^\circ\text{F}$], stirring occasionally to prevent local overheating and taking care to avoid the entrapment of air.

8. Procedure

8.1 The specific details of operation vary somewhat for the various types of viscometers. See the detailed descriptions of viscometers in Annex A2 for instructions for using the type of viscometer selected. In all cases, however, follow the general procedure described in 8.2 to 8.8.

8.2 Maintain the bath at the test temperature within $\pm 0.01^\circ\text{C}$ [$\pm 0.02^\circ\text{F}$]. Apply the necessary corrections, if any, to all thermometer readings.

8.3 Select a clean, dry viscometer which will give an efflux time greater than 60 s and preheat to test temperature.

8.4 Charge the viscometer in the manner dictated by the design of the instrument, as prescribed in Annex A2.

8.5 Allow the charged viscometer to remain in the bath long enough to reach the test temperature, as prescribed in Annex A2.

8.6 Start the flow of asphalt in the viscometer as prescribed in Annex A2.

8.7 Measure to within 0.1 s, the time required for the leading edge of the meniscus to pass from the first timing mark to the second. If this efflux time is less than 60 s, select a viscometer of smaller capillary diameter and repeat the operation.

8.8 Upon completion of the test, clean the viscometer thoroughly by several rinsings with an appropriate solvent completely miscible with the sample, followed by a completely volatile solvent. Dry the tube by passing a slow stream of filtered dried air through the capillary for 2 min, or until the last trace of solvent is removed. Alternatively, the viscometer may be cleaned in a glass cleaning oven, at a temperature not to exceed 500°C [932°F], followed by rinses with distilled or deionized water, residue-free acetone, and filtered dry air. Periodically, if deposits are observed, clean the instrument with a strong acid cleaning solution to remove organic deposits, rinse thoroughly with distilled water and residue-free acetone, and dry with filtered dry air.

NOTE 3—The cleaning oven may burn off the glue that connects the viscometer tube to the holder.

8.8.1 Chromic acid cleaning solution may be prepared by adding, with the usual precautions, 800 mL of concentrated sulphuric acid to a solution of 92 g of sodium dichromate in 458 mL of water. The use of similar commercially available sulphuric acid cleaning solutions is acceptable. Nonchromium-containing, strongly oxidizing acid cleaning solutions may be substituted so as to avoid the disposal problems of chromium-containing solutions.

8.8.2 Use of alkaline glass cleaning solutions may result in a change of viscometer calibration, and is not recommended.

9. Calculation

9.1 Calculate the kinematic viscosity to three significant figures using the following equation:

$$\text{Kinematic viscosity, mm}^2/\text{s [cSt]} = Ct \quad (1)$$

where:

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

t = efflux time, s.

NOTE 4—Other ASTM publications on viscosity that may be used in conjunction with results determined in accordance with this test method are Test Methods D445 and D341, Standard D2493, and Specifications D446.

10. Report

10.1 Always report the test temperature with the result, for example:

$$\text{Kinematic viscosity at } 60^\circ\text{C} = 75.6 \text{ mm}^2/\text{s [cSt]} \quad (2)$$

11. Precision and Bias

11.1 Criteria for judging the acceptability of viscosity test results obtained by this method are given in Table 1.

11.2 The bias of the procedure in this test method cannot be determined because no material having an accepted reference value is available.

12. Keywords

12.1 asphalt; capillary; kinematic; viscometer; viscosity

ANNEXES

(Mandatory Information)

A1. CALCULATION OF VISCOSITY OF A NEWTONIAN LIQUID

A1.1 The viscosity of a Newtonian liquid is calculated from its kinematic viscosity by multiplying the kinematic viscosity by the density of the liquid at the test temperature.

A1.2 For paving grade asphalts, density at 135°C [275°F] can be conveniently determined from specific gravity at 25/25°C [77/77°F] by multiplying by a factor of 0.934 g/cm³; or from the specific gravity at 15.5/15.5°C [60/60°F] by multiplying by a factor of 0.931 g/cm³. These factors are based on an average coefficient of expansion for asphalt cement of

0.00061/°C [0.00034/°F].

A1.3 When the specific gravity of the asphalt is not known, a satisfactory determination of the viscosity at 135°C [275°F] can be obtained by multiplying the kinematic viscosity at 135°C [275°F] by an assumed density of 0.948 g/cm³. This density value is equivalent to a specific gravity of 1.015 at 25/25°C [77/77°F]. Tests on a large number of asphalts indicate that the error introduced by this assumed density will not exceed ±3 %.

A2. REVERSE-FLOW VISCOMETERS**A2.1 Scope**

A2.1.1 The reverse-flow viscometers for transparent and opaque liquids include the Cannon-Fenske Opaque,³ Zeitfuchs Cross-Arm,⁴ BS/IP/RF, and Lantz-Zeitfuchs viscometers. Unlike the modified Ostwald and suspended-level viscometers, the sample of liquid flows into a timing bulb not previously wet by sample, thus allowing the timing of liquids whose thin films are opaque. Reverse-flow viscometers are used for the measurement of kinematic viscosities of opaque and transparent liquids up to 100 000 mm²/s [cSt].

A2.2 Apparatus

A2.2.1 For the reverse-flow viscometers, detailed drawings of the size designations, approximate constants, kinematic viscosity range, capillary diameter, and bulb volumes for each viscometer are shown in Figs. A2.1-A2.4. The kinematic viscosity range is based on a 60-s minimum flow-time in this method; the same viscometers are also described in Specifications D446 with a different viscosity range based on a 200-s minimum flow-time as prescribed in Test Method D445. The lengths of the upper tubes K, L, M and N in Figs. A2.1-A2.4 may be increased (or decreased) to conform to the immersion requirements of 6.3.

A2.3 Operating Instructions

A2.3.1 A standard operating procedure is contained in Section 7. Additional operating instructions for the reverse-flow viscometers are outlined in A2.3.2-A2.3.8 with emphasis on procedures that are specific to a particular instrument or this group of instruments.

A2.3.2 Select a clean, dry viscometer that will give a flow time greater than 60 s.

A2.3.3 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 may contain lint, solid particles, etc., filter as prescribed in Test Method D445.

A2.3.3.1 To charge the Cannon-Fenske Opaque viscometer, invert the viscometer and apply suction to the tube *L*, immersing tube *N* in the liquid sample. Draw liquid through tube *N*, filling bulb *D* to fill mark *G*. Wipe excess sample off tube *N* and invert the viscometer to its normal position. Mount the viscometer in the constant-temperature bath, keeping tube *L* vertical. When the sample travels through capillary *R* and fills bulb *A* approximately half full, arrest its flow by placing a stopper in the tube *L*.

A2.3.3.2 Mount the Zeitfuchs Cross-Arm viscometer in the constant-temperature bath, keeping tube *N* vertical. Introduce sample through tube *N* taking care not to wet the sides of tube *N*, into the cross-arm *D* until the leading edge stands within 0.5 mm of fill mark *G* on the siphon tube.

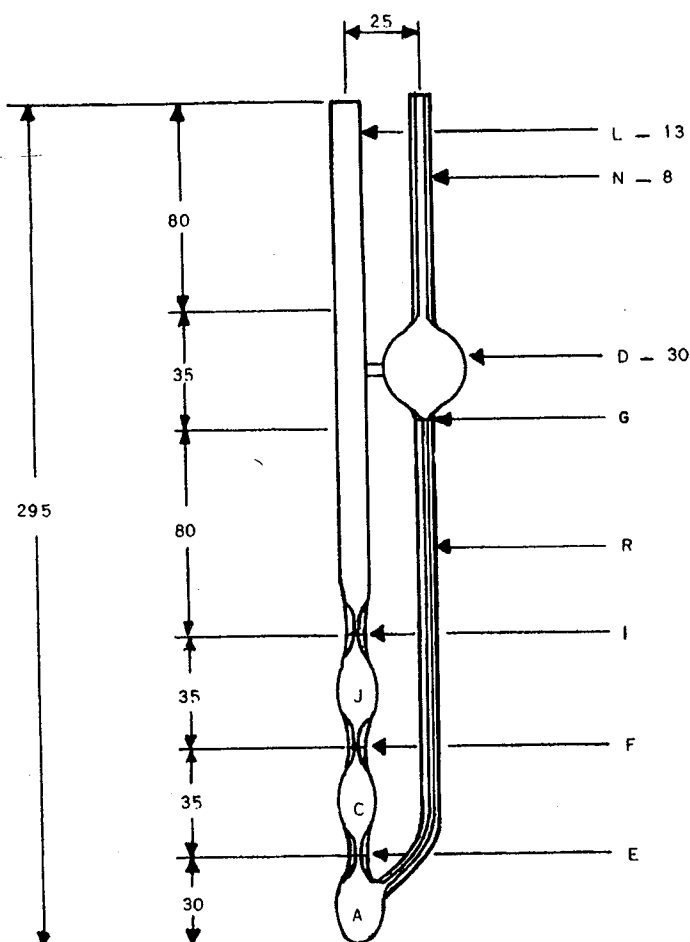
NOTE A2.1—The volume of the test specimen is dependent on the location of the fill mark *G*. When the flow time of the lower meniscus is being measured between timing marks *E* and *F*, the upper meniscus shall be in the horizontal cross-arm *D*, thus making the location of fill mark *G* critical.

A2.3.3.3 Mount the Lantz-Zeitfuchs viscometer in the constant-temperature bath keeping tube *N* vertical. Introduce sufficient sample through tube *N* to completely fill bulb *D*, overflowing slightly into overflow bulb *K*. If the sample is poured at a temperature above test temperature, wait 15 min for the sample in the viscometer to attain bath temperature and add more sample to overflow slightly into bulb *K*. This 15-min period is part of and not in addition to the equilibration time of A2.3.4.

A2.3.3.4 Mount the BS/IP/RF viscometer in the constant-temperature bath keeping tube *L* vertical. Pour sample through tube *N* to a point just above filling mark *G*; allow the sample to flow freely through capillary *R*, taking care that the liquid column remains unbroken, until the lower meniscus is about 5

³ Cannon, M. R., and Fenske, M. R., "Viscosity Measurement—Opaque Liquids," *Industrial and Engineering Chemistry*, Analytical Edition, Vol 13, 1941, p. 2910.

⁴ Zeitfuchs, E. H., "Kinematic Viscometer for Opaque and Very Viscous Liquids," *Oil and Gas Journal*, Vol 44, No. 36, 1946, p. 98.


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NOTE 1—All dimensions are in millimetres.

Dimensions and Kinematic Viscosity Ranges

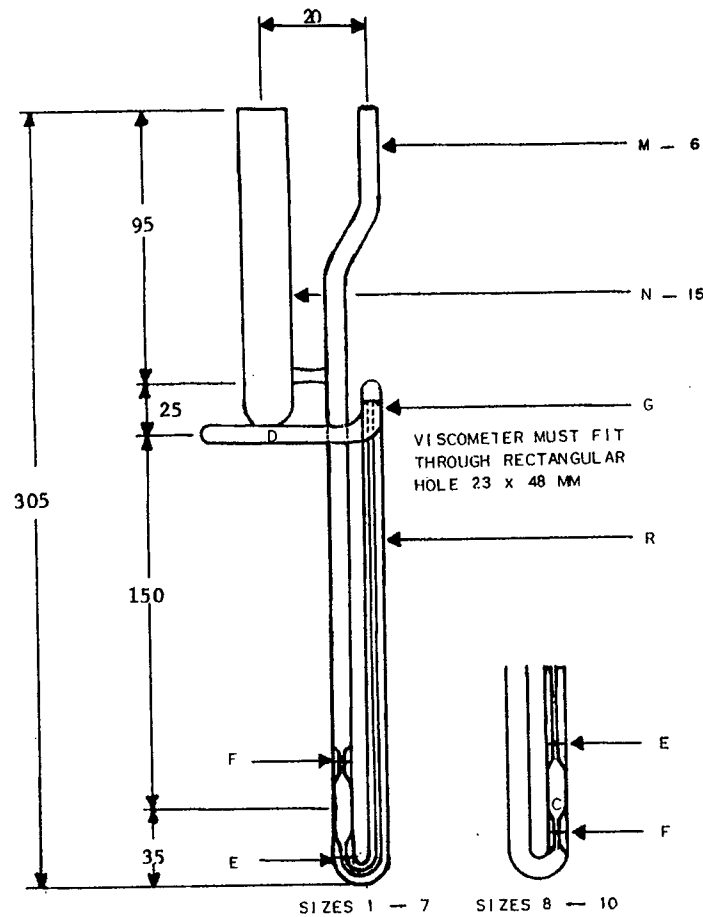
Size No.	Approximate Constant, mm^2/s [cSt/s]	Kinematic Viscosity Range, mm^2/s [cSt]	Inside Diameter of Tube, R, mm ($\pm 2\%$)	Inside Diameter of Tube N and G Tubes E, F, and I, mm ($\pm 5\%$)	Volume Bulbs A, C, and J, mL ($\pm 5\%$)	Volume Bulb D, mL ($\pm 5\%$)
200	0.1	6 to 100	1.02	3.2	2.1	11
300	0.25	15 to 250	1.26	3.4	2.1	11
350	0.5	30 to 500	1.48	3.4	2.1	11
400	1.2	72 to 1200	1.88	3.4	2.1	11
450	2.5	150 to 2500	2.20	3.7	2.1	11
500	8	480 to 8000	3.10	4.0	2.1	11
600	20	1200 to 20 000	4.00	4.7	2.1	13

FIG. A2.1 Cannon-Fenske Opaque Viscometer for Opaque and Transparent Liquids

mm below the filling mark *H* and then arrest its flow by closing the timing tube with a cork or rubber stopper in tube *L*. Add more liquid if necessary to bring the upper meniscus slightly above mark *G*. After allowing the sample to attain bath temperature (see A2.3.4) and any air bubbles to rise to the surface, gently loosen the stopper allowing the sample to flow to the lower filling mark *H* and again arrest flow. Remove the excess sample above filling mark *G* by inserting the special pipet until its cork rests on top of tube *N*; apply gentle suction until air is drawn through. The upper meniscus shall coincide with mark *G*.

A2.3.4 Allow the viscometer to remain in the constant-temperature bath a sufficient time to ensure that the sample reaches temperature equilibrium (10 min minimum, 30 min maximum).

A2.3.5 For the Cannon-Fenske Opaque and BS/IP/RF viscometers, remove the stopper in tubes *N* and *L*, respectively, and allow the sample to flow by gravity. For the Zeitfuchs Cross-Arm viscometer, apply slight vacuum to tube *M* (or pressure to tube *N*) to cause the meniscus to move over the siphon tube and about 30 mm below the level of tube *D* in


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NOTE 1—All dimensions are in millimetres.

Dimensions and Kinematic Viscosity Ranges

Size	Approximate Constant, mm^2/s^2 [cSt/s]	Kinematic Viscosity Range, mm^2/s [cSt]	Inside Diameter of Tube R, mm ($\pm 2\%$)	Length of Tube R, mm ($\pm 2\%$)	Lower Bulb Volume, mL ($\pm 5\%$)	Horizontal Tube Diameter, mm ($\pm 5\%$)
4	0.10	6 to 100	0.64	210	0.3	3.9
5	0.3	18 to 300	0.84	210	0.3	3.9
6	1.0	60 to 1000	1.15	210	0.3	4.3
7	3.0	180 to 3000	1.42	210	0.3	4.3
8	10.0	600 to 10 000	1.93	165	0.25	4.3
9	30.0	1800 to 30 000	2.52	165	0.25	4.3
10	100.0	6000 to 100 000	3.06	165	0.25	4.3

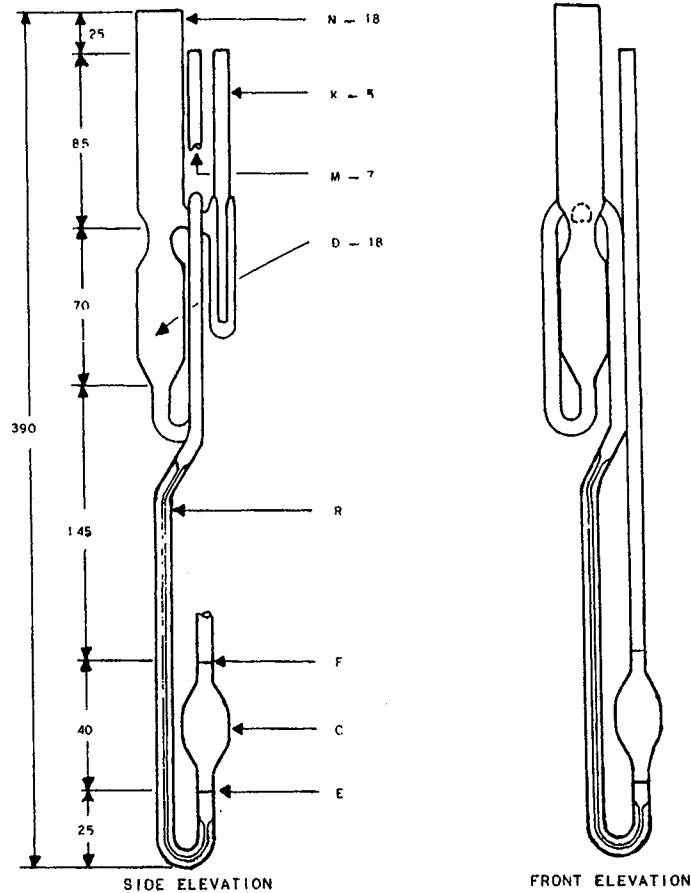
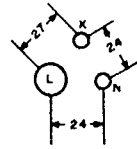
FIG. A2.2 Zeifuchs Cross-Arm Viscometer for Transparent and Opaque Liquids

capillary R; gravity flow is thus initiated. For the Lantz-Zeifuchs viscometer, apply slight vacuum to tube M (or pressure to tube N with tube K closed) until the lower meniscus is opposite the lower timing mark E; allow the sample to flow by gravity.

A2.3.6 Measure to the nearest 0.1 s the time required for the leading edge of the meniscus to pass from timing mark E to timing mark F as prescribed in Section 8 of this method.

A2.3.7 Calculate the viscosity as prescribed in Section 9 of this method.

A2.3.8 Clean the viscometer as prescribed in 8.8 of this method.

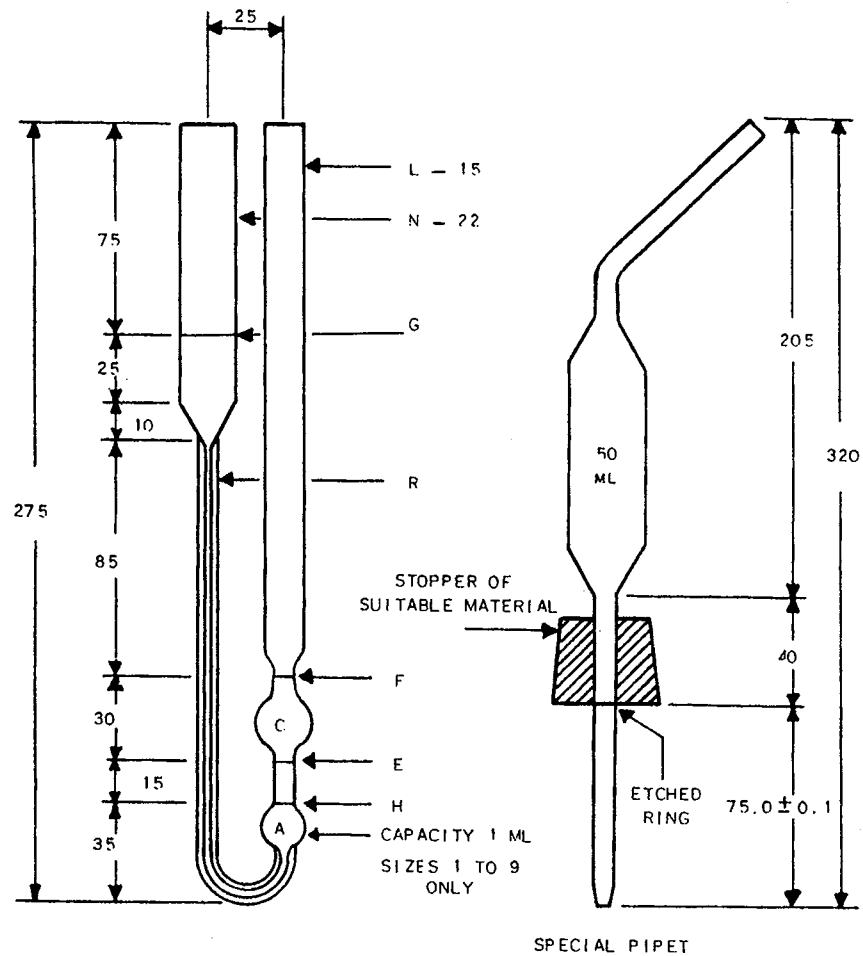

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NOTE 1—All dimensions are in millimetres.

Dimensions and Kinematic Viscosity Ranges

Size No.	Approximate Constant, $\text{mm}^2/\text{s}^2[\text{cSt}/\text{s}]$	Kinematic Viscosity Range, $\text{mm}^2/\text{s} [\text{cSt}]$	Inside Diameter of Tube <i>R</i> , mm ($\pm 2\%$)	Length of Tube <i>R</i> , mm ($\pm 2\%$)	Volume, Bulb <i>C</i> , mL ($\pm 5\%$)
5	0.3	18 to 300	1.65	490	2.7
6	1.0	60 to 1000	2.25	490	2.7
7	3.0	180 to 3000	3.00	490	2.7
8	10.0	600 to 10 000	4.10	490	2.7
9	30.0	1800 to 30 000	5.20	490	2.7
10	100.0	6000 to 100 000	5.20	490	0.85

FIG. A2.3 Lantz-Zeitfuchs Type Reverse Flow Viscometer for Opaque Liquids


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NOTE 1—All dimensions are in millimetres.

Dimensions and Kinematic Viscosity Ranges

Size No.	Approximate Constant, $\text{mm}^2/\text{s}^2[\text{cSt}/\text{s}]$	Kinematic Viscosity Range, mm^2/s [cSt]	Inside Diameter of Tube R, mm ($\pm 2\%$)	Length of Tube R, mm ($\pm 2\%$)	Inside Diameter at E, F, and G, mm ($\pm 2\%$)	Volume Bulb C, mL ($\pm 5\%$)
4	0.1	6 to 100	1.26	185	3.0 to 3.3	4.0
5	0.3	18 to 300	1.64	185	3.0 to 3.3	4.0
6	1.0	60 to 1000	2.24	185	3.0 to 3.3	4.0
7	3.0	180 to 3000	2.93	185	3.3 to 3.6	4.0
8	10	600 to 10 000	4.00	185	4.4 to 4.8	4.0
9	30	1800 to 30 000	5.5	185	6.0 to 6.7	4.0
10	100	6000 to 100 000	7.70	210	7.70	4.0
11	300	18 000 to 300 000	10.00	210	10.00	4.0

FIG. A2.4 BS/IP/RF U-Tube Reverse Flow Viscometer for Opaque Liquids

A3. CALIBRATION OF VISCOMETERS

TABLE A3.1 Viscosity Oil Standards

Viscosity Oil Standards Conforming to ASTM Standards	Approximate Kinematic Viscosity, mm ² /s [cSt]		
	At 40°C [104°F]	At 50°C [122°F]	At 100°C [212°F]
S 60	54	35	7.5
S 200	180	110	20
S 600	520	310	50
S 2000	1600	800	72
S 8000	6700	3200	240
S 30 000	23 000	11 000	630

A3.1 Scope

A3.1.1 This annex describes the materials and procedures used for calibrating or checking the calibration of viscometers used in this method.

A3.2 Reference Materials

A3.2.1 *Viscosity Oil Standards* conforming to ASTM viscosity oil standards⁵ having the approximate kinematic viscosities shown in Table A3.1. Certified kinematic viscosity values established are supplied with each sample.

A3.3 Calibration

A3.3.1 *Calibration of Routine Viscometer by Means of Liquid Viscosity Standards*—Select from Table 1 a liquid standard having a minimum efflux time of 200 s at the calibration temperature (preferably 37.8°C [100°F]). Determine the efflux time to the nearest 0.1 s by the procedure described in Section 8, and calculate the viscometer constant, C , as follows:

$$C = v/t \quad (\text{A3.1})$$

where:

v = viscosity for the standard liquid, mm²/s [cSt] and
 t = efflux time, s.

A3.3.2 The viscometer constant is independent of temperature for the Zeitfuchs Cross-Arm, Lantz-Zeitfuchs and BS/IP/RF U-Tube viscometers.

A3.3.3 The Cannon-Fenske Opaque viscometer has a fixed volume of sample charged at the fill temperature. If the test temperature differs from the fill temperature, the viscometer constant is calculated as follows:

$$\text{Viscometer constant, } C_t = C_o [1 + F(T_t - T_f)] \quad (\text{A3.2})$$

where:

C_o = viscometer constant when filled and tested at the same temperature,
 T = temperature,

⁵ The viscosity oil standards are available in 1-pt containers from Cannon Instrument Co. 2139 High Tech Road, State College, Pa. 16803. Additionally the Cannon Instrument Co. has available viscosity standards N600, N2000, and N8000, which have approximate kinematic viscosities at 60°C [140°F] of 160 mm²/s [cSt], 440 mm²/s [cSt], and 1600 mm²/s [cSt], respectively; standards N600 and N2000 have approximate kinematic viscosities at 135°C [275°F] of 12 and 26 mm²/s [cSt] respectively.

F = temperature dependence factor (see A3.3.4), and
 t, f subscripts = values at the test temperature and at the fill temperature, respectively.

A3.3.4 Calculate the temperature dependence constant by means of the following equation:

$$\text{Factor, } F = 4\alpha V/\pi d^2 h = [4V(\rho_f - \rho_t)]/[\pi d^2 h \rho_t (T_t - T_f)] \quad (\text{A3.3})$$

where:

v = volume of charge, cm³,
 d = average diameter, cm, of the meniscus in the upper reservoir,
 h = average driving head, cm,
 α = coefficient of thermal expansion of the test sample between the fill temperature and the test temperature,
 ρ = density, g/cm³, and
 t, f subscripts = as defined in A3.3.3.

A3.3.5 If the viscometer is used at a location other than the calibrating laboratory, the C constant should be corrected for the difference in the acceleration of gravity, g , at the two locations as follows:

$$C_2 = (g_2/g_1) \times C_1 \quad (\text{A3.4})$$

where:

C_2 = the calibration constant in the testing laboratory,
 C_1 = the calibration constant in the calibrating laboratory,
 g_2 = the acceleration of gravity at the testing laboratory, and
 g_1 = the acceleration of gravity at the calibrating laboratory.

Certificates for viscometers should state the value of g at the location of the calibrating laboratory. Failure to correct for gravity can result in errors of 0.2 %.

A3.3.6 *Calibration of Routine Viscometer by Means of Standard Viscometer*—Select any petroleum oil having an efflux time of at least 200 s. Select also a standard viscometer of known C constant. This viscometer may be a master viscometer which has been calibrated by the “step-up” procedure using viscometers of successively larger diameters starting with distilled water at the basic viscosity standard (see Test Method D2162) or a routine viscometer of the same type that has been calibrated by comparison with a master viscometer. Calibrated viscometers are available from a number of commercial suppliers.

A3.3.6.1 Mount the standard viscometer together with the viscometer to be calibrated in the same bath and determine the efflux times of the oil by the procedure described in Section 8.

A3.3.6.2 Calculate the C constant as follows:

$$C_1 = (t_2 \times C_2)/t_1 \quad (\text{A3.5})$$

where:

C_1 = C constant of routine viscometer,
 t_1 = efflux time to nearest 0.1 s in routine viscometer,
 C_2 = C constant of standard viscometer, and
 t_2 = efflux time to nearest 0.1 s in standard viscometer.

APPENDIX**(Nonmandatory Information)****X1. ICE POINT DETERMINATION AND RECALIBRATION OF LIQUID-IN-GLASS KINEMATIC VISCOSITY THERMOMETERS**

X1.1 To achieve an accuracy of $\pm 0.02^{\circ}\text{C}$ [$\pm 0.04^{\circ}\text{F}$] for calibrated liquid-in-glass kinematic viscosity thermometers, it is required that a check at the ice point be made and the corrections altered for the change seen in the ice point. It is recommended that the interval of checking be every six months; for a new thermometer, check monthly for the first six months.

X1.2 A detailed procedure for the measurement of the ice point and recalibration of thermometers is described in 6.4 of Test Method E77. The suggestions in the following sections of this appendix are given specifically for the mercury-in-glass kinematic viscosity thermometers and may not apply to other thermometers.

X1.2.1 The ice point reading of liquid-in-glass kinematic viscosity thermometers shall be taken within 60 min after being at the test temperature for not less than 3 min. The ice point reading shall be expressed to the nearest 0.01°C or 0.02°F .

X1.2.2 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 crushed ice and add sufficient distilled and preferably pre-cooled 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 packing the ice gently about the stem, to a depth approximately one scale division below the 0°C [32°F] graduation. It may be necessary to repack the ice around the thermometer because of melting.

X1.2.3 After at least 3 min have elapsed, tap the stem gently, and observe the reading. Successive readings taken at least 1 min apart should agree within one tenth of a division.

X1.2.4 Record the ice point reading and compare it with the previous reading. If the reading is found to be higher or lower than the reading corresponding to a previous calibration, readings at all other temperatures will be correspondingly increased or decreased.

X1.2.5 The ice point procedure given in X1.1-X1.2.4 is used for the recalibration of liquid-in-glass kinematic viscosity thermometers, and a complete new calibration of the thermometer is not necessary in order to meet the accuracy ascribed to this design thermometer.

X1.3 It is recommended that these kinematic viscosity thermometers be stored vertically when not in use so as to avoid the separation of the liquid column.

X1.4 It is recommended that these kinematic viscosity thermometers be read to the nearest $\frac{1}{5}$ of a division using appropriate magnification. Since these thermometers are typically in a kinematic viscosity bath (which has vision through the front), the thermometer is read by lowering the thermometer such that the top of the liquid column is 5 to 15 mm below the surface of the bath liquid. Be careful to ensure that the expansion chamber at the top of the thermometer is above the lid of the constant temperature bath. If the expansion chamber is at elevated or lowered temperatures from ambient temperatures, a significant error can occur. This error can be as much as one or two thermometer divisions. A reading glass such as used for reading books may be useful to ensure reading the scale to $\frac{1}{5}$ of a division.

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Designation: D95 – 05 (Reapproved 2010)

Highway Transportation Standard
AASHTO No. T55



Designation: Manual of Petroleum Measurement Standards (MPMS), Chapter 10.5

Designation: 74/82 (88)

Standard Test Method for Water in Petroleum Products and Bituminous Materials by Distillation¹

This standard is issued under the fixed designation D95; 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 water in the range from 0 to 25 % volume in petroleum products, tars, and other bituminous materials by the distillation method.

NOTE 1—Volatile water-soluble material, if present, may be measured as water.

1.2 The specific products considered during the development of this test method are listed in Table 1. For bituminous emulsions refer to Test Method D244. For crude oils, refer to Test Method D4006 (API *MPMS* Chapter 10.2).

NOTE 2—With some types of oil, satisfactory results may be obtained from Test Method D1796 (API *MPMS* Chapter 10.6).

1.3 The values stated in SI units are to be regarded as the standard. The values 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 Section 6.

2. Referenced Documents

2.1 *ASTM Standards*:²

D244 Test Methods and Practices for Emulsified Asphalts

D1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)

D4006 Test Method for Water in Crude Oil by Distillation
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products

E123 Specification for Apparatus for Determination of Water by Distillation

2.2 *API Standards*:³

MPMS Chapter 8.1 Manual Sampling of Petroleum and Petroleum Products (ASTM Practice D4057)

MPMS Chapter 8.2 Automatic Sampling of Petroleum and Petroleum Products (ASTM Practice D4177)

MPMS Chapter 8.3 Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products (ASTM Practice D5854)

MPMS Chapter 10.2 Determination of Water in Crude Oil by the Distillation Method (ASTM Test Method D4006)

MPMS Chapter 10.6 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure) (ASTM Test Method D1796)

3. Terminology

3.1 *Definitions*:

3.1.1 *bituminous material, n—in petroleum technology*, a black or dark-colored very viscous liquid or semi-solid composed principally of high molecular weight condensed aromatic, or naphthenic compounds, or both.

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and the API Committee on Petroleum Measurement, and is the direct responsibility of Subcommittee D02.02.01 the joint ASTM-API Committee on Temperature Determination.

Current edition approved May 1, 2010. Published May 2010. Originally approved in 1921. Last previous edition approved in 2005 as D95–05. DOI: 10.1520/D0095-05R10.

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 Published as Manual of Petroleum Measurement Standards. Available from American Petroleum Institute (API), 1220 L. St., NW, Washington, DC 20005-4070, <http://www.api.org>.

4. Summary of Test Method

4.1 The material to be tested is heated under reflux with a water-immiscible solvent, which co-distills with the water in the sample. Condensed solvent and water are continuously separated in a trap, the water settling in the graduated section of the trap and the solvent returning to the still.

5. Significance and Use

5.1 A knowledge of the water content of petroleum products is important in the refining, purchase, sale, and transfer of products.

5.2 The amount of water as determined by this test method (to the nearest 0.05 or 0.1 volume %, depending on the trap size used) may be used to correct the volume involved in the custody transfer of petroleum products and bituminous materials.

5.3 The allowable amount of water may be specified in contracts.

6. Solvent-Carrier Liquid

6.1 A solvent-carrier liquid appropriate to the material being tested (see Table 1) shall be used.

6.1.1 *Aromatic Solvent*—The following aromatic solvents are acceptable:

6.1.1.1 *Industrial Grade Xylene*—(Warning—Flammable. Vapor harmful.)

6.1.1.2 A blend of 20 volume % industrial grade toluene and 80 volume % industrial grade xylene. (Warning—Flammable. Vapor harmful.)

6.1.1.3 *Petroleum Naphtha or Coal Tar Naphtha*, free of water, yielding not more than 5% distillates at 125°C (257°F) and not less than 20% at 160°C (320°F) and with a relative density (specific gravity) not lower than 0.8545 at 15.56/15.56°C (60/60°F). (Warning—Extremely flammable. Harmful if inhaled. Vapors may cause fire.)

6.1.2 *Petroleum Distillate Solvent*—A petroleum distillate solvent, 5% boiling between 90 and 100°C (194 and 212°F) and 90% distilling below 210°C (410°F), shall be used. Percent may be determined by mass or by volume. (Warning—Flammable. Vapor harmful.)

6.1.3 *Volatile Spirits Solvent*—The following volatile spirits solvents are acceptable:

6.1.3.1 *Petroleum Spirit*, with a boiling range from 100 to 120°C (212 to 248°F). (Warning—Flammable. Vapor harmful.)

6.1.3.2 *Iso-octane*, of 95% purity or better. (Warning—Extremely flammable. Harmful if inhaled. Vapors may cause fire.)

TABLE 1 Type of Solvent-Carrier Liquid Versus Material to Be Tested

Type of Solvent-Carrier Liquid	Material to be Tested
Aromatic	asphalt, tar, coal tar, water gas tar, road tar, cut-back bitumin, liquid asphalt, tar acid
Petroleum distillate	road oil, fuel oil, lubricating oil, petroleum sulfonates
Volatile spirits	lubricating grease

6.2 *Solvent Blank*—The water content of the solvent shall be determined by distilling an equivalent amount of the same solvent used for the test sample in the distillation apparatus and testing as outlined in Section 10. The blank shall be determined to the nearest scale division and used to correct the volume of water in the trap in Section 11.

7. Apparatus

7.1 *General*—The apparatus comprises a glass or metal still, a heater, a reflux condenser, and a graduated glass trap. The still, trap, and condenser may be connected by any suitable method that produces a leakproof joint. Preferred connections are ground joints for glass and O-rings for metal to glass. Typical assemblies are illustrated in Fig. 1, Fig. 2, and Fig. 3. The stills and traps should be chosen to cover the range of materials and water contents expected. On assembly, care shall be taken to prevent the joints from freezing or sticking. Always apply a very thin film of stopcock grease to prevent the glassware joints from seizing.

7.2 *Still*—A glass or metal vessel with a short neck and suitable joint for accommodating the reflux tube of the trap shall be used. Vessels of 500, 1000, and 2000-mL nominal capacity have proved satisfactory.

7.3 *Heater*—A suitable gas burner or electric heater may be used with the glass still. A gas ring burner with ports on the inside circumference shall be used with the metal still. The gas ring burner shall be of such dimensions that it may be moved up and down the vessel when testing materials that are likely to foam or solidify in the still.

7.4 *Glassware*—Dimensions and descriptions of typical glassware for use in this test method are provided in Specification E123.

NOTE 3—Instead of standardizing on a particular apparatus specification with respect to dimensions and style, a given apparatus will be deemed satisfactory when accurate results are obtained by the standard addition technique described in Section 9.

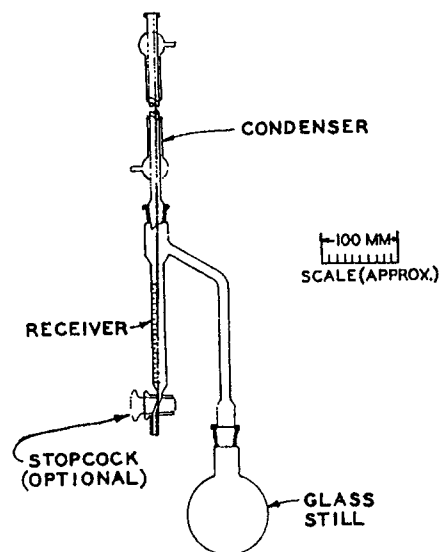


FIG. 1 Typical Assembly with Glass Still

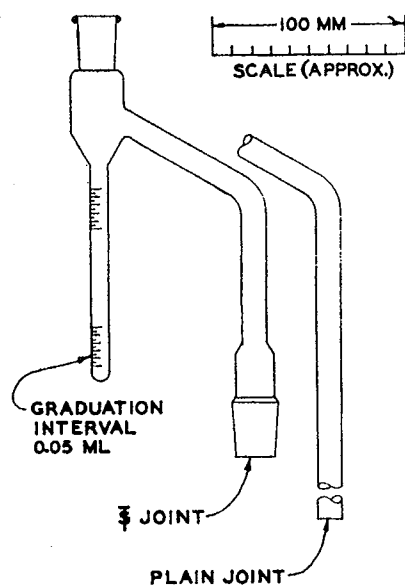


FIG. 2 Two-millilitre Receiver Showing Alternative Connections to Glass Still

8. Sampling

8.1 Sampling is defined as all steps required to obtain an aliquot of the contents of any pipe, tank, or other system and to place the sample into the laboratory test container. Only representative samples obtained as specified in Practices D4057 (API *MPMS* Chapter 8.1) and D4177 (API *MPMS* Chapter 8.2) shall be used for this test method.

8.2 The size of the test portion should be based on the expected water content of the sample, such that the water yield does not exceed the capacity of the trap (unless a trap with a stopcock is used permitting excess water to be withdrawn into a graduated cylinder).

8.3 Practice D5854 (API *MPMS* Chapter 8.3) contains information on sampling and homogenization efficiency of unknown mixers. This test method should not be followed without strict adherence to Practice D5854 (API *MPMS* Chapter 8.3).

9. Verification

9.1 The accuracy of the graduation marks on the trap shall be certified or verified, using only national or international standards, such as National Institute of Standards and Technology (NIST)⁴ traceable equipment. Verification shall be with a traceable 5 mL Micro Burette or Micro Pipette, readable to the nearest 0.01 mL.

9.1.1 In styles A, B, C, and D, as specified in Table 2 (Table 1 in Specification E123), each subdivision (that is, 0.1 mL through 1.0 mL) in the conical portion of the tube shall be verified. Thereafter, each major subdivision (that is, 2.0 mL, 3.0 mL, 4.0 mL, and up to the total volume of the trap) shall be verified.

9.1.2 In styles E and F, as specified in Table 2, each major subdivision (0.1 mL, 1.0 mL, 2.0 mL, 4.0 mL, and 5.0 mL in

the case of Style E; 0.05 mL, 0.5 mL, 1.0 mL, 1.5 mL, and 2.0 mL in the case of Style F) shall be verified.

9.2 The entire glassware assembly shall be verified prior to first use and at a regular frequency thereafter as follows.

9.2.1 Put 400 mL of dry (0.02 % water maximum) xylene or the solvent to be utilized in the analysis of unknown samples into the apparatus and test in accordance with Section 10. When complete, discard the contents of the trap and add the volume of water as specified as first test in Table 3 directly to the distillation flask and test in accordance with Section 10.

9.2.2 Repeat the test in 9.2.1, and add the volume specified as second test in Table 3 directly to the flask. The assembly of the apparatus is satisfactory only if the trap readings are within the tolerances specified in Table 3.

9.3 A reading outside the permissible limits suggests a malfunction resulting from vapor leaks, too rapid boiling, inaccuracies in calibration of the trap, or ingress of extraneous moisture. Eliminate these factors before repeating the verification.

10. Procedure

NOTE 4—The precision of this test method will be affected by water droplets adhering to surfaces in the apparatus and therefore not settling into the water trap to be measured. To minimize the problem, all apparatus must be cleaned chemically at least daily to remove surface films and debris, which hinder free drainage of water in the test apparatus. More frequent cleaning is recommended if the nature of samples being run causes persistent contamination.

10.1 Measure a suitable amount of sample to an accuracy of $\pm 1\%$ and transfer it to the still.

10.2 Measure ordinary liquid samples in a graduated cylinder of an appropriate size. Rinse the material adhering to the cylinder with one 50-mL and two 25-mL portions of the solvent-carrier liquid (see Section 6 and Table 1). Drain the cylinder thoroughly after the sample transfer and each rinsing.

10.3 Weigh solid or viscous materials directly into the still and add 100 mL of the selected solvent-carrier liquid. In cases of material with a low-water content when large samples must be used, a solvent-carrier liquid volume in excess of 100 mL may be necessary.

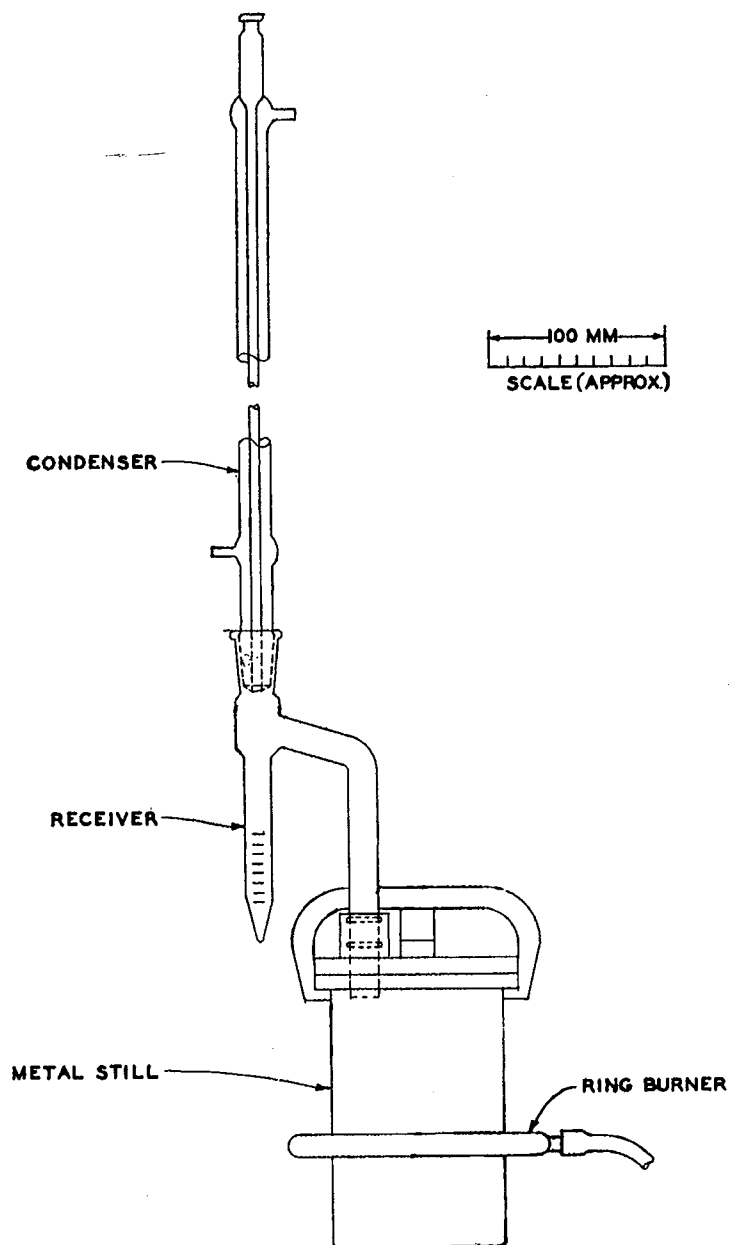
10.4 Glass beads or other boiling aids may be added, if necessary, to reduce bumping.

10.5 Assemble the components of the apparatus, as illustrated in Fig. 1, Fig. 2, and Fig. 3, choosing the trap in accordance with the expected water content of the sample and making all connections vapor and liquid tight. If a metal still with a removable cover is used, insert a gasket of heavy paper, moistened with solvent, between the still body and the cover. The condenser tube and trap must be chemically clean to ensure free drainage of water into the bottom of the trap. Insert a loose cotton plug in the top of the condenser to prevent condensation of atmospheric moisture inside it. Circulate cold water through the jacket of the condenser.

10.6 Apply heat to the still, adjusting the rate of boiling so that condensed distillate discharges from the condenser at the rate of two to five drops per second. If the metal still is used, start heating with the ring burner about 76 mm (3 in.) above the bottom of the still and gradually lower the burner as the distillation proceeds. Continue distillation until no water is

⁴ National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, <http://www.nist.gov>.

D95 - 05 (2010)




NOTE—Trap shall be 15 to 16 mm in inside diameter.

FIG. 3 Typical Assemblies with Metal Still

TABLE 2 Specifications and Sizes of Traps

Description							
Style	Top of Graduated Tube	Bottom of Graduated Tube	Bottom of Vapor Tube	Size of Trap mL	Range mL	Smallest Scale Division, mL	Scale Error Maximum, mL
A	ST Joint	Conical	ST Joint	10	0 to 1.0	0.1	0.05
					>1.0 to 10.0	0.2	0.1
B	ST Joint	Conical	ST Joint	25	0 to 1.0	0.1	0.05
					>1.0 to 25	0.2	0.1
C	ST Joint	Conical	Plain	25	1.0 to 25	0.2	0.1
						0.2	0.1
D	ST Joint	Conical	Plain	25	0 to 5.0	0.1	0.05
						0.1	0.05
E	ST Joint	Round	ST Joint	5	0 to 5.0	0.05	0.025
						0.1	0.1
F	ST Joint	Round	ST Joint	2	0 to 10.0	0.1	0.1
						0.05	0.025


D95 – 05 (2010)
TABLE 3 Permissible Limits in Millilitres

Capacity of Receiver at 20°C		Volume of Water Added to Flask at 20°C	Permissible Limits for Recovered Water at 20°C
Round trap 2	1 st Test	1	1 ± 0.05
	2 nd Test	1.9	1.9 ± 0.05
5 (0.05 mL subdivisions)	1 st Test	1	1 ± 0.05
	2 nd Test	4.5	4.5 ± 0.05
5 (0.1 mL subdivisions)	1 st Test	1	1 ± 0.1
	2 nd Test	4.5	4.5 ± 0.1
10	1 st Test	5	5 ± 0.1
	2 nd Test	9	9 ± 0.1
Conical trap 10	1 st Test	1	1 ± 0.1
	2 nd Test	9	9 ± 0.2
25	1 st Test	12	12 ± 0.2
	2 nd Test	24	24 ± 0.2

visible in any part of the apparatus except in the trap and the volume of water in the trap remains constant for 5 min. If there is a persistent ring of water in the condenser tube, carefully increase the rate of distillation or cut off the condenser water for a few minutes.

10.7 When the evolution of water is complete, allow the trap and contents to cool to room temperature. Dislodge any drops of water adhering to the sides of the trap with a glass or polytetrafluoroethylene (PTFE) rod or other suitable means and transfer them to the water layer. Read the volume of the water in the trap to the nearest scale division.

10.8 A solvent blank shall be established, as outlined in 6.2.

11. Calculation

11.1 Calculate the water in the sample, as weight or volume percent, in accordance with the basis on which the sample was taken, as follows:

11.1.1 Water, % (V/V) =

$$\frac{(\text{Volume in water trap, mL}) - (\text{Water in solvent blank, mL})}{\text{Volume in test sample, mL}} \times 100 \quad (1)$$

11.1.2 Water, % (V/m) =

$$\frac{(\text{Volume of water in trap, mL}) - (\text{Water in solvent blank, mL})}{\text{Mass of test sample, g}} \times 100 \quad (2)$$

12. Report

12.1 Report the results as the water content to the nearest 0.05% if the 2-mL receiver has been used and to the nearest 0.1% if the 10-mL or 25-mL receiver has been used and to the nearest subdivision if a 5-mL receiver has been used with a 100-mL or 100-g sample.

13. Precision and Bias

13.1 *Precision*—The criteria described in 13.1.1 and 13.1.2 should be used to judge the acceptability of results when using the 10 or 25-mL traps. The precision when using the 2-mL trap or a 5-mL trap has not been established.

NOTE 5—Practice D6300 was not used in obtaining precision data.

13.1.1 *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 values in Table 4 in only one case in twenty.

13.1.2 *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 the test method, exceed the values in Table 4 in only one case in twenty.


13.2 *Bias*—As there is no accepted reference material suitable for determining bias for the procedure described in this test method for measuring water in petroleum products and bituminous materials by distillation, no statement about bias is made.

14. Keywords

14.1 bituminous materials; distillation; petroleum products; solvent carrier liquid; water by distillation; water content

TABLE 4 Precision

Type	Water Collected, mL	Difference, mL
Repeatability	0.0–1.0	0.1
	1.1–25	0.1 mL or 2 % of the mean, whichever is greater
Reproducibility	0.0–1.0	0.2
	1.1–25	0.2 mL or 10 % of the mean, whichever is greater

 **D95 – 05 (2010)**

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Designation: D 402 – 08

American Association State
Highway and Transportation Officials Standard
AASHTO No.: T78

27/74 (88)

Standard Test Method for Distillation of Cutback Asphaltic (Bituminous) Products¹

This standard is issued under the fixed designation D 402; 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. This method was adopted as a joint ASTM-IP standard in 1961.

1. Scope

1.1 This test method covers a distillation test for cutback asphaltic (bituminous) products.

1.2 The values given in SI units are to be regarded as the standard. The inch-pound units given 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 limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

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

D 370 Practice for Dehydration of Oil-Type Preservatives

E 1 Specification for ASTM Liquid-in-Glass Thermometers

E 133 Specification for Distillation Equipment

E 220 Test Method for Calibration of Thermocouples By Comparison Techniques

2.2 IP Standards:

IP 123/ASTM D 86, Distillation of Petroleum Products Thermometers as specified in IP Standards

Crow Receiver as specified in British Standards 658:1989

¹ This test method is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.46 on Durability and Distillation Tests.

Current edition approved July 15, 2008. Published August 2008. Originally approved in 1934. Last previous edition approved in 2002 as D 402 – 02.

In the IP, this 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.

C.O.3—Standard Methods for Testing Tar and its Products
(Published by the U.K. Standardization of Tar Products
Tests Committee)

3. Summary of Method

3.1 Two hundred millilitres of the sample are distilled in a 500-mL flask, at a controlled rate, to a temperature in the liquid of 360°C (680°F), and the volumes of distillate obtained at specified temperatures are measured. The residue from the distillation, and also the distillate, may be tested as required.

4. Significance and Use

4.1 This procedure measures the amount of the more volatile constituents in cutback asphaltic products. The properties of the residue after distillation are not necessarily characteristic of the bitumen used in the original mixture, nor of the residue which may be left at any particular time after field application of the cutback asphaltic product. The presence of silicone in the cutback may affect the distillation residue by retarding the loss of volatile material after the residue has been poured into the residue container.

5. Apparatus

5.1 *Distillation Flask*, 500-mL side-arm, having the dimensions shown in Fig. 1.

5.2 *Condenser*, standard glass-jacketed, of nominal jacket length from 200 to 300 mm and overall tube length of 450 ± 10 mm (see Fig. 3).

5.3 *Adapter*, heavy-wall (1-mm) glass, with reinforced top, having an angle of approximately 105°. The inside diameter at the large end shall be approximately 18 mm, and at the small end, not less than 5 mm. The lower surface of the adapter shall be on a smooth descending curve from the larger end to the smaller. The inside line of the outlet end shall be vertical, and the outlet shall be cut or ground (not fire-polished) at an angle of $45 \pm 5^\circ$ to the inside line.

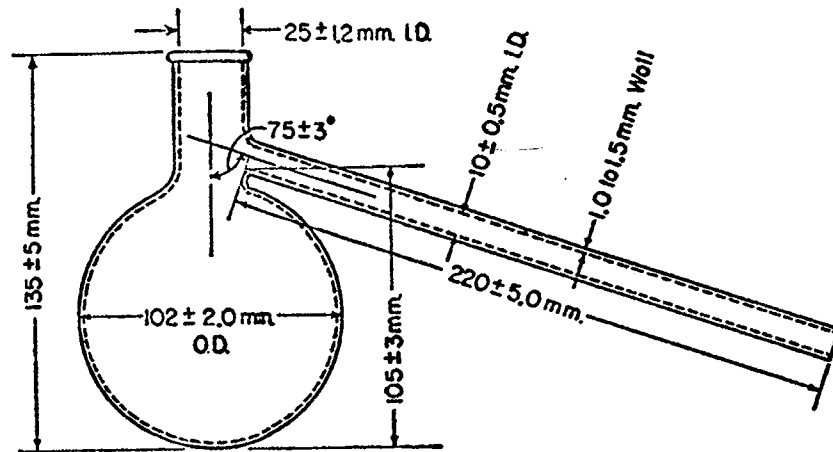


FIG. 1 Distillation Flask

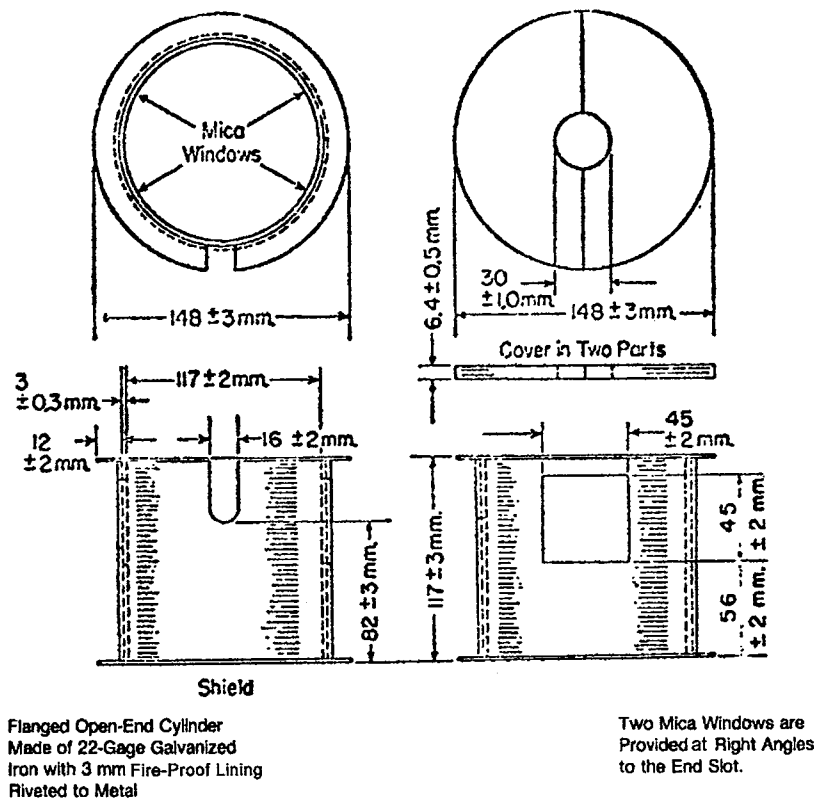


FIG. 2 Shield

5.4 *Shield*, steel, lined with 3-mm fire proof insulation and fitted with transparent mica windows, of the form and dimensions shown in Fig. 2, used to protect the flask from air currents and to reduce radiation. The cover (top) shall be made in two parts of 6.4-mm fire proof insulation.

5.5 *Shield and Flask Support*—Two 15-cm² sheets of 16-mesh Chromel wire gauze on a tripod or ring.

5.6 *Heat Source*—

5.6.1 Adjustable Tirrill-type gas burner or equivalent.

5.6.2 An electric heater equipped with a transformer capable of controlling from 0 to 750 W. The shield and support shall be a refractory with an opening of 79 mm, with the upper surface beveled to 86 mm to accommodate the specified 500-mL flask. When the flask is placed on the refractory, there should be a distance of approximately 3 mm between the bottom of the flask and the heating elements.

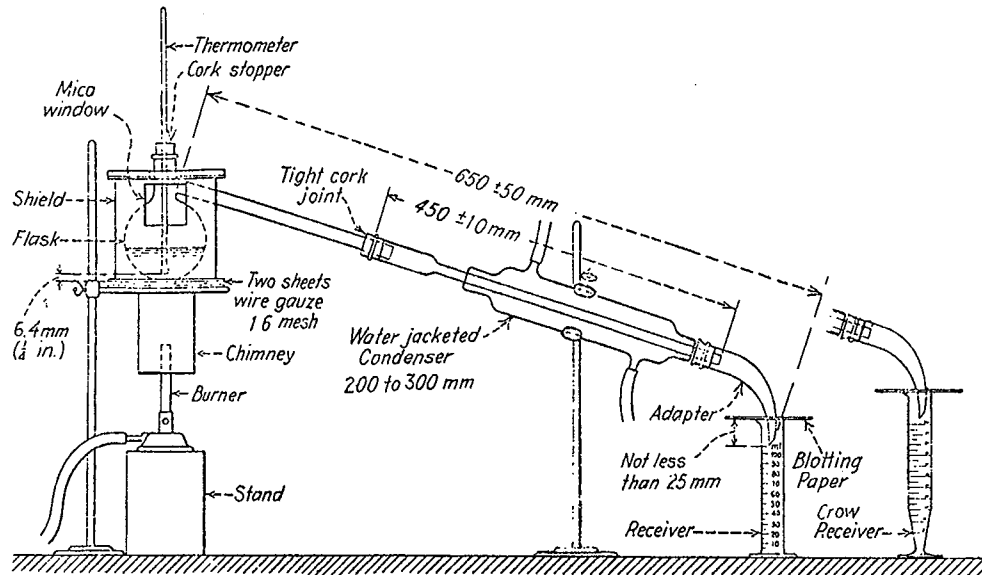


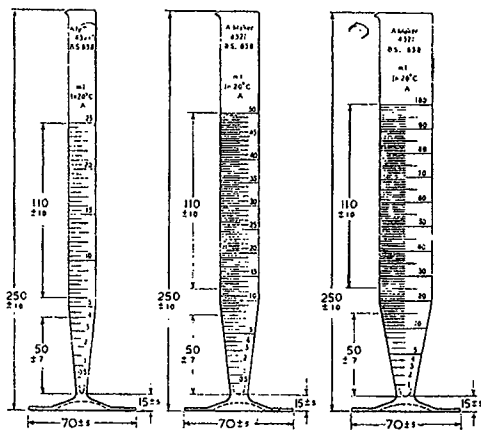
FIG. 3 Distillation Apparatus

5.7 Receiver—A standard 100-mL graduated cylinder conforming to dimensions of Specification E 133, or a 100-mL Crow receiver as shown in Fig. 4 of this test method.

NOTE 1—Receivers of smaller capacity having 0.1-mL divisions may be used when low volumes of total distillate are expected and the added accuracy required.

5.8 Residue Container—A seamless metal container with slip on cover of 75 ± 5 mm in diameter, and 55 ± 5 mm in height.

5.9 Thermometric Device—ASTM High Distillation Thermometers having a range from -6 to 400°C (20 to 760°F) and conforming to the requirements for Thermometers 8C (8F) as prescribed in Specification E 1, or IP Thermometer 6C conforming to IP Specifications for Standard Thermometers, or an equivalent thermometric device that has been calibrated in accordance with Test Method E 220. ASTM 8C Thermometers shall be used for referee testing.



All dimensions are in millimetres

FIG. 4 Crow Receivers of Capacity 25, 50, and 100 mL

6. Hazards

6.1 Warning—Mercury has been designated by the United States Environmental Protection Agency (EPA) and many state agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—<http://www.epa.gov/mercury/faq.htm>—for additional information. Users should be aware that selling mercury, mercury-containing products, or both, into your state may be prohibited by state law.

7. Sampling

7.1 Stir the sample thoroughly, warming if necessary, to ensure homogeneity before removal of a portion for analysis.

7.2 If sufficient water is present to cause foaming or bumping, dehydrate a sample of not less than 250 mL by heating in a distillation flask sufficiently large to prevent foaming over into the side arm. When foaming has ceased, stop the distillation. If any light oil has distilled over, separate and pour this back into the flask when the contents have cooled just sufficiently to prevent loss of volatile oil. Mix the contents of the flask thoroughly before removal for analysis. An alternative procedure is described in Test Method D 370.

8. Preparation of Apparatus

8.1 Calculate the weight of 200 mL of the sample from the specific gravity of the material at $15.6/15.6^\circ\text{C}$. Weigh this amount ± 0.5 g into the 500-mL flask.

8.2 Place the flask in the shield supported by two sheets of gauze on a tripod or ring. Connect the condenser tube to the tubulature of the flask with a tight cork joint. Clamp the condenser so that the axis of the bulb of the flask through the center of its neck is vertical. Adjust the adapter over the end of

the condenser tube so that the distance from the neck of the flask to the outlet of the adapter is 650 ± 50 mm (see Fig. 3).

8.3 Insert the thermometric device through a tightly fitting cork in the neck of the flask so that the bulb of the thermometric device rests on the bottom of the flask. Raise the thermometric device approximately 6 mm from the bottom of the flask using the scale divisions or a reference mark on the thermometric device to estimate the 6 mm distance above the top of the cork.

8.4 Protect the burner by a suitable shield or chimney. Place the receiver so that the adapter extends at least 25 mm but not below the 100-mL mark. Cover the graduate closely with a piece of blotting paper, or similar material, suitably weighted, which has been cut to fit the adapter snugly.

8.5 The flask, condenser tube, adapter, and receiver shall be clean and dry before starting the distillation. Place the seamless residue container on its cover in an area free from drafts.

8.6 Pass cold water through the condenser jacket. Use warm water if necessary to prevent formation of solid condensate in the condenser tube.

9. Procedure

9.1 Correct the temperatures to be observed in the distillation if the elevation of the laboratory at which the distillation is made deviates 150 m or more from sea level. Corrected temperatures for the effect of altitude are shown in Table 1 and Table 2. If the prevailing barometric pressure in millimetres of mercury is known, correct the temperature to be observed with the corrections shown in Table 3. *Do not correct for the emergent stem of the thermometer (if used).*

NOTE 2—Table 3 covers a wide range of temperatures from 160 to 360°C (320 to 680°F) and is to be preferred for world-wide specifications other than ASTM/IP specifications.

9.2 Apply heat so that the first drop of distillate falls from the end of the flask side-arm in 5 to 15 min. Conduct the distillation so as to maintain the following drop rates, the drop count to be made at the tip of the adapter:

TABLE 1 Corrected Distillation Temperatures for Various Altitudes, °C

Elevation above Sea Level, m	Distillation Temperatures for Various Altitudes, °C				
-300	192	227	262	318	362
-150	191	226	261	317	361
0	190	225	260	316	360
150	189	224	259	315	359
300	189	223	258	314	358
450	188	223	257	313	357
600	187	222	257	312	356
750	186	221	256	311	355
900	186	220	255	311	354
1050	185	220	254	310	353
1200	184	219	254	309	352
1350	184	218	253	308	351
1500	183	218	252	307	351
1650	182	217	251	306	350
1800	182	216	250	306	349
1950	181	216	250	305	348
2100	180	215	249	304	347
2250	180	214	248	303	346
2400	179	214	248	303	346

TABLE 2 Corrected Distillation Temperatures for Various Altitudes, °F

Elevation above sea level, m (ft)	Distillation Temperatures for Various Altitudes, °F				
-300	377	440	503	604	684
-150	375	438	502	602	682
0	374	437	500	600	680
150	373	436	499	598	678
300	371	434	497	597	676
450	370	433	495	595	675
600	369	431	494	593	673
750	368	430	493	592	671
900	366	429	491	590	669
1050	365	427	490	589	668
1200	364	426	488	587	666
1350	363	425	487	586	665
1500	362	424	486	584	663
1650	360	422	484	583	661
1800	359	421	483	581	660
1950	358	420	482	580	658
2100	357	419	481	579	657
2250	356	418	479	577	655
2400	355	416	478	576	654

TABLE 3 Factors for Calculating Temperature Corrections

Nominal Temperatures, °C (°F)	Correction ^A per 10 mm Hg Difference in Pressure, °C (°F)
160 (320)	0.514 (0.925)
175 (347)	0.531 (0.957)
190 (374)	0.549 (0.989)
225 (437)	0.591 (1.063)
250 (482)	0.620 (1.116)
260 (500)	0.632 (1.138)
275 (527)	0.650 (1.170)
300 (572)	0.680 (1.223)
315.6 (600)	0.698 (1.257)
325 (617)	0.709 (1.277)
360 (680)	0.751 (1.351)

^A To be subtracted in case the barometric pressure is below 760 mm Hg; to be added in case barometric pressure is above 760 mm Hg.

50 to 70 drops per minute to 260°C (500°F)
 20 to 70 drops per minute between 260 and 316°C (500 and 600°F)
 Not over 10 min to complete distillation from 316 to 360°C (600 to 680°F)

9.2.1 Record the volumes of distillate to the nearest 0.5 mL in the receiver at the corrected temperatures. If the volume of distillate recovered is critical, use receivers graduated in 0.1-mL divisions and immersed in a transparent bath maintained at $15.6 \pm 3^\circ\text{C}$.

NOTE 3—Some cutback asphaltic products yield either no distillate or very little distillate over portions of the temperature range to 316°C (600°F). In this case it becomes impractical to maintain the above distillation rates. For such cases the intent of the method shall be met if the rate of rise of temperature exceeds 5°C (9°F)/min.

9.3 When the temperature reaches the corrected temperature of 360°C (680°F), turn off the heat and remove the flask containing the thermometric device. With the flask in a pouring position, remove the thermometric device and immediately pour the contents into the residue container. The total time from turning off the heat to starting the pour shall not exceed 60 s. When pouring, the side-arm should be substantially horizontal to prevent condensate in the side-arm from being returned to the residue.

NOTE 4—The formation of skin on the surface of a residue during


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cooling entraps vapors which will condense and cause higher penetration results when they are stirred back into the sample. If skin begins to form during cooling, it should be gently pushed aside. This can be done with a spatula with a minimum of disturbance to the sample.

9.4 Allow the condenser and any distillates trapped in the condenser neck to drain into the receiver and record the total volume of distillate collected as total distillate to 360°C (680°F).

9.5 When the residue has cooled until fuming just ceases, stir thoroughly and then, when the material reaches $135 \pm 5^\circ\text{C}$ ($275 \pm 9^\circ\text{F}$), pour into the receptacles for testing for properties such as penetration, viscosity, or softening point. Proceed as required by the appropriate ASTM or IP method from the point that follows the pouring stage.

9.6 If desired, the distillate, or the combined distillates from several tests, may be submitted to a further distillation, in accordance with Test Method D 86 – IP 123, or, when the distillate is of coal-tar origin, Method C.O.3.

10. Calculation and Report

10.1 *Asphaltic Residue*—Calculate the percent residue to the nearest 0.1 as follows:

$$R = [(200 - TD)/200] \times 100 \quad (1)$$

where:

R = residue content, in volume percent, and
 TD = total distillate recovered to 360°C (680°F), mL.

10.1.1 Report as the residue from distillation to 360 (680°F), percent volume by difference.

10.2 *Total Distillate*— Calculate the percent total distillate to the nearest 0.1 as follows:

$$TD \% = (TD/200) \times 100 \quad (2)$$

10.2.1 Report as the total distillate to 360°C (680°F), volume percent.

10.3 Distillate Fractions:

10.3.1 Determine the percentages by volume of the original sample by dividing the observed volume (in millilitres) of the fraction by 2. Report to the nearest 0.1 as volume percent as

follows:

Up to 190°C (374°F)
 Up to 225°C (437°F)
 Up to 260°C (500°F)
 Up to 316°C (600°F)

10.3.2 Determine the percentages by volume of total distillate by dividing the observed volume in millilitres of the fraction by the millilitres recovered to 360°C (680°F) and multiplying by 100. Report to the nearest 0.1 as the distillate, volume percent of total distillate to 360°C (680°F) as follows:

Up to 190°C (374°F)
 Up to 225°C (437°F)
 Up to 260°C (500°F)
 Up to 316°C (600°F)

10.4 Where penetration, viscosity, or other tests have been carried out, report with reference to this test method as well as to any other method used. *Example*—Penetration (ASTM D5 or IP 49) of residue from ASTM D402/IP 27.

11. Precision and Bias

11.1 The following criteria shall be used for judging the acceptability of results (95 % probability):

11.1.1 *Repeatability*— Duplicate values by the same operator shall not be considered suspect unless the determined percentages differ by more than 1.0 volume % of the original sample.

11.1.2 *Reproducibility*— The values reported by each of two laboratories, shall not be considered suspect unless the reported percentages differ by more than the following:

Distillation Fractions, volume percent of the original sample:		
Up to 175°C (347°F)		3.5
Above 175°C (347°F)		2.0
Residue, Volume percentage by difference from the original sample		2.0

11.2 Criteria for judging variability of test results on the distillation residue have not been determined.

12. Keywords

12.1 cutback asphalt; distillate; residue

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Designation: D1310 – 01 (Reapproved 2007)

Standard Test Method for Flash Point and Fire Point of Liquids by Tag Open-Cup Apparatus¹

This standard is issued under the fixed designation D1310; 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 by Tag Open-Cup Apparatus of the flash point and fire point of liquids having flash points between -18 and 165°C (0 and 325°F) and fire points up to 325°F .

1.2 This test method, when applied to paints and resin solutions that tend to skin over or that are very viscous, gives less reproducible results than when applied to solvents.

NOTE 1—In order to conserve time and sample, the fire point of a material may be determined by the Tag Open-Cup Method by continuing the heating of the specimen to its fire point. Fire points may also be determined by Test Method D92, which should be used for fire points beyond the scope of this test method.

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

1.5 *This standard does not purport to address the safety problems 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.*

¹ This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

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2. Referenced Documents

2.1 ASTM Standards:²

- D92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester
- D850 Test Method for Distillation of Industrial Aromatic Hydrocarbons and Related Materials
- D1015 Test Method for Freezing Points of High-Purity Hydrocarbons
- D1016 Test Method for Purity of Hydrocarbons from Freezing Points
- D1078 Test Method for Distillation Range of Volatile Organic Liquids
- D1364 Test Method for Water in Volatile Solvents (Karl Fischer Reagent Titration Method)
- D2268 Test Method for Analysis of High-Purity *n*-Heptane and Isooctane by Capillary Gas Chromatography
- D2699 Test Method for Research Octane Number of Spark-Ignition Engine Fuel
- D2700 Test Method for Motor Octane Number of Spark-Ignition Engine Fuel
- E1 Specification for ASTM Liquid-in-Glass Thermometers

3. Terminology

3.1 Definitions:

3.1.1 *flash point, n*—the lowest temperature, corrected to a pressure of 760 mm Hg (101.3 kPa, 1013 mbar), at which application of an ignition source causes the vapor of the specimen to ignite by the procedure described.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *fire point, n*—the lowest temperature at which a specimen sustains burning for a minimum of 5 s by the procedure described.

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.

4. Summary of Method

4.1 The specimen is placed in the cup of a Tag Open-Cup Apparatus and heated at a slow, but constant rate. A small test flame is passed at a uniform rate across the cup at specified intervals until a flash occurs. To determine the fire point, the test is continued until the application of the test flame causes the specimen to ignite and burn for at least 5 s.

5. Significance and Use

5.1 Flash point and fire point of a liquid are physical properties that may be used to define their flammability hazards. The flash point may be used to classify materials in government regulations.

6. Apparatus

6.1 *Flash Tester*—Tag Open-Cup Apparatus (Fig. 1), as described in detail in Annex A1.

6.2 *Shield*, as described in detail in Annex A1.

6.3 *Thermometers*, conforming to Specification E1, as listed in Table 1.

6.4 *Flasks*, 500-mL, two, with rubber stoppers.

7. Materials

7.1 *Water-Glycol Solution* (1 + 1), for flash points from -18 to 93°C (0 to 200°F).

7.2 *Solid Carbon Dioxide-Acetone or other Coolant*.

7.3 *Silicone Fluid*, inert, high boiling, having a flash point exceeding the test temperatures by at least 60°C (110°F) for flash and fire points from 93 to 165°C (200 to 325°F).

7.4 *n-Heptane*,³ for determination of flash points from -18 to 16°C (0 to 60°F). See Annex A2 for specifications.

7.5 *p-Xylene*,³ for determination of flash points from 16 to 93°C (60 to 200°F). See Annex A2 for specifications.

7.6 *Isopropanol (isopropyl alcohol)*,⁴ for determination of flash points from 16 to 93°C (60 to 200°F). See Annex A2 for specifications.

7.7 *Diethylene Glycol*,⁵ for determination of flash points from 93 to 165°C (200 to 325°F). See Annex A2 for specifications.

8. Assembly and Preparation of Apparatus

8.1 Place the tester in a level position on a solid table free of vibration, in a location free of perceptible draft, and in a dim light. Maintain a room temperature of $24 \pm 3^{\circ}\text{C}$ ($75 \pm 5^{\circ}\text{F}$) throughout the test. Other room temperatures may be specified on agreement between buyer and seller.

NOTE 2—For materials with vapors or products of pyrolysis that are objectionable, it is permissible to place the apparatus with shield in a fume hood with the ventilation turned off. The ventilation can then be turned on at completion of the test, or when and if fumes become objectionable.

³ Satisfactory *n*-heptane and *p*-xylene can be obtained from Special Products Division, Chemical Department, Phillips Petroleum Company, Drawer O, Borger, TX 79607.

⁴ Satisfactory isopropanol may be obtained from Exxon Chemical, Americus P. O. Box 3272, Houston, TX 77001, Shell Chemical Co., One Shell Plaza, Houston, TX 77002, or Union Carbide Co., P. O. Box 8361, South Charleston, WV 25303.

⁵ Satisfactory diethylene glycol may be obtained from Union Carbide Co., S. Charleston, WV.

8.2 Adjust the horizontal and vertical positions of the taper so that the jet passes on the circumference of a circle having a radius of at least 6 in. (150 mm). The jet should pass across the center of the cup at right angles to a diameter passing through the thermometer and in a plane $\frac{1}{8}$ in. (3.2 mm) above the upper edge of the cup as measured from the center of the orifice.

8.3 Using the leveling device as a gage, adjust the height of the taper so that the center of the orifice is exactly $\frac{1}{8}$ in. (3.2 mm) above the top edge of the glass cup when it is in place. It is imperative that this adjustment be made as accurately as possible. Raising or lowering the taper can be achieved by bending it slightly or preferably by adding and removing thin metal shims as required from between the taper and the vertical supporting member of the swivel holder.

8.4 With the glass cup in place in the bath, adjust the thermometer holder so that the thermometer is supported firmly in a vertical position halfway between the center and edge of the cup and on a line passing through the center of the cup and the pivot of the taper. Place the thermometer so that the bottom of the bulb is $\frac{1}{4}$ in. (6.4 mm) from the inner bottom of the cup.

8.5 Set the draft shield around the tester so that the sides form right angles with each other and the tester is well toward the back of the shield.

9. Procedure

9.1 *Flash Points from -18 to 16°C (0 to 60°F)* (**Warning**—Meticulous attention to all details relating to the taper, size of taper flame, rate of temperature increase, and rate of passing the taper over the sample is necessary for good results.)

9.1.1 Equip two 500-mL flasks with rubber stoppers through which are inserted ASTM 33C (33F) thermometers. Cool a quantity of 1 + 1 water-glycol solution in one stoppered 500-mL flask to approximately -30°C (-20°F) by immersing the flask in a solid carbon dioxide-acetone bath or other coolant. Use extreme care not to contaminate the water-glycol solution with either acetone or carbon dioxide.

9.1.2 Pour the cooled water-glycol solution into the tester bath to a predetermined level 3.2 mm ($\frac{1}{8}$ in.) below the top when the cup is in place. An overflow is desirable for controlling the liquid level in the bath.

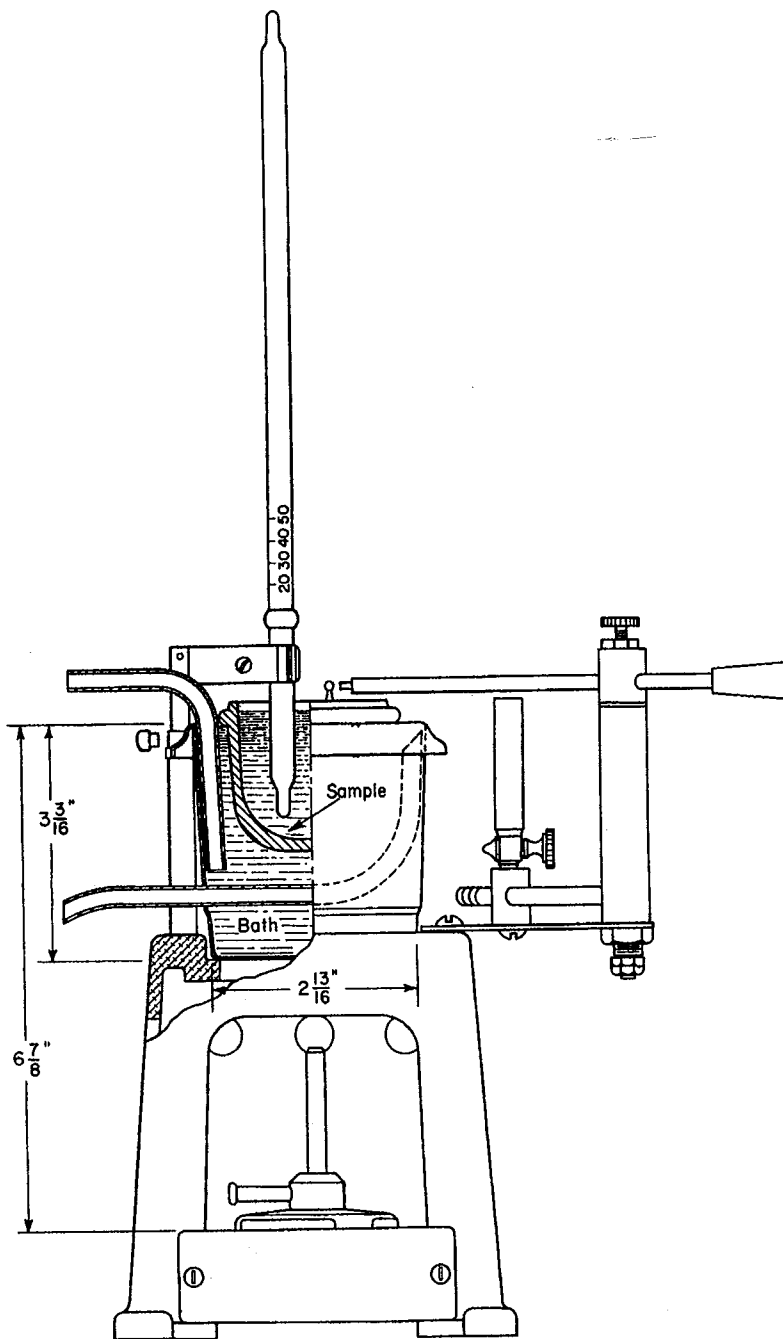
9.1.3 At the same time the water-glycol coolant is being chilled, cool a portion of the sample to approximately -25°C (-10°F) in the second stoppered 500-mL flask. If solid carbon dioxide and acetone or other volatile solvents are used as a coolant, extreme care must be exercised to avoid contamination of the sample. Cool the glass cup and place it in the bath. Position the appropriate thermometer (Table 1) as described in 8.4 and fill the cup with cooled sample to a depth approximately 3.2 mm ($\frac{1}{8}$ in.) below the edge as determined by the leveling device.

NOTE 3—Remove all bubbles from the surface of the liquid before starting a determination.

9.1.4 Light the ignition flame and adjust it to form a flame of spherical shape matching in size the $\frac{5}{32}$ -in. (4.0-mm) sphere on the apparatus or the $\frac{5}{32}$ -in. hole in the leveling device.

9.1.5 Make the final adjustment of the specimen level in the cup when the temperature is 20°F (10°C) below the anticipated flash point. Two trial determinations may be necessary to select

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Metric Equivalents

in.	mm
2 ¹³ / ₁₆	71.4
3 ³ / ₁₆	81
6 ⁷ / ₈	174.5

FIG. 1 Tag Open-Cup Apparatus

TABLE 1 Thermometers

	ASTM Thermometer Number	Thermometer Range	Thermometer Subdivisions
<i>Flash Point/ Fire Point:</i>			
0 to 60°F	33F-75	-36.5 to + 107.5°F	0.5°F
-18 to 15°C	33C-75	-38 to + 42°C	0.2°C
60 to 200°F	9F-75	20 to 230°F	1°F
15 to 93°C	9C-75	-5 to 110°C	0.5°C
200 to 325°F	35F-79	194 to 338°F	0.5°F
93 to 165°C	35C-79	90 to 170°C	0.2°C

the proper temperature at which to adjust the liquid level. A hypodermic syringe or medicine dropper provides a convenient means of adding or removing sample from the cup.

9.1.6 Allow the temperature of the specimen to increase spontaneously—without applying any heat—until the rate of temperature rise decreases to 2°F (1°C)/min. At this point, apply heat to maintain an increase in temperature at a rate of $2 \pm 0.5^\circ\text{F}$ ($1 \pm 0.25^\circ\text{C}$)/min.

NOTE 4—With viscous materials, this rate of heating cannot always be maintained.

9.1.7 Determine the approximate flash point by passing the taper flame across the specimen at intervals of 2°F (1°C). Make the first pass of the taper flame immediately after the final adjustment of the specimen level, as in 9.1.5. The time required to pass the ignition flame across the surface of the liquid should be 1 s. Each pass must be in one direction only, and the taper should be kept in the “off” position at one or the other end of the swing except when the flame is applied to the specimen. In case the material tends to “creep” over the edge of the cup, carefully wipe the edge with absorbant tissue to remove frost and liquid, just prior to passage of the taper over the cup.

NOTE 5—When determining the flash point or fire point, or both, of viscous liquids and those liquids that tend to form a surface film, the following procedure is suggested: About 15 s before the taper is passed over the surface, insert the end of a stirring rod to a depth of about ½ in. (15 mm) in approximately a vertical position. Move the rod from side-to-side of the cup for three or four complete passes following approximately the path of the taper, remove, and make the test.

NOTE 6—Discontinue heating and checking flash point if the specimen boils before flashing. Record that the material has no flash point prior to boiling.

9.1.8 Continue with procedure in 9.4.

9.2 *Flash Points from 60 to 200°F* (16 to 93°C) (See 9.1 and Notes 3-5):

9.2.1 Fill the bath with cold water or water-glycol solution to a predetermined level ⅛ in. (3.2 mm) below the top when the cup is in place. The bath liquid should be at least 30°F (17°C) below the anticipated flash point.

9.2.2 If necessary, cool a portion of the sample to at least 20°F (10°C) below the anticipated flash point. Exercise adequate care to avoid contamination of the sample with coolant liquid or vapors. Fill the glass cup with the cooled sample to a depth approximately ⅛ in. (3.2 mm) below the edge as determined by the leveling device, with the proper thermometer (see Table 1) positioned as described in 8.4.

9.2.3 For final adjustment of the specimen level, see 9.1.5.

9.2.4 Light the ignition flame and adjust it as described in 9.1.4.

9.2.5 Apply heat to the liquid bath and adjust so that the temperature of the specimen increases at a rate of $2 \pm 0.5^\circ\text{F}$ ($1 \pm 0.25^\circ\text{C}$)/min.

9.2.6 Determine the approximate flash point by passing the taper flame across the specimen at intervals of 2°F (1°C) as described in 9.1.7.

9.2.7 Continue with procedure in 9.4.

9.3 *Procedure for Flash Points from 200 to 325°F* (93 to 165°C) (See 9.1 and Notes 3-5):

9.3.1 Fill the bath with a high-boiling inert silicone fluid to a predetermined level ⅛ in. (3.2 mm) below the top when the cup is in place.

9.3.2 With the appropriate thermometer (see Table 1) properly positioned (8.4), fill the glass cup with sample at room temperature to a depth slightly more than ⅛ in. (3.2 mm) below the edge as determined by the leveling device.

9.3.3 For final adjustment of the specimen level, see 9.1.5.

9.3.4 Light the ignition flame and adjust it as described in 9.1.4.

9.3.5 Apply full heat to the liquid bath; when the temperature of the specimen reaches approximately 190°F (90°C), adjust the heat input so that the temperature of the specimen increases at a rate of $2 \pm 0.5^\circ\text{F}$ ($1 \pm 0.25^\circ\text{C}$)/min.

NOTE 7—The heaters on some testers do not have sufficient capacity to maintain the proper rate of heating when the temperature approaches 250°F (120°C) or above. The heat input to the liquid bath may be increased if necessary by using a variable transformer to increase the voltage to the heater or by wrapping the bath with electrical heating tape. The application of suitable insulation to the outside of the bath to prevent heat loss is also permissible. The important factor is to maintain the rate of temperature increase of the specimen at $2 \pm 0.5^\circ\text{F}$ ($1 \pm 0.25^\circ\text{C}$)/min.

9.3.6 Determine the approximate flash point by passing the taper flame across the specimen at intervals of 2°F (1°C) as described in 9.1.7.

9.4 Determine and record not less than three test values, as follows:

9.4.1 After the initial test to determine the approximate flash point of the materials, repeat the procedure by cooling a fresh portion of the sample, the glass cup, the bath solution, and the thermometer to more than 20°F (10°C) below the approximate flash point. When the temperature of the specimen is exactly 20°F below the approximate flash point, adjust the center of the liquid level to ⅛ in. (3.2 mm) below the upper edge of the cup as determined with the leveling device placed across the diameter of the cup.

9.4.2 Resume heating, or allow the temperature to rise spontaneously in the case of materials flashing below 60°F (16°C). Following the instructions given in 9.1.7, pass the taper flame across the specimen at two intervals of 5°F (3°C) and then at intervals of 2°F (1°C) until the flash point is reached.

9.5 *Fire Point (Sustained Burning):*

9.5.1 Except for the initial test, after determining the flash point, continue heating the apparatus so that the temperature of the specimen increases at the rate of $2 \pm 0.5^\circ\text{F}$ ($1 \pm 0.25^\circ\text{C}$)/min. At intervals of 2°F (1°C), pass the taper across the surface of the specimen as described in 9.1.7 and in Note 6 and determine the temperature at which burning is sustained

for 5 s after ignition (fire point). Time the duration of burning from the time the taper (ignition source) has completed its passage across the surface of the liquid.

9.5.2 Determine and record the results obtained from the continuation of three or more flash point tests (9.4).

10. Instrument Verification

NOTE 8—The height of the taper is very important. Raising the taper 0.01 in. (0.25 mm) increases the flash point about 2°F (1°C). Each apparatus should be verified about once a week, if in constant use; or on each occasion of use, when used only occasionally.

10.1 *Flash Points from 0 to 60°F* (–18 to 16°C) (see Note 8).

10.1.1 Make a determination of the flash point of *n*-heptane that meets the specifications set forth in Annex A2, corrected for barometric pressure (see Section 11). If the flash point differs from $23 \pm 3^\circ\text{F}$ ($-5 \pm 1.5^\circ\text{C}$), adjust the height of the taper and rerun *n*-heptane until the flash point is within the stated tolerance.

10.2 *Flash Points from 60 to 200°F* (16 to 93°C) (see Note 8).

10.2.1 Make a determination of the flash point of *p*-xylene that meets the specifications set forth in Annex A2, corrected for barometric pressure (see Section 11). If the flash point differs from $92 \pm 3^\circ\text{F}$ ($33 \pm 1.5^\circ\text{C}$), adjust the height of the taper and rerun *p*-xylene until the flash point is within the stated tolerance.

10.3 *Flash Points from 200 to 325°F* (93 to 165°C) (see Note 8).

10.3.1 Make a determination of the flash point diethylene glycol that meets the specifications set forth in Annex A2, corrected for barometric pressure (see Section 11). If the flash point from $295 \pm 10^\circ\text{F}$ ($146 \pm 5^\circ\text{C}$), adjust the height of the taper and rerun diethylene glycol until the flash point is within the stated tolerance.

11. Correction for Barometric Pressure

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

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

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

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

where:

C = observed flash point in °C

F = observed flash point in °F

p = ambient barometric pressure in kPa

P = ambient barometric pressure in mm Hg.

NOTE 9—The barometric pressure used in this calculation is the ambient pressure for the laboratory at the time of the 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.

11.2 Round off the corrected flash point to the nearest 1°F (0.5°C).

12. Report

12.1 Report the mean of not less than three corrected recorded tests, other than the initial test, to the nearest 1°F (0.5°C). Three multiple runs are acceptable for averaging if the difference between the extreme values does not exceed 7°F (4°C) (95 % confidence level).

13. Precision ⁶

13.1 The following criteria should be used for judging the acceptability of results between 0 and 200°F (–18 and 93°C) at the 95 % confidence level.

13.1.1 *Repeatability*—Two results, each the mean of three determinations, obtained by the same operator should be considered suspect if they differ by more than 4°F (2°C).

13.1.2 *Reproducibility*—Two results, each the mean of three determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 7°F (4°C).

13.2 The following criteria should be used for judging flash points between 200 and 325°F (93 and 165°C) at the 95 % confidence level.

13.2.1 *Repeatability*—Two results, each the mean of three determinations, obtained by the same operator should be considered suspect if they differ by more than 9°F (5°C).

13.2.2 *Reproducibility*—Two results, each the mean of three determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 12°F (7°C).

13.3 On the basis of an interlaboratory test of the method, using viscous, heavily-pigmented materials that tended to form a surface film, the within-laboratory standard deviation was found to be 3°F (2°C) and the between-laboratories standard deviation was found to be 7°F (4°C). Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

13.3.1 *Repeatability*—Two results, each the mean of three determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 5°C (9°F).

13.3.2 *Reproducibility*—Two results, each the mean of three determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 13°C (24°F).

13.4 The precision of fire points will be determined. See Test Method D92 for precision using the Cleveland Open Cup.

13.5 *Bias*—No estimate of bias of flash point and fire point tests can be determined, as no absolute values are available. The tests are equipment and method dependent.

14. Keywords

14.1 fire point; flash point; liquids; paints; resin solutions; Tag tester

⁶ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1002.

ANNEXES

(Mandatory Information)

A1. APPARATUS

A1.1 *Tag Open-Cup Apparatus*, shown in Fig. 1. It consists of the following parts, which must conform to the dimensions shown, and have the additional characteristics as noted:

A1.1.1 *Copper Bath*, preferably equipped with a constant-level overflow so placed as to maintain the bath liquid level $\frac{1}{8}$ in. (3.2 mm) below the rim of the glass cup.

A1.1.2 *Thermometer Holder*, supplied with the tester as shown in Fig. 1 to support the thermometer firmly in a vertical position.

NOTE A1.1—The spring clamp holding the thermometer part may be replaced by a set-screw facing out away from the bath as shown in Fig. 1. This may make it easier and safer to extinguish the fire after the fire point has been reached.

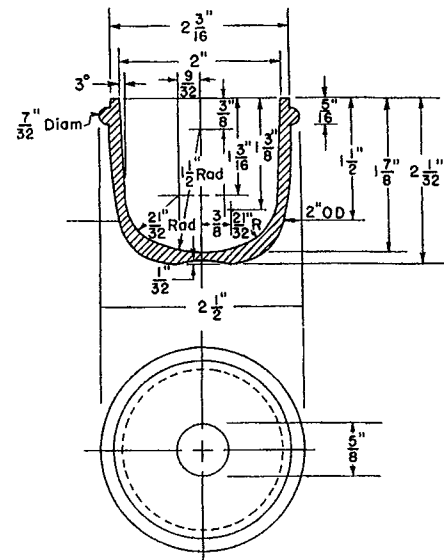
A1.1.3 *Glass Test Cup* (Fig. A1.1), of molded clear glass, annealed, heat-resistant, and free from surface defects.

A1.1.4 *Leveling Device*, or gage, for proper adjustment of the liquid in the cup (Fig. A1.2) made of polished aluminum or stainless steel $\frac{1}{8}$ in. (3.2 mm) thick, with two projections for adjusting the liquid level in the glass cup to $0.125 \text{ in.} \pm 0.003 \text{ in.}$ ($3.18 \pm 0.08 \text{ mm}$) below the top edge or rim of the cup. This leveling device may also be used to adjust the size of the test flame and for gaging the height of the taper above the edge of the cup.

A1.1.5 *“Micro” or Small Gas Burner*, of suitable dimensions for heating the bath. A screw clamp may be used to help regulate the gas. A small electric heater controlled by a variable power transformer may be used.

A1.1.6 *Ignition Taper*, a small, straight blow-pipe type gas burner with the tip approximately $\frac{1}{16}$ in. (1.5 mm) in diameter and the orifice $\frac{1}{32}$ in. (0.8 mm) in diameter. The ignition taper should be maintained in a fixed horizontal plane above the test cup by means of a swivel device so that the test flame passes on the circumference of a circle having a radius of at least 6 in. (150 mm).

A1.1.7 *Draft Shield*, consisting of two rectangular sheets of noncombustible material, 24 by 28 in. (610 by 710 mm),


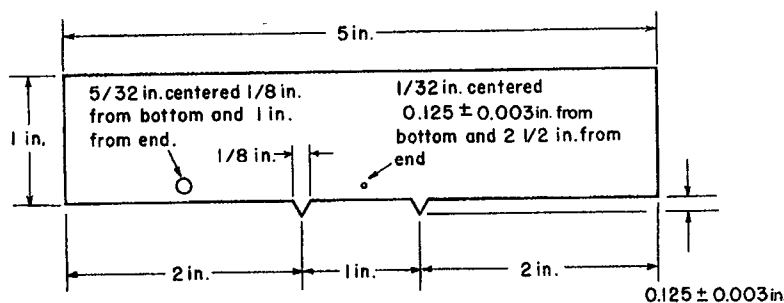


Metric Equivalents

in.	mm	in.	mm
$\frac{1}{32}$	0.8	$1\frac{1}{16}$	30.1
$\frac{7}{32}$	5.5	$1\frac{3}{8}$	34.9
$\frac{9}{32}$	7.0	$1\frac{1}{2}$	38.1
$\frac{5}{16}$	7.5	$1\frac{7}{8}$	47.6
$\frac{3}{8}$	9.5	2	50.8
$\frac{5}{8}$	15.9	$2\frac{1}{32}$	51.6
$2\frac{1}{32}$	16.6	$2\frac{3}{16}$	55.5
		$2\frac{1}{2}$	63.5

FIG. A1.1 Glass Test Cup

fastened together along the 28-in (710-mm) side, preferably by hinges. A triangular sheet, 24 by 24 by 34 in. (610 by 610 by 860 mm), is fastened by hinges to one of the lateral sheets (to form a top when shield is open). The interior of the draft shield shall be painted a flat black. A draft-free fume hood may be used.


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Metric Equivalents

in.	mm
1/32	0.8
1/8	3.2
0.125 ±	3.18 ± 0.08
0.003	
5/32	4.0
1	25.4
2	50.8
2 1/2	63.5
5	127

FIG. A1.2 Leveling Device for Adjusting Liquid Level in Test Cup, Height of Taper above Cup, and Size of Test Flame

A2. SPECIFICATIONS FOR *n*-HEPTANE, *p*-XYLENE, ISOPROPANOL, AND DIETHYLENE GLYCOL

A2.1 Specifications for *n*-Heptane⁵ (ASTM Knock Test Reference Fuel)—*n*-Heptane shall conform to the following requirement:

A2.1.1 Purity—99.75 % when determined by Test Method D2268.

NOTE A2.1—This is the same grade of *n*-heptane specified in Test Methods D2699 and D2700.

A2.2 Specifications for *p*-Xylene (Flash Point Check Grade)⁵—*p*-Xylene shall conform to the following requirements:

A2.2.1 Specific Gravity—15.56/15.56°C—0.863 ± 0.003 max.

A2.2.2 Boiling Range—2°C max from start to dry point, when tested in accordance with Test Method D850 or Test Method D1078. The range shall include the boiling point of pure *p*-xylene, which is 138.35°C (281.03°F).

A2.2.3 Freezing point 11.23°C, min calculated in accordance with Test Method D1016, from the experimentally determined freezing point, measured by Test Method D1015.

A2.3 Specifications for Isopropanol (Isopropyl Alcohol) 91 % (Volume);⁶ Isopropanol shall conform to the following requirements:

A2.3.1 Specific Gravity—0.8180 ± 0.0005 at 20/20°C as determined by means of a calibrated pycnometer.


A2.3.2 Distillation Range—Shall entirely distill within a 1.0°C range that shall include the temperature 80.4°C as determined by Test Method D1078.

A2.4 Specifications for Diethylene Glycol¹¹—Diethylene glycol shall conform to the following requirements:

A2.4.1 Specific Gravity—1.1185 ± 0.0015 at 20/20°C as determined by means of a calibrated pycnometer.

A2.4.2 Distillation Range—Shall entirely distill within a 5.0 range which shall include the temperature 245.8°C as determined by Test Method D1078.

A2.4.3 Water—Not more than 0.2 % as determined by Test Method D1364.

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State of West Virginia

VENDOR PREFERENCE CERTIFICATE

Certification and application* is hereby made for Preference in accordance with West Virginia Code, §5A-3-37. (Does not apply to construction contracts). West Virginia Code, §5A-3-37, provides an opportunity for qualifying vendors to request (at the time of bid) preference for their residency status. Such preference is an evaluation method only and will be applied only to the cost bid in accordance with the West Virginia Code. This certificate for application is to be used to request such preference. The Purchasing Division will make the determination of the Resident Vendor Preference, if applicable.

1. Application is made for 2.5% resident vendor preference for the reason checked:

- Bidder is an individual resident vendor and has resided continuously in West Virginia for four (4) years immediately preceding the date of this certification; or, Bidder is a partnership, association or corporation resident vendor and has maintained its headquarters or principal place of business continuously in West Virginia for four (4) years immediately preceding the date of this certification; or 80% of the ownership interest of Bidder is held by another individual, partnership, association or corporation resident vendor who has maintained its headquarters or principal place of business continuously in West Virginia for four (4) years immediately preceding the date of this certification; or, Bidder is a nonresident vendor which has an affiliate or subsidiary which employs a minimum of one hundred state residents and which has maintained its headquarters or principal place of business within West Virginia continuously for the four (4) years immediately preceding the date of this certification; or,

2. Application is made for 2.5% resident vendor preference for the reason checked:

- Bidder is a resident vendor who certifies that, during the life of the contract, on average at least 75% of the employees working on the project being bid are residents of West Virginia who have resided in the state continuously for the two years immediately preceding submission of this bid; or,

3. Application is made for 2.5% resident vendor preference for the reason checked:

- Bidder is a nonresident vendor employing a minimum of one hundred state residents or is a nonresident vendor with an affiliate or subsidiary which maintains its headquarters or principal place of business within West Virginia employing a minimum of one hundred state residents who certifies that, during the life of the contract, on average at least 75% of the employees or Bidder's affiliate's or subsidiary's employees are residents of West Virginia who have resided in the state continuously for the two years immediately preceding submission of this bid; or,

4. Application is made for 5% resident vendor preference for the reason checked:

- Bidder meets either the requirement of both subdivisions (1) and (2) or subdivision (1) and (3) as stated above; or,

5. Application is made for 3.5% resident vendor preference who is a veteran for the reason checked:

- Bidder is an individual resident vendor who is a veteran of the United States armed forces, the reserves or the National Guard and has resided in West Virginia continuously for the four years immediately preceding the date on which the bid is submitted; or,

6. Application is made for 3.5% resident vendor preference who is a veteran for the reason checked:

- Bidder is a resident vendor who is a veteran of the United States armed forces, the reserves or the National Guard, if, for purposes of producing or distributing the commodities or completing the project which is the subject of the vendor's bid and continuously over the entire term of the project, on average at least seventy-five percent of the vendor's employees are residents of West Virginia who have resided in the state continuously for the two immediately preceding years.

Bidder understands if the Secretary of Revenue determines that a Bidder receiving preference has failed to continue to meet the requirements for such preference, the Secretary may order the Director of Purchasing to: (a) reject the bid; or (b) assess a penalty against such Bidder in an amount not to exceed 5% of the bid amount and that such penalty will be paid to the contracting agency or deducted from any unpaid balance on the contract or purchase order.

By submission of this certificate, Bidder agrees to disclose any reasonably requested information to the Purchasing Division and authorizes the Department of Revenue to disclose to the Director of Purchasing appropriate information verifying that Bidder has paid the required business taxes, provided that such information does not contain the amounts of taxes paid nor any other information deemed by the Tax Commissioner to be confidential.

Under penalty of law for false swearing (West Virginia Code, §61-5-3), Bidder hereby certifies that this certificate is true and accurate in all respects; and that if a contract is issued to Bidder and if anything contained within this certificate changes during the term of the contract, Bidder will notify the Purchasing Division in writing immediately.

Bidder: _____

Signed: _____

Date: _____

Title: _____

*Check any combination of preference consideration(s) indicated above, which you are entitled to receive.

RFQ No. _____

STATE OF WEST VIRGINIA
Purchasing Division

PURCHASING AFFIDAVIT

West Virginia Code §5A-3-10a states: No contract or renewal of any contract may be awarded by the state or any of its political subdivisions to any vendor or prospective vendor when the vendor or prospective vendor or a related party to the vendor or prospective vendor is a debtor and the debt owed is an amount greater than one thousand dollars in the aggregate.

DEFINITIONS:

"Debt" means any assessment, premium, penalty, fine, tax or other amount of money owed to the state or any of its political subdivisions because of a judgment, fine, permit violation, license assessment, defaulted workers' compensation premium, penalty or other assessment presently delinquent or due and required to be paid to the state or any of its political subdivisions, including any interest or additional penalties accrued thereon.

"Debtor" means any individual, corporation, partnership, association, limited liability company or any other form or business association owing a debt to the state or any of its political subdivisions. "Political subdivision" means any county commission; municipality; county board of education; any instrumentality established by a county or municipality; any separate corporation or instrumentality established by one or more counties or municipalities, as permitted by law; or any public body charged by law with the performance of a government function or whose jurisdiction is coextensive with one or more counties or municipalities. "Related party" means a party, whether an individual, corporation, partnership, association, limited liability company or any other form or business association or other entity whatsoever, related to any vendor by blood, marriage, ownership or contract through which the party has a relationship of ownership or other interest with the vendor so that the party will actually or by effect receive or control a portion of the benefit, profit or other consideration from performance of a vendor contract with the party receiving an amount that meets or exceed five percent of the total contract amount.

EXCEPTION: The prohibition of this section does not apply where a vendor has contested any tax administered pursuant to chapter eleven of this code, workers' compensation premium, permit fee or environmental fee or assessment and the matter has not become final or where the vendor has entered into a payment plan or agreement and the vendor is not in default of any of the provisions of such plan or agreement.

Under penalty of law for false swearing (*West Virginia Code §61-5-3*), it is hereby certified that the vendor affirms and acknowledges the information in this affidavit and is in compliance with the requirements as stated.

WITNESS THE FOLLOWING SIGNATURE

Vendor's Name: _____

Authorized Signature: _____ Date: _____

State of _____

County of _____, to-wit:

Taken, subscribed, and sworn to before me this ___ day of _____, 20__.

My Commission expires _____, 20__.

AFFIX SEAL HERE

NOTARY PUBLIC _____

Please use this document for technical questions, posing in question format.

Technical Questions for RFQ: _____

Vendor Name: _____

Questions:

2.0 Scope of Work

2.1 Technical Requirements

2.3 Delivery

3.0 Invoicing

4.0 Award

Other:
