Designation: D5236 – 17

Standard Test Method for
Distillation of Heavy Hydrocarbon Mixtures (Vacuum Potstill Method)[1]

This standard is issued under the fixed designation D5236; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method covers the procedure for distillation of heavy hydrocarbon mixtures having initial boiling points greater than 150 °C (300 °F), such as heavy crude oils, petroleum distillates, residues, and synthetic mixtures. It employs a potstill with a low pressure drop entrainment separator operated under total takeoff conditions. Distillation conditions and equipment performance criteria are specified and typical apparatus is illustrated.

1.2 This test method details the procedures for the production of distillate fractions of standardized quality in the gas oil and lubricating oil range as well as the production of standard residue. In addition, it provides for the determination of standard distillation curves to the highest atmospheric equivalent temperature possible by conventional distillation.

1.3 The maximum achievable atmospheric equivalent temperature (AET) is dependent upon the heat tolerance of the charge. For most samples, a temperature up to 565 °C (1050 °F) can be attained. This maximum will be significantly lower for heat sensitive samples (for example, heavy residues) and might be somewhat higher for nonheat sensitive samples.

1.4 The recommended distillation method for crude oils up to cutpoint 400 °C (752 °F) AET is Test Method D2892. This test method can be used for heavy crude oils with initial boiling points greater than 150 °C (302 °F). However, distillation curves and fraction qualities obtained by these methods are not comparable.

1.5 This test method contains the following annexes:

1.5.1 *Annex A1*—Test Method for Determination of Temperature Response Time,

1.5.2 *Annex A2*—Practice for Calibration of Sensors,

1.5.3 *Annex A3*—Test Method for Dehydration of a Wet Sample of Oil,

1.5.4 *Annex A4*—Practice for Conversion of Observed Vapor Temperature to Atmospheric Equivalent Temperature (AET), and

1.5.5 *Annex A5*—Test Method for Determination of Wettage.

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

1.7 *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 warnings, see 6.5.4.2, 6.5.6.3, 6.9.3, 9.5, 9.7, and A2.3.1.3.

1.8 **WARNING**—Mercury has been designated by many regulatory 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 and/or mercury containing products into your state or country may be prohibited by law.

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.08 on Volatility.

Current edition approved May 1, 2017. Published May 2017. Originally approved in 1992. Last previous edition approved in 2013 as D5236 – 13. DOI: 10.1520/D5236-17.

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

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

D941 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Lipkin Bicapillary Pycnometer (Withdrawn 1993)[3]
D1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer
D1250 Guide for Use of the Petroleum Measurement Tables
D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
D1480 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer
D2892 Test Method for Distillation of Crude Petroleum (15-Theoretical Plate Column)
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D5002 Test Method for Density and Relative Density of Crude Oils by Digital Density Analyzer

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *boil-up rate, n*—the quantity of vapor entering the distillation head per unit time.

3.1.1.1 *Discussion*—It is approximately equal to the takeoff rate, differing only by the parasitic heat losses. It is expressed in millilitres per hour for a head of any given internal diameter or millilitres per hour per square centimetre of cross-sectional area of the throat for comparative purposes.

3.1.2 *condenser, n*—the apparatus connected to the outlet of the distillation head in which condensation of the product occurs.

3.1.3 *distillation flask, n*—the flask, of glass or metal, in which the charge is boiled.

3.1.3.1 *Discussion*—The flask is sometimes called a kettle or pot.

3.1.4 *distillation head, n*—the section immediately above the distillation flask containing the entrainment separator.

3.1.5 *distillation pressure (or operating pressure), n*—the pressure measured in the distillation head just before the outlet to the recovery system.

3.1.6 *distillation temperature (or vapor temperature), n*—the temperature of the vapors in the distillation head at the point of measurement.

3.1.7 *loading, n*—the volume of charge relative to the cross-sectional area of the neck.

3.1.8 *pressure drop, n*—the difference between the operating pressure and the pressure measured in the distillation flask.

3.1.8.1 *Discussion*—It is a result of the friction developed by driving the vapors through the system expressed in kilopascals (mm Hg).

3.1.9 *spillover point, n*—the lowest point in the head above the entrainment separator over which the vapors can flow to the condensing region.

3.1.10 *static hold-up (or wettage), n*—the amount of liquid material remaining on the inside of the walls of the apparatus after the distillation has been completed.

3.1.10.1 *Discussion*—In this test method, it includes wettage of the distillation flask in the case of the steel flasks, but not in the case of glass flasks that are removed for weighing after the distillation is completed.

3.1.11 *takeoff rate, n*—the quantity of product removed per unit time.

3.1.11.1 *Discussion*—It is approximately equal to the boil-up rate differing only by parasitic heat losses.

4. Summary of Test Method

4.1 A weighed volume of sample is distilled at absolute pressures between 6.6 kPa and 0.013 kPa (50 mm Hg and 0.1 mm Hg) at specified distillation rates. Cuts are taken at preselected temperatures. Records of vapor temperature, operating pressure, and other variables are made at intervals, including at each cutpoint.

4.2 The mass of each fraction is obtained. Distillation yields by mass are calculated from the mass of each fraction relative to the total mass recovery.

4.3 The density of each fraction is obtained. Distillation yields by volume are calculated from the volume computed for each fraction at 15 °C (59 °F) relative to the total recovery.

4.4 Distillation curves of temperature versus mass or volume percent, or both, are drawn using the data from 4.2 and 4.3.

5. Significance and Use

5.1 This test method is one of a number of tests conducted on heavy hydrocarbon mixtures to characterize these materials for a refiner or a purchaser. It provides an estimate of the yields of fractions of various boiling ranges.

5.2 The fractions made by this test method can be used alone or in combination with other fractions to produce samples for analytical studies and quality evaluations.

5.3 Residues to be used in the manufacture of asphalt can also be made but may not always be suitable. The long heat soaking that occurs in this test method may alter some of the properties.

NOTE 1—While the practice of reblending distillates with residue can be done to produce a lighter residue, it is not recommended because it produces blends with irregular properties.

5.4 Details of cutpoints must be mutually agreed upon before the test begins.

5.5 This is a complex procedure involving many interacting variables. It is most important that at the time of first use of a new apparatus, its components be checked as detailed in Annex A1 and Annex A2 and that the location of the vapor temperature sensor be verified as detailed in 6.5.3 and Fig. 1.

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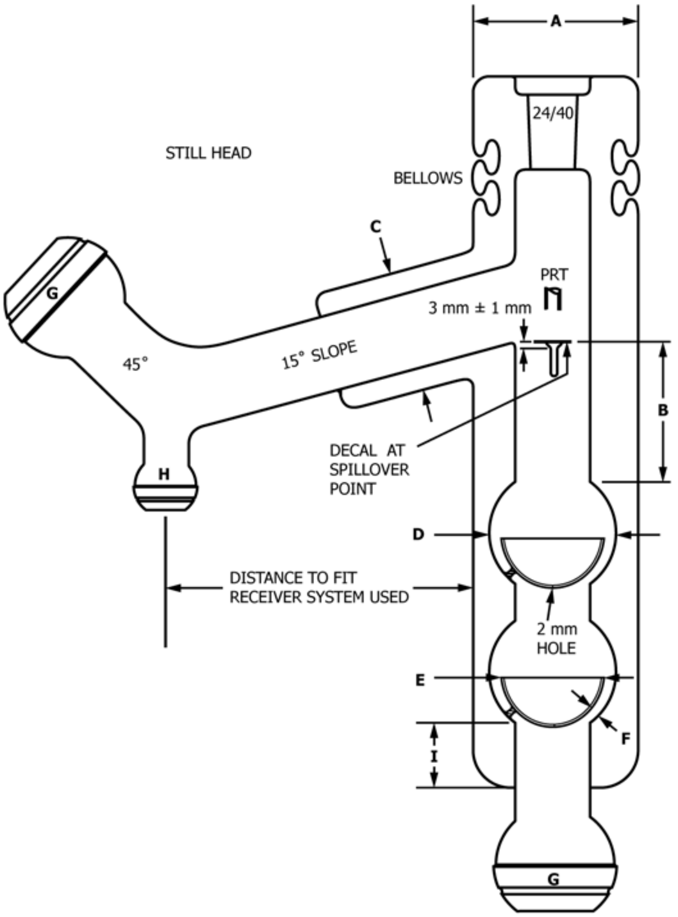
[3] The last approved version of this historical standard is referenced on www.astm.org.



STILL HEAD DIMENSION CHART

Size	A	B	C	D	E	F	G	H	I
25 mm	85 mm	75 mm	64 mm	47 mm ID	40 mm OD	4–5 mm	35/25	28/15	35 mm
36 mm	90 mm	75 mm	64 mm	68 mm ID	57 mm OD	5–6 mm	65/40	35/25	35 mm
50 mm	110 mm	100 mm	75 mm	94 mm ID	79 mm OD	7–9 mm	75/50	35/25	45 mm
70 mm	140 mm	100 mm	100 mm	131 mm ID	111 mm OD	10–11 mm	102/75	50/30	70 mm

FIG. 1 Distillation Head

6. Apparatus

6.1 Four sizes of apparatus, based upon the internal diameter of the distillation head (25 mm, 36 mm, 50 mm, and 70 mm), are allowed.[4] The apparatus (see Fig. 2) consists of a flask with heating mantles, an upper compensator, and a head containing an entrainment separator. Attached to the head are the vapor temperature sensor, a connection for the vacuum gauge, a condenser, a rundown line, a product receiver(s), and a vacuum pumping line with pump. The parts are connected by vacuum-tight joints to facilitate servicing.

6.2 *Distillation Flask:*

6.2.1 The sizes specified for flasks are at least 50 % larger than the size of the charge to provide space for suppression of foam and for bubble breaking. The size of the charge for each size of still is determined from the loading factor. The recommended loading factor is between 200 mL and 400 mL of charge per square centimetre of cross-sectional area in the neck of the head. Table 1 shows the range of charge volume that is recommended with each size of apparatus.

6.2.2 Flasks are made of borosilicate glass except those larger than 10 L, which are made of stainless steel for reasons of safety.

6.2.3 The flask is fitted with a thermowell reaching to within 6 mm of the bottom and offset from the center to avoid a stirring bar. In the case of glass flasks, the bottom shall be slightly flattened or slightly concave, but not perfectly flat to facilitate the rotation of the magnetic stirrer. Steel flasks can have a cooling coil for rapid quenching of the distillation in an emergency. Fig. 3 shows a typical example.

6.3 *Stirring System*—A magnetically driven stirring bar approximately 3 mm diameter and 20 mm long shall be provided for the glass flasks, or 6 mm diameter by 50 mm long for the steel flasks. The edges shall be rounded to minimize grinding the wall of the flask. The external magnetic drive must be capable of rotating the bar in the flask when located directly below and touching the mantle. The drive can be used to

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[4] Cooke, *Industrial and Engineering Chemistry*, Vol 55, 1963, p. 36.

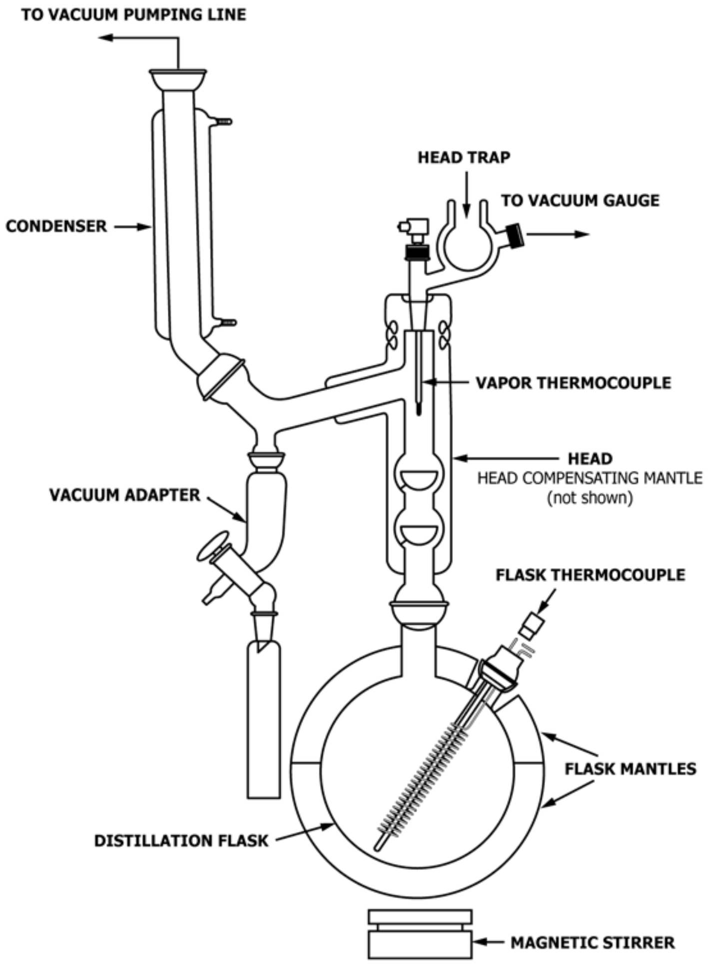


FIG. 2 Apparatus

TABLE 1 Standard Charge and Flask Size

Inside Diameter, mm	Throat Cross-Sectional Area, cm^2	Charge, L	Flask, L
25	5	1–2	2–3
36	10	2–4	3–6
50	20	4–8	6–12
70	40	8–16	12–24

support the apparatus above. An adjustable jacking mechanism is recommended for raising and lowering the stirrer.

6.4 *Heating System:*

6.4.1 The flask shall be heated by means of a nickel-reinforced quartz fabric heating mantle on the lower half so that boiling rates of up to 150 mL/h per cm^2 of the cross-sectional area of the neck can be maintained. A heat density of 0.5 W/cm^2 is adequate. Usually two or more circuits are used to improve heat control by applying automatic heat to the bottom circuit.

6.4.2 A temperature sensor shall be located between the wall of the flask and the mantle for control of the skin temperature.

6.4.3 The upper half of the flask shall be covered with a mantle to compensate for heat losses. A heat density of 0.2 W⁄cm^2 is adequate.

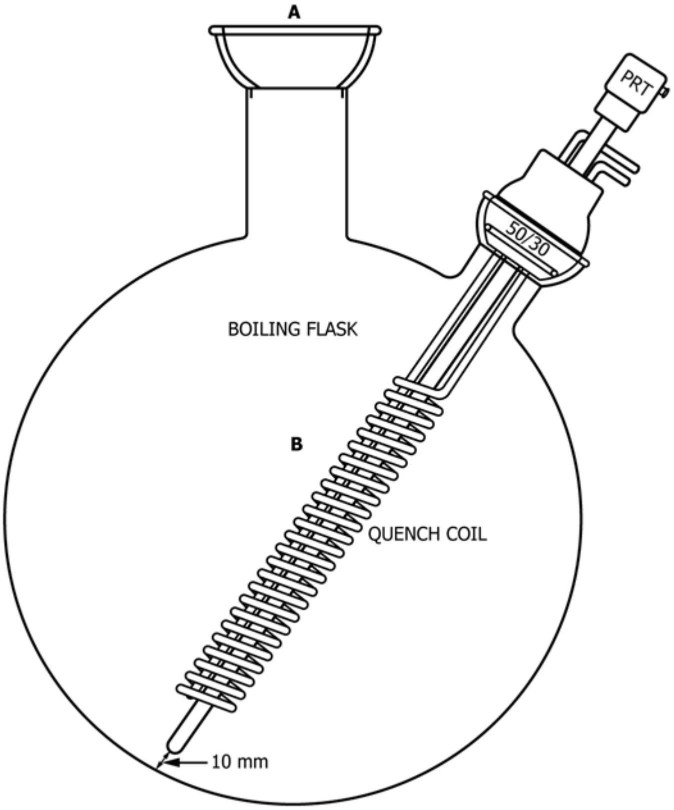
6.5 *Distilling Head:*

6.5.1 The head shall conform to the details shown in Fig. 1. It shall be made of borosilicate glass and be totally enclosed in a silvered glass vacuum jacket having a permanent vacuum of less than 0.0001 kPa (0.00075 mm Hg).

6.5.2 The head shall be enclosed in a heat-insulating system such as a glass fabric mantle capable of maintaining the outer wall of the glass vacuum jacket at a temperature 5 °C below the internal vapor temperature in the head. For this purpose, the vacuum jacket shall have a temperature sensor fastened to the outer wall of the jacket at a point level with the vapor temperature sensor and opposite to the outlet arm of the head.

6.5.3 The head shall be fitted with an adapter to support the vapor temperature sensor so that it is held centered in the neck with the top of the sensing tip 3 mm ± 1 mm below the spillover point. This dimension can be checked by removing the temperature sensor and inserting in its place a copper wire having a short right angle bend at the bottom. By feeling for the spillover point, the distance from the top joint of the adaptor can be found. Laying the wire on the temperature sensor will then permit checking of this dimension.

6.5.4 The vapor temperature sensor shall be either a platinum resistance thermometer, a thermocouple with the junction head fused to the lower tip of the well, or any other device



SYSTEM SIZE	A	B
25 mm	35/25	3 L
36 mm	65/40	6 L
50 mm	75/50	12 L
70 mm	102/75	24 L

FIG. 3 Distillation Flask

which meets the requirements in 6.5.4 and 6.5.4.1. It shall have a response time of less than 60 s as described in Annex A1.

6.5.4.1 The vapor temperature measuring device shall have an accuracy of 0.5 °C or better and be measured with a resolution of 0.1 °C or better.

6.5.4.2 The vapor temperature measuring device shall be calibrated over the full range of useful temperatures in combination with its associated instrument at the time of first use and at least once per year thereafter as described in A2.2.2. Alternatively, certified sensors may be used, provided the calibration of the sensor and its associated recording instrument can be traced back to a primary temperature standard. Recalibrate when either the sensor or the instrument is repaired or serviced. (**Warning—**Vapor temperature measurement is one of the two major sources of error in distillation data.)

6.5.4.3 Verification of the calibration of the vapor temperature measuring devices is to be made on a regular basis. Verification at least once a month is recommended. Verification of the calibration of the sensors can be accomplished potentiometrically by the use of standard precision resistance or by distilling a pure compound with accurately known boiling point, as described in A2.2.3.

6.5.5 A head trap as illustrated in Fig. 4 shall be fitted to the adapter described in 6.5.3 for connection to the vacuum sensor. It shall be kept filled with crushed dry ice at all times while in service.

6.5.6 A vacuum sensor shall be connected to the sidearm of the trap. The sensor shall be capable of reading the pressure with a precision equal to or better than 0.00133 kPa (0.01 mm Hg), whichever is greater. A non-tilting McLeod gauge or other primary reference device can achieve this accuracy when properly used, but a mercury manometer will permit this accuracy only down to a pressure of about 1 kPa and then only when read with a good cathetometer (an instrument based on a telescope mounted on a vernier scale to determine levels very accurately). Also, electronic sensors of the diaphragm type have been found satisfactory. Vacuum gauges based on hot wires, radiation, or conductivity detectors are not recommended.

6.5.6.1 Primary standards, such as the non-tilting McLeod gauge, mercury manometer, or other analogous primary standard pressure devices, can be used without calibration when properly used and maintained. Alternatively, a tensimeter or

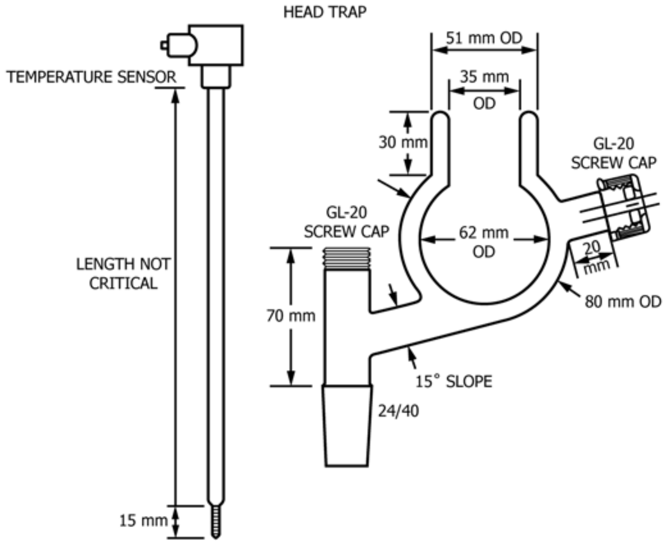
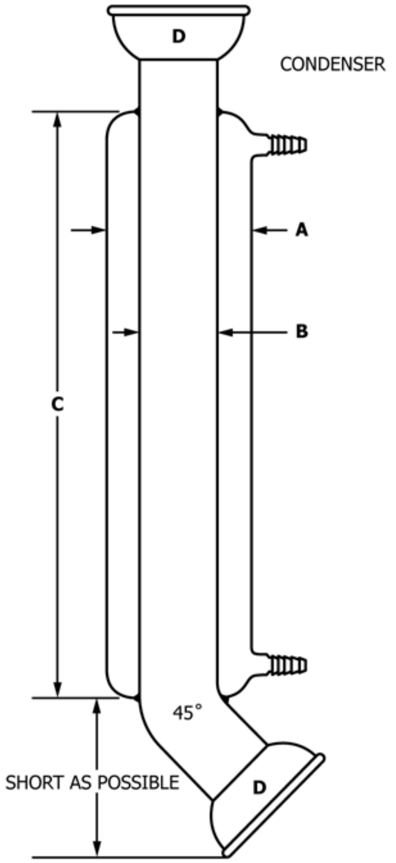


FIG. 4 Head Trap and Temperature Sensor



CONDENSER DIMENSION CHART

System Size	A	B	C	D
25 mm	51 mm	28 mm	300 mm	35/25
36 mm	75 mm	45 mm	300 mm	65/40
50 mm	80 mm	54 mm	400 mm	75/50
70 mm	120 mm	80 mm	400 mm	102/75

FIG. 5 Condenser

certified electronic sensors may be used, provided the calibration of the sensor and its associated recording instrument can be traced back to a primary pressure standard.

6.5.6.2 Noncertified gauges shall be calibrated from a nontilting McLeod gauge, another primary reference device, or a secondary electronic standard traceable to a primary standard. A basic calibration procedure is described in A2.3. Recalibrate when either the sensor or the instrument is repaired or serviced.

6.5.6.3 Verification of the calibration of pressure sensors is to be made on a regular basis. A frequency of at least once a week is recommended. Verification of the calibration of the sensors can be accomplished using the procedures described in A2.3 or against a certified reference system. (**Warning—**Measurement of vacuum (operating pressure) is one of the two major sources of error in the distillation procedure. It is therefore of prime importance that the instructions on calibration and verification be followed with great care and on a routine basis.)

6.6 *Condenser*—A condenser made of borosilicate glass shall be connected to the outlet arm of the head (see Fig. 5). It shall have sufficient capacity to condense essentially all vapors and capable of operating at coolant temperatures up to 70 °C to prevent wax buildup.

6.7 *Pumping Line:*

6.7.1 A pumping line shall be connected from the outlet of the condenser to the vacuum pump. The pumping line can be made of heavy-walled rubber or light metal tubing, but its inside diameter must be greater than half the inside diameter of the outlet of the condenser and less than 2 m long.

6.7.2 A surge tank of a size at least equal to the capacity of the flask shall be inserted in the pumping line adjacent to the pump.

6.7.3 An isolation valve of a diameter at least equal to the diameter of the pumping line shall be connected between the surge tank and the vacuum pump.

6.7.4 A dewar-type trap made of borosilicate glass, such as that illustrated in Fig. 5, shall be placed between the top of the distillation head and the vacuum sensor. It shall be kept filled with crushed dry ice at all times during the distillation to protect the vacuum system from contamination with residual vapors.

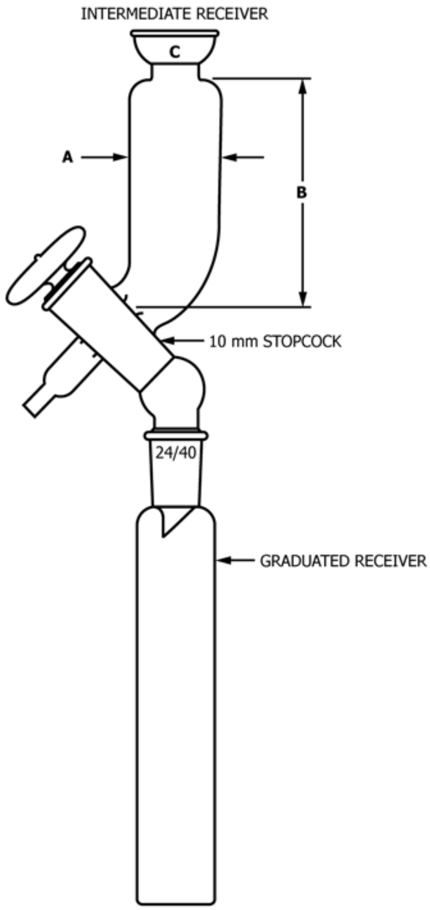
6.8 *Vacuum Source*—A single-stage mechanical vacuum pump capable of maintaining a steady pressure in the system at all operating pressures shall be connected to the pumping line. Automatic or manual control can be used.

6.9 *Recovery System:*

6.9.1 The recovery system is connected to the lower outlet of the product condenser and consists of a vacuum adapter to permit removal of distillate receivers without disturbing the pressure in the system. A suitable manual device is illustrated in Fig. 6.

6.9.2 Alternatively, either automatic or manual devices can be used to collect part or all of the fractions within the system without disturbing the operating pressure until the end of the run. Heating must be provided when needed to maintain the product in the liquid state.

6.9.3 The product receivers shall be made of borosilicate glass and large enough for the size of the fractions to be



INTERMEDIATE RECEIVER DIMENSION CHART

System Size	A	B	C
25 mm	45 mm	120 mm	35/25
36 mm	51 mm	120 mm	35/25
50 mm	64 mm	150 mm	50/30
70 mm	75 mm	150 mm	50/30

FIG. 6 Receiver System

collected. They shall be calibrated to the nearest 1 % from the bottom. (**Warning—**This apparatus operates under high vacuum and high temperature. It is recommended that these stills be kept in an enclosure to ensure that in case of an implosion, the operator and others nearby are protected from flying debris, but that the front, at least, be transparent and removable for access to controls and so forth. Automated stills, which are left unattended for long periods, should be equipped with an automatic fire extinguisher, automatic quench, and alarm.)

7. Sampling

7.1 Obtain the sample for distillation in accordance with instructions given in Practice D4057 or Practice D4177. The sample can also be a residue from Test Method D2892.

7.2 The sample must be in a closed container when received and show no evidence of leakage.

7.3 If the sample looks waxy or has solidified, warm it enough to liquefy it and ensure that it is thoroughly mixed before using.

7.4 If, upon examination, there is evidence of water in the sample, perform a preliminary distillation as described in Annex A3.

8. Preparation of Apparatus

8.1 Clean and dry all glass parts and assemble them with freshly lubricated joints as shown in Fig. 2. In the case of ball joints, use only enough lubricant to produce a thin continuous film. An excess of lubricant can promote leakage. The rings of O-ring joints should be made of Vitron-A,[5] or silicone of equivalent hardness, and be lightly lubricated.

8.2 Tare the receivers to the nearest 0.1 % of the weight of the charge.

8.3 To check for leaks, pump the system down to a pressure of approximately 0.05 kPa (0.4 mm Hg) and isolate it from the vacuum source. If, after 1 min, the rise in pressure is no greater than 0.01 kPa (0.075 mm Hg), the system is acceptable. If the rise in pressure is greater than 0.01 kPa (0.075 mm Hg) in 1 min, the gauge and its connections must be examined and leaks corrected before proceeding.

8.4 Calibrate the temperature and pressure sensors as described in Annex A2.

9. Procedure

9.1 Determine the density of the sample by one of the following test methods: Test Method D941, D1217, D1480, D5002, or D1298. Refer to Guide D1250 to correct densities to 15 °C.

9.2 Insert the stirring bar.

9.3 From Table 1, determine the volume of the charge and calculate the mass to be charged by multiplying its density by the desired volume.

9.4 Weigh this mass of charge into the flask to the nearest 0.1 %. In the case of flasks too large to handle, the flask can be put in place and the charge drawn in from a container (weighed with its transfer line) using a pressure of 90 kPa to 95 kPa in the still. The charge may need to be warmed to facilitate transfer. Its mass can be determined from the difference.

9.5 Attach the flask to the column (in the case of smaller flasks), and put on all the heating mantles. Put the stirring device in place and turn it on. (**Warning—**Ensure that the safety shield is in place.)

9.6 A contiguous cutting scheme may be achieved in one of two ways, while remaining within the scope of the key aspects of this method. It may be achieved by gradually reducing the pressure over the course of the distillation (dynamic) or done stepwise, by slowing (or stopping) the takeoff rate to allow lowering of the operating pressure to achieve the final cut temperature. In each case, this must be done keeping in mind the necessity to avoid starving the distillation (due to a slow takeoff rate), while at the same time avoiding entrainment (by reducing too quickly the pressure applied to the system).

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[5] Vitron A is a registered trademark of DuPont E.I. De Nemours and Co., Wilmington, DE 19898.

TABLE 2 Operating Pressures and Distillation Rates

Operating Pressure, kPa (mm Hg)	Boil-Up Rate, mL/(h·x·cm^2)	Take-Off Rate, mL/h			
		25 mm	36 mm	50 mm	70 mm
6.67 (50)	90–150	450–750	900–1500	1800–3000	3600–6000
1.33 (10)	75–125	375–625	750–1250	1500–2500	3000–5000
0.133 (1)	45–75	225–375	450–750	900–1500	1800–3000
0.0400 (0.3)	30–50	150–250	300–500	600–1000	1200–2000
0.0133 (0.1)	10–20	50–100	100–200	200–400	400–800

9.7 Apply heat to the flask at a rate that will raise the temperature of the charge quickly, but no faster than 300 °C/h (540 °F/h). Do not exceed a skin temperature on the flask of 400 °C (750 °F) or cracking may result on the walls of the flask. (**Warning—**Some hydrocarbon mixtures cannot tolerate 400 °C for any useful length of time. Reducing the skin temperature may be necessary in these cases.)

9.8 Turn on the head compensation mantle and maintain the outer wall of the glass vacuum jacket at a temperature approximately 40 °C below the temperature of the liquid in the flask.

9.9 Reduce the pressure in the system gradually to a suitable starting pressure. Choose from Table 2 the highest pressure that is consistent with the expected initial boiling point as well as the lowest pressure that is consistent with the maximum cutpoint, using Fig. 7 as a guide. A pressure of 0.133 kPa (1.0 mm Hg) has been found satisfactory for starting a material having an initial boiling point of 343 °C (650 °F) AET, such as residues from Test Method D2892 distillations.

NOTE 2—Degassing of the charge is sometimes evident before the actual distillation begins. This appears as bubbling at the surface without generation of condensable vapors.

9.10 When distillation begins, evidenced by vapors entering the neck of the flask, reduce the heat input to a level that will maintain the chosen distillation rate from Table 2 (see Note 2). Adjust the heat compensator on the head to maintain the outer wall of the glass vacuum jacket at a temperature 5 °C below the vapor temperature.

NOTE 3—Although a range of distillation rates is permitted, 80 % of the maximum allowed is recommended.

9.11 In cases in which the observed initial vapor temperature will be 150 °C (302 °F) or lower, it is desirable to refrigerate the first fraction receiver to ensure the retention of light ends. If solid, waxy material appears on the walls, warm the receiver with an infrared heat lamp or hot air gun to liquify the product in the receiver in order to improve the accuracy of the reading. In automatic operation, the receivers must be thermostated at a temperature high enough to ensure that no solidification takes place and low enough to prevent evaporation of light material.

9.12 When using the dynamic method of pressure reduction, calculate a projected final cutpoint using the operating pressure and the differential between the vapor and pot temperature as the operating envelope. Estimating that the difference between the vapor and the pot temperature remain *relatively* constant, determine if the final vapor temperature can be achieved at this pressure while remaining within the recommended limitations of the flask temperature (see 9.18). If the final cutpoint cannot be achieved at the starting pressure, the pressure should be gradually lowered toward an operating pressure that will allow the final cut to be taken. This must be done bearing in mind the associated takeoff rates for the vacuum pressures indicated in Table 2 and the limitations of the pot temperature. The pressure should be lowered enough to allow the takeoff rate to accelerate briefly while the operator is remaining vigilant to avoid entrainment. The distillation rate at the operating pressure should fall within the recommendations stated in Table 2 and should be allowed to stabilize for at least 2 min before arriving at a cutpoint. Experience has shown that reduced crude oil samples typically run well at 0.133 kPa for up to 25 % to 30 % of the charge volume. Subsequent lowering of the operating pressure, as described above, has yielded satisfactory results. Repeat this procedure throughout the remainder of the distillation until an operating pressure has been attained that will allow the final cut temperature to be reached while remaining within the recommended confines of the maximum pot temperature and temperature/time constraints of flask temperature (see 9.18).

9.13 When the receiver is full, or when a cutpoint is reached, isolate the receiver or move to the next one, as the case may be.

9.13.1 In manual operation, isolate the receiver using the vacuum adaptor and vent it to atmospheric pressure before replacing it with another tared receiver. Apply vacuum, and when the new receiver is at approximately system pressure, reconnect it to the system.

9.13.2 In automatic operation, receivers are changed automatically and do not normally need further attention.

9.14 Record the following observations:

9.14.1 Time in hours and minutes,

9.14.2 Volume of distillate in millilitres,

9.14.3 Vapor temperature to nearest 0.5 °C,

9.14.4 Liquid temperature in the flask in °C,

9.14.5 Pressure in the head to nearest 1 %, and

9.14.6 Atmospheric equivalent temperature by calculation as prescribed in Annex A4.

9.15 Proceed to 9.18.

9.16 Alternatively, the stepwise method can be achieved by initializing the distillation and operating at the pressure at which the distillation stabilizes (see 9.9). Continue taking product and making cuts until the final cutpoint is achieved or until the temperature of the boiling liquid reaches approximately 290 °C (554 °F).

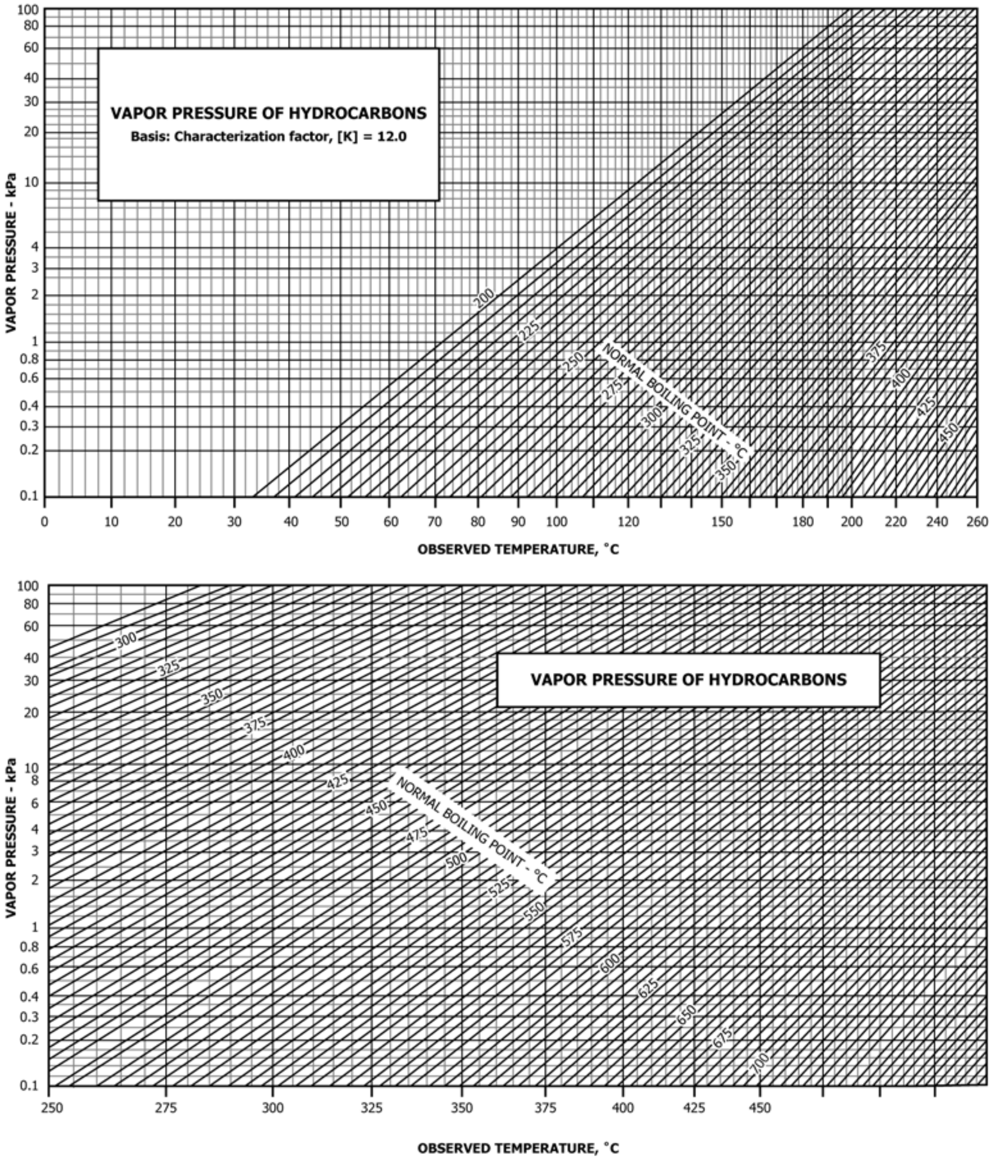


FIG. 7 Correction of Vapor Temperature to Atmospheric Equivalent

9.17 At this point, if the final cutpoint cannot be achieved before reaching 320 °C (608 °F) in the boiling liquid, reduce the heat input to zero until the distillation slows or stops. This will take 2 min to 10 min depending on the amount of material in the flask. Reduce the pressure slowly to a level that will allow for a reasonable amount of overhead product to evolve at the new pressure level. A pressure reduction by a factor of five or six has been shown to be necessary to produce a viable quantity of overhead at the new pressure level.

9.18 Restore the heat to about 90 % of the previous level and then adjust to give the desired rate at the lower level (see Table 2). Do not take any cuts until the pressure has stabilized at the new level for at least 2 min. Repeat 9.16 and 9.17 until a pressure level has been reached that will allow for achieving the final cutpoint before the boiling liquid reaches a temperature of 320 °C (608 °F).

9.19 Continue taking product as long as there is no indication of incipient cracking. Addition of heat to the flask to maintain product rate should be done with great care. It is recommended to achieve the final cutpoint in less than 1 h after the flask temperature has risen above 310 °C (590 °F).

9.20 The distillation shall be discontinued immediately as soon as signs of incipient cracking are observed (see Note 4).

NOTE 4—Cracking will significantly affect the quality of the cuts and the residue, for example, the densities and viscosities would be significantly lower than those obtained without cracking.

NOTE 5—Incipient cracking is usually first observed from a distinct and persistent rise in pressure (for example, >10 % of pressure set point) or an increase of the demand on the vacuum pump capacity. However, automatic vacuum controllers tend to mask these phenomena. Other signs of incipient cracking are the accumulation of thin black deposits on the glassware through the column or the appearance of a smoke-like vapor in the system after the condenser.

9.21 When either the final cutpoint or 90 volume % has been distilled or incipient cracking is observed, discontinue the distillation. Discontinue heat input to the flask and heating jacket at once and slightly raise the pressure of the system by reducing the vacuum pump capacity. Allow the residue to cool while stirring.

NOTE 6—Beyond 90 volume % distilled, the flask may be too near dryness for safe operation.

9.22 Remove the flask compensating mantle, or in the case of steel flasks, turn on the air in the quench coil.

9.23 When the temperature of the residue has fallen below 150 °C (302 °F), remove and weigh the flask and contents to determine the mass of the residue. For larger stills, the residue can be discharged through the charging line using a positive pressure of about 10 kPa in the still.

9.24 Weigh all overhead fractions to within 0.1 % of the charge mass.

9.25 Determine the relative density of all fractions and convert to 15 °C (59 °F) using Guide D1250 where applicable.

9.26 In the case of the smaller stills, recover the wettage by boiling up a small quantity of solvent such as toluene in a separate flask to wash the head and condenser. Evaporate the solvent in a hood assisted by a stream of air and weigh directly. This wettage may be treated as a separate fraction and its density estimated or blended into the residue before inspections are made. The latter must be done if the residue is to be analyzed for other than density. For larger stills, follow instructions given in Annex A5. Note that the holdup in the latter case includes both the overhead wettage and the wettage of the flask with residue and must be considered a separate fraction. Density must be measured in this case.

10. Calculation

10.1 Calculate the weight recovery by adding the masses of all the fractions plus the holdup or wettage. The total must be between 99.6 % and 100.1 % of the weight of the charge to be acceptable. Show the actual loss on the record, and prorate the loss among all fractions.

10.2 Calculate the volume of each fraction by dividing the mass of each fraction by its relative density.

11. Report

11.1 A summary sheet for the run must include the following:

11.1.1 The mass of the charge in grams,

11.1.2 The density of the charge in grams per millilitre at 15 °C to four significant figures,

11.1.3 The volume of the charge in millilitres at 15 °C,

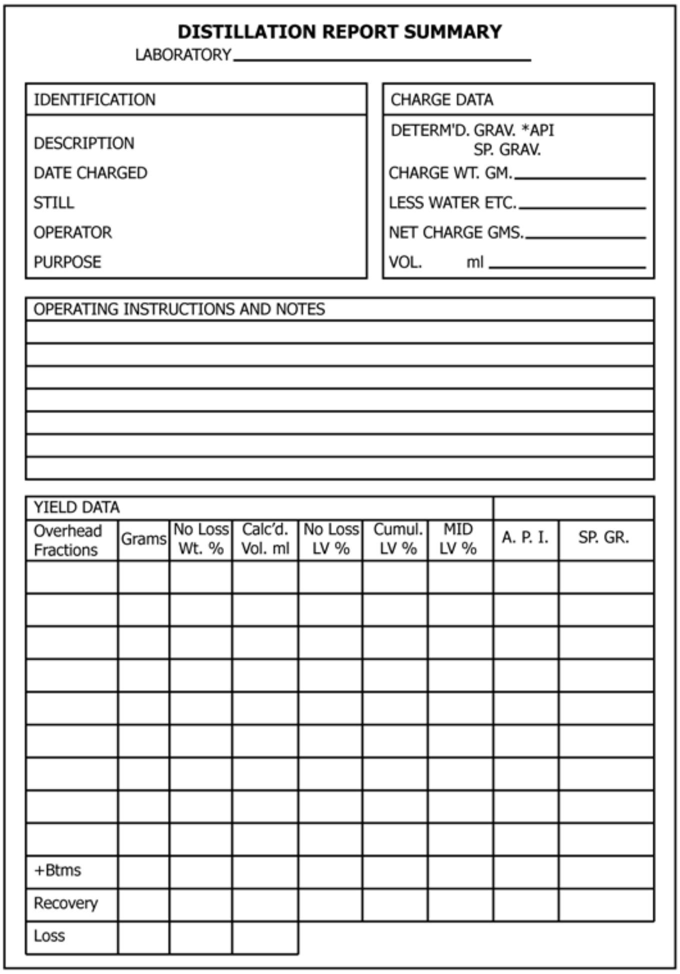
11.1.4 The gain or loss in mass and volume to the nearest 0.1 %,

11.1.5 A listing of the fractions in order of boiling point with the residue recorded last, and

11.1.6 The cumulative mass and volume percentages.

11.2 The observations recorded in 9.14 during the distillation are normally included as a second sheet attached to the summary sheet. Examples of a distillation report and record are illustrated in Fig. 8 and Fig. 9.

11.3 Plot curves of temperature in degrees Celsius AET as ordinates against the percents by mass and volume calculated for the fractions above. A smooth curve through this plot constitutes the final distillation curve.



DISTILLATION REPORT SUMMARY
LABORATORY ____________

IDENTIFICATION	CHARGE DATA
DESCRIPTION	DETERM'D. GRAV. *API SP. GRAV.
DATE CHARGED	CHARGE WT. GM. ________
STILL	LESS WATER ETC. ________
OPERATOR	NET CHARGE GMS. ________
PURPOSE	VOL. ml ________

OPERATING INSTRUCTIONS AND NOTES

YIELD DATA								
Overhead Fractions	Grams	No Loss Wt. %	Calc'd. Vol. ml	No Loss LV %	Cumul. LV %	MID LV %	A. P. I.	SP. GR.
+Btms								
Recovery								
Loss								

FIG. 8 Distillation Report

12. Precision and Bias

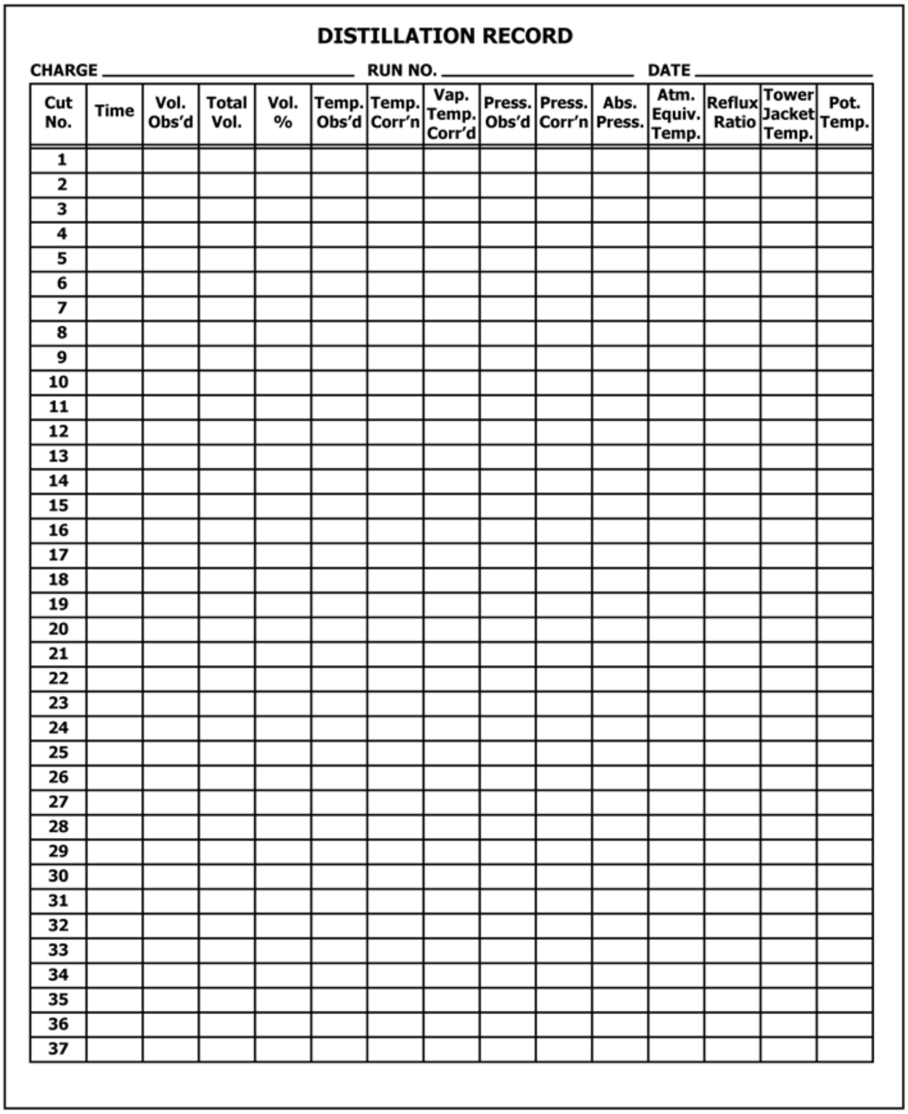
12.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory test results is as follows:[6]

NOTE 7—The following precision data were developed from data obtained from a 1986 cooperative program (six samples, five laboratories), a 1988 cooperative program (three samples, four laboratories), and individual laboratory data on different samples (five samples, three laboratories). Although these data do not meet the statistical requirements of RR:D02-1007, due to the time and cost involved it is unlikely that an additional cooperative program will be initiated soon.

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

---

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1288. Contact ASTM Customer Service at service@astm.org.



DISTILLATION RECORD

CHARGE ________________ RUN NO. ________________ DATE ________________

Cut No.	Time	Vol. Obs'd	Total Vol.	Vol. %	Temp. Obs'd	Temp. Corr'n	Vap. Temp. Corr'd	Press. Obs'd	Press. Corr'n	Abs. Press.	Atm. Equiv. Temp.	Reflux Ratio	Tower Jacket Temp.	Pot. Temp.
1														
2														
3														
4														
5														
6														
7														
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FIG. 9 Distillation Record

Liquid Volume (LV), % Distilled	Repeatability, °C
10	6.1
20	4.5
30	6.1
40	4.9
50	5.7
60	4.1
70	4.8
80	4.9
90	4.4

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

Liquid Volume (LV), % Distilled	Reproducibility, °C
10	16.9
20	12.8
30	13.5
40	11.2
50	14.2
60	8.4
70	11.4
80	5.1
90	4.4

12.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method for measuring vacuum distillation characteristics, bias has not been determined.

13. Keywords

13.1 crude oils; distillation; heavy oils; potstill; residue; vacuum

ANNEXES

(Mandatory Information)

A1. TEST METHOD FOR DETERMINATION OF TEMPERATURE RESPONSE TIME

A1.1 Scope

A1.1.1 The test method in this annex is for the determination of temperature response time based upon the rate of cooling of the sensor under prescribed conditions.

A1.2 Significance and Use

A1.2.1 This test method is performed to ensure that the sensor is able to respond to changes in temperature fast enough that no error due to lag is introduced in a rapidly rising temperature curve.

A1.2.2 The importance of this test method is greatest under vacuum conditions when the heat content of the vapors is minimal.

A1.3 Procedure

A1.3.1 Arrange a 1 L beaker of water on a hot plate with a glass thermowell supported vertically in the water. Maintain the temperature of the water at 80 °C ± 5°C (175 °F ± 9°F).

A1.3.2 Connect the sensor to an instrument, preferably with a digital readout, with readability to 0.1 °C. Alternatively, connect the sensor to a strip chart recorder of suitable range allowing interpolation to 0.1 °C (0.2 °F). Set the chart speed at 30 cm/h for readability.

A1.3.3 Insert the sensor into a hole in the center of one side of a closed cardboard box about 30 cm on a side. Hold the sensor in place by a friction fit on the joint. Allow the sensor to reach equilibrium temperature. Record the temperature when it becomes stable.

A1.3.4 Remove the sensor and insert it into the heated thermowell in the beaker of water. After the sensor has reached a temperature of 70 °C (158 °F), remove it and immediately insert it into the hole in the box. Note with a stopwatch, or record on the strip chart, the time interval while the sensor cools from 30 °C (54 °F) above to 5 °C (9 °F) above the temperature recorded in A1.3.3.

A1.3.5 A time interval in excess of 60 s is unacceptable.

A1.4 Precision and Bias

A1.4.1 No statement is made concerning either the precision or bias of this annex for determining the temperature response time because the result is used to determine whether there is conformance to the stated criteria in this test method.

A2. PRACTICE FOR CALIBRATION OF SENSORS

A2.1 Principle

A2.1.1 This practice deals with the basic calibration of temperature sensors and vacuum sensors and their associated recording instruments.

A2.1.2 The temperature sensor with its associated instrument is calibrated by observing and recording the temperature of the melting point and boiling point of pure compounds or eutectic mixtures.

A2.1.3 The vacuum sensor and its associated instrument is calibrated against a McLeod gauge or a certified reference gauge over the full operating range of pressure.

A2.2 Temperature Sensors

A2.2.1 *Apparatus*—A suitable apparatus is shown in Fig. A2.1. For the freezing point of water, a Dewar flask filled with crushed ice and water can be substituted. For the boiling point of water, use an equilibrium still or ebulliometer, a Tensimeter as shown in Fig. A2.2, or other apparatus for measuring vapor-liquid equilibrium.

A2.2.2 *Procedure A—Vapor Temperature Sensor Calibration by Melting Point:*

A2.2.2.1 Ensure that approximately 0.5 mL of silicone oil or other inert liquid is in the bottom of the thermowell and insert one or more thermocouples or other sensors connected to their respective measuring instruments.

A2.2.2.2 Heat the melting point bath to a temperature 10 °C above the melting point of the metal inside and hold at this temperature for at least 5 min to ensure that all the metal has melted.

A2.2.2.3 Discontinue heat input to the melting point bath and observe and record the cooling curve. When the curve exhibits a plateau of constant temperature for longer than 1 min, the temperature of the recorded plateau is accepted as the calibration temperature. If the freezing plateau is too short, it can be prolonged by employing some heat during the cooling cycle. Alternatively, the melt bath may have become contaminated or excessively oxidized. In this case, replace the metal.

A2.2.2.4 Record the calibration temperature at each of the points in Table A2.1 to the nearest 0.1 °C.

A2.2.2.5 Set up a correction table by listing the correction to be added algebraically to the observed temperature to give the true temperature at each calibration point. A graphical plot of

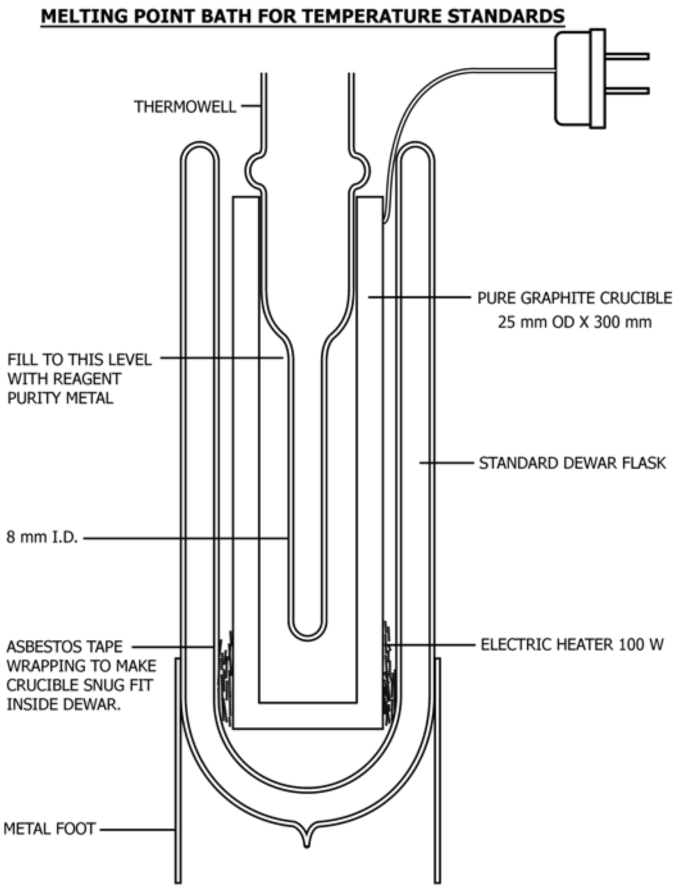

FIG. A2.1 Melting Point Bath for Temperature Standards

the above corrections connected by a smooth curve may be helpful in routine use.

A2.2.3 *Procedure B—Vapor Temperature Sensor Verification by Boiling Point:*

A2.2.3.1 This practice is not regarded as primary temperature reference but is an acceptable procedure for verification of calibrated temperature sensors.

A2.2.3.2 Set up the instrument for measuring vapor-liquid equilibrium, following the instructions of the manufacturer. Use only pure (>99.9 % purity) liquids with an accurately known boiling point. A list of some materials, which have been found suitable for this purpose, is given in Table A2.2.

NOTE A2.1—A tensimeter is a convenient instrument to measure vapor-liquid equilibrium.

A2.2.3.3 Follow the instructions of the instrument manufacturer and record the temperature at each of the points in Table A2.2 to the nearest 0.1 °C.

A2.2.3.4 Set up a table by listing the correction to be added to the observed temperature to give the true temperature at each of the verification points. If the corrections obtained differ by more then 0.4 °C from the corrections obtained during calibration (see A2.2.2.1), the system shall be considered as out of control and the sensor, with its associated instrument, shall be recalibrated.

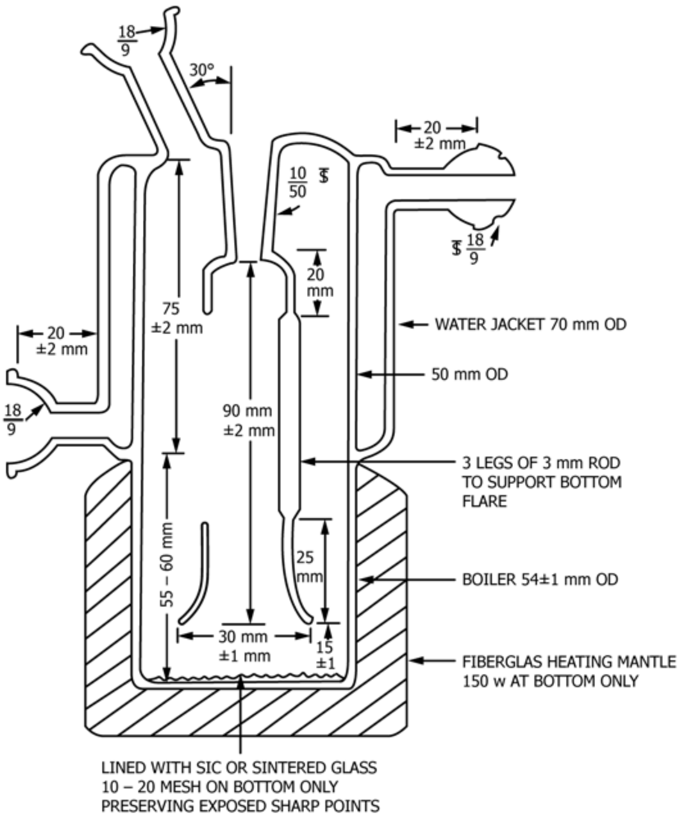

FIG. A2.2 Tensimeter

TABLE A2.1 Primary Temperature Standards (Melting Points)

Material		Temperature, °C
Ice	Melting point	0.0
Tin:Lead:Cadmium (50:32:18)	Melting point	145.0
Sn	Melting point	231.9
Pb	Melting point	327.4

TABLE A2.2 Boiling Points at Atmospheric Pressure

Material	Temperature, °C
Water	100.0
n-Heptane	98.5
Tetrahydronaphthalene	207.2
Tetradecane	252.5

A2.3 Vacuum Sensors

A2.3.1 *Apparatus:*

A2.3.1.1 Assemble a vacuum manifold such as that shown in Fig. A2.3. It must be capable of maintaining steady pressures within 1 % at all desired levels.

A2.3.1.2 Primary standards for the measurement of absolute pressure for pressures below 13.3 kPa such as the non-tilting McLeod gauge and other primary reference devices must be used as they are calibrated from measurement of the device's dimensions.

NOTE A2.2—The general principles of construction of McLeod gauges and other primary standard devices are well established. The dimensions and tolerances of such a gauge are beyond the scope of this test method.

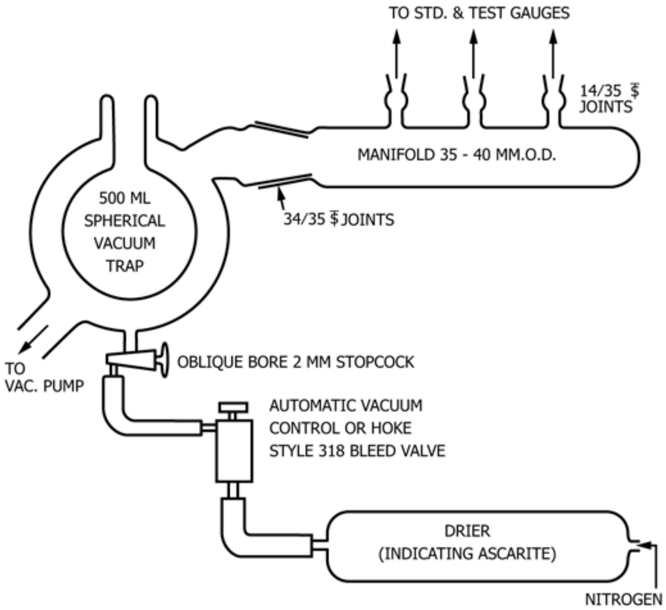

FIG. A2.3 Calibration of Vacuum Gauges

A2.3.1.3 Choose a McLeod gauge with a range such that the desired calibration pressure falls between 10 % and 90 % of the scale. Before refilling with clean mercury, heat the empty reference McLeod gauge at 250 °C for at least 30 min at a pressure below 10 Pa (0.075 mm Hg). Thereafter, carefully protect the reference gauge from exposure to moisture such as that from atmospheric air. The use of two reference McLeod gauges of different pressure ranges is recommended as a precaution. If they agree at the test pressure, it is an indication that the system is free of moisture and other condensables. (**Warning—**Mercury vapor is poisonous. Harmful or fatal if inhaled or ingested.)

A2.3.1.4 Alternatively, certified secondary gauges, electronic or otherwise, can be used, provided the output can be traced back to a primary standard. Secondary gauges shall be re-certified at a regular basis, but at least once a year.

A2.3.2 *Procedure:*

A2.3.2.1 Set up the test manifold such as that shown in Fig. A2.3. Ensure that the test manifold is clean, dry, leak-free, and can be maintained at a steady pressure at the required level. A suitable leak test is to pump down to a pressure below 0.01 kPa. Isolate the pump and the bleed valve. Allow the system to stabilize for at least 2 min. If the pressure rises more than 10 % in the next 2 min, the system must be checked for leaks and corrected before continuing with calibration.

A2.3.2.2 Connect the reference (primary) vacuum gauge(s) and the gauge(s) to be calibrated to the manifold. The gauges shall have such a range that the desired calibration pressure falls between 10 % and 90 % of the scale. Insert a dry ice trap between the manifold and the vacuum pump. Adjust the pressure to the required level for the test, and run a final leak test as above.

A2.3.2.3 After steady conditions have been maintained for at least 3 min, make readings of all gauges and compare with the reference gauge.

A2.3.2.4 Repeat the above procedure at the other required pressure levels. At least three pressure levels, covering between 10 % and 90 % of the scale, are required for each test gauge.

A2.3.2.5 Make up a chart of corrections to be added at each pressure level for each gauge tested. This can be used for interpolation when necessary. Alternatively, the settings of electronic gauges, if so equipped, may be adjusted to eliminate the error.

A3. TEST METHOD FOR DEHYDRATION OF A WET SAMPLE OF OIL

A3.1 Scope

A3.1.1 The test method in this annex is for dehydrating a wet sample of oil (>0.1 % water) prior to vacuum distillation and determining the water content.

A3.2. Summary of Test Method

A3.2.1 A sufficient quantity of the sample is distilled under atmospheric pressure to 150 °C, the hydrocarbon fraction decanted, and dry components recombined. The mass percent of water is calculated.

A3.3. Significance and Use

A3.3.1 Dehydration is important in order to allow the subsequent distillation to proceed smoothly.

A3.4. Apparatus

A3.4.1 The dehydration of a wet sample requires apparatus such as that shown in Fig. 1. Fit the distillation flask with a capillary line for the passage of nitrogen into the liquid.

A3.5. Procedure

A3.5.1 Decant any bulk water that may be present. Weigh by difference to the nearest gram, the required volume of wet sample into a distillation flask containing a magnetic stirrer.

A3.5.2 Attach the flask to the distillation head and pass a slow (8 cm^3/s) stream of nitrogen through the capillary. Vent the condenser through a trap maintained at the temperature of dry ice (−70 °C (−94 °F)).

A3.5.3 Apply heat to the flask, regulating it to attain a moderate rate. Remove distillate slowly until water ceases to distill and continue for an additional 3 % to 5 % of distillate.

A3.5.4 Shut off the heating system. Cool the flask and contents to below 175 °C temperature.

A3.5.5 Weigh the distillate fraction and residue.

A3.5.6 To separate the water from the distillate fraction, cool to −5 °C and decant the hydrocarbon liquid. Weigh the water.

A3.5.7 Remove the condenser and rinse it with alcohol or acetone to remove adhering drops of water. Dry with air and replace it.

A3.5.8 Recombine the cooled decanted fractions with the distillation residue, observing the usual precautions against losses. If the reblending is done in the original flask, this flask can be used for the subsequent distillation. Do not recombine the trap fraction.

A3.5.9 Record the quantity of dry oil recovered.

A3.6 Calculation

A3.6.1 Calculate the mass percent of water using Eq A3.1:

$$W = \frac{100\,A}{B} \qquad \text{(A3.1)}$$

where:

A = mass of water recovered, g,
B = mass of charge, g,
W = mass percent of water, and
100 = percentage constant.

A3.7 Precision and Bias

A3.7.1 No statement is made concerning either the precision or bias of this annex for mass percent water because the test method in this annex is used for sample preparation for Test Method D5236.

A4. PRACTICE FOR CONVERSION OF OBSERVED VAPOR TEMPERATURE TO ATMOSPHERIC EQUIVALENT TEMPERATURE (AET)

A4.1 Scope

A4.1.1 This practice is for conversion of the actual distillation temperature obtained at sub-ambient pressure to atmospheric equivalent temperature (AET) corresponding to the equivalent boiling point at atmospheric pressure, 101.3 kPa (760 mm Hg), by means of equations derived by Maxwell and Bonnell.[7]

A4.2. Significance and Use

A4.2.1 Final data on atmospheric equivalent temperatures are to be obtained by computation. Fig. 7 is provided only as a guide in estimating the AET during distillation.

A4.3. Calculation

A4.3.1 Convert observed vapor temperature to atmospheric equivalent temperature using Eq A4.1:

$$AET = \frac{748.1A}{[1/(T+273.15)]+0.3861A - 0.00051606} - 273.15 \qquad \text{(A4.1)}$$

where:

AET = atmospheric equivalent temperature, °C, and
T = observed vapor temperature, °C.

A4.3.1.1 If the operating pressure ≥0.266 kPa (≥2 mm Hg), calculate A using Eq A4.2 or Eq A4.3:

$$A = \frac{5.143222 - 0.972546 \log_{10} P}{2579.329 - 95.76 \log_{10} P} \qquad \text{(A4.2)}$$

where:

P = operating pressure, kPa, or

$$A = \frac{5.994295 - 0.972546 \log_{10} P}{2663.129 - 95.76 \log_{10} P} \qquad \text{(A4.3)}$$

where:

P = operating pressure, mm Hg.

A4.3.1.2 If the operating pressure <0.266 kPa (<2 mm Hg), calculate A using Eq A4.4 or Eq A4.5:

$$A = \frac{5.897249 - 0.987672 \log_{10} P}{2962.909 - 43.00 \log_{10} P} \qquad \text{(A4.4)}$$

where:

P = operating pressure, kPa, or

$$A = \frac{6.761559 - 0.987672 \log_{10} P}{3000.538 - 43.00 \log_{10} P} \qquad \text{(A4.5)}$$

where:

P = operating pressure, mm Hg.

A4.3.2 The equations are correct only for fractions that have a Watson *K*-factor of 12.0 ± 0.2. The *K*-factor shall be assumed to be 12 and any effect of *K*-factor ignored unless there is mutual agreement to the contrary.

A4.3.3 If correction is required, calculate the *K*-factor using Eq A4.6:

$$K = \frac{\sqrt[3]{1.8\,(B+273.15)}}{D} \qquad \text{(A4.6)}$$

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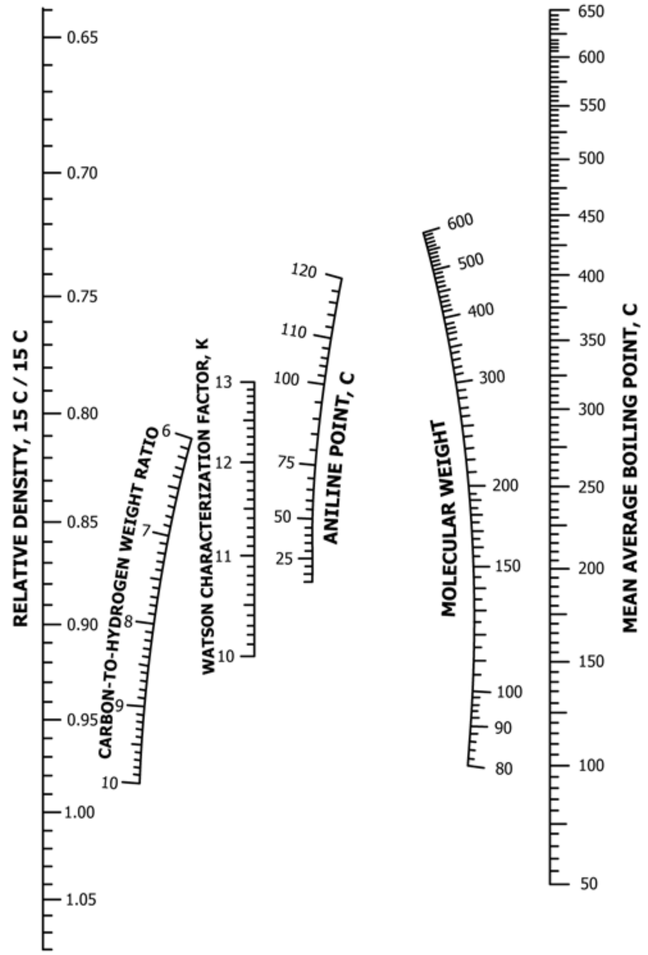
[7] Maxwell and Bonnell, *Industrial Engineering Chemistry*, Vol 49, 1957, p. 1187.



NOTE 1—Reprinted from API Technical Data Book, June 1980, by permission of American Petroleum Institute.

FIG. A4.1 Watson Characterization Factor of Petroleum Fractions

where:

B = mean average boiling point, °C, and
D = relative density at 15.6/15.6 °C.

By custom, either the mid-vapor temperature of the fraction or the midpoint of a gas chromatographic distillation of the fraction can be used for the mean average boiling point. In either case the method must be specified.

A4.3.3.1 An estimate of the *K*-factor can be made using Fig. A4.1.

A4.3.4 Calculate the correction to be applied to the AET using Eq A4.7:

$$t = -1.4[K - 12]\left[\log_{10}\left(\frac{P_a}{P_o}\right)\right] \qquad (A4.7)$$

where:

t = correction, °C,
P_a = atmospheric pressure, kPa (mm Hg), and
P_o = observed pressure, kPa (mm Hg).

A4.3.4.1 An estimate of the correction can be made using Fig. A4.2.

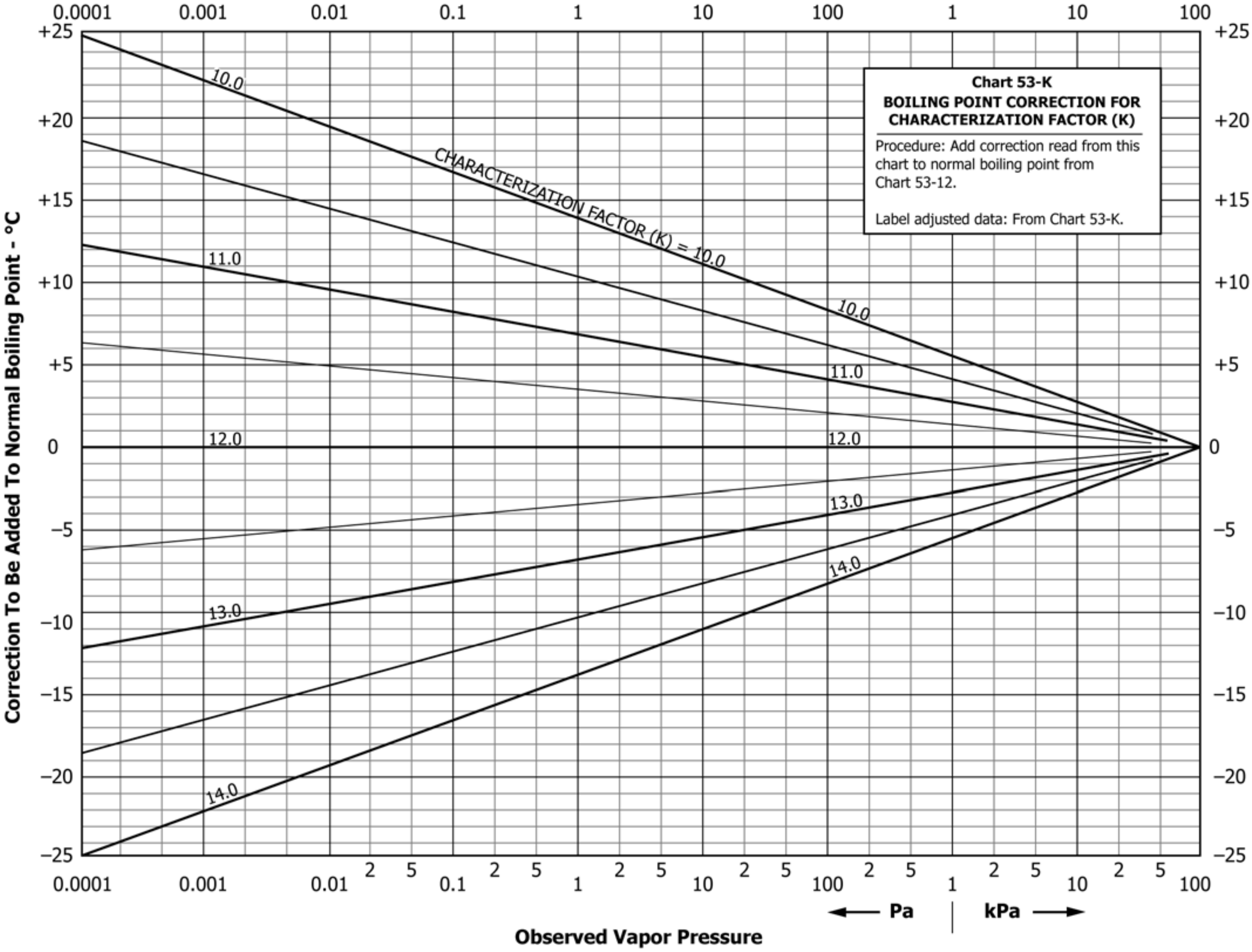


FIG. A4.2 Boiling Point Corrections for *K*—Factor

A5. TEST METHOD FOR DETERMINATION OF WETTAGE

A5.1 Scope

A5.1.1 The test method in this annex is for determining the amount of material that remains on the inside walls of the apparatus after a distillation is complete. It is intended for use mainly with stills having flasks too large for easy dismantling, but can also be used for smaller stills.

A5.2. Summary of Test Method

A5.2.1 A small charge of solvent is distilled in the dirty apparatus after a run. The residue is discharged and then freed of solvent to recover the wettage.

A5.3. Significance and Use

A5.3.1 Distillation apparatus can retain up to 0.5 % of a charge on their inside surfaces at the end of a run.

A5.3.2 Wettage includes that of the flask because the flask is not removed for separate treatment.

A5.4. Procedure

A5.4.1 Charge the dirty still with a volume of toluene equal to 10 % to 20 % of a normal charge.

A5.4.2 Apply heat and boil the toluene until all the upper parts are well rinsed (about 3 min), and shut down.

A5.4.3 After the still has cooled, recover the liquid from flask and distill off the solvent in a hood. Elimination of the last traces can be assisted by a gentle stream of air.

A5.4.4 Weigh the recovered wettage and determine its density.

A5.4.5 Treat the wettage as a separate fraction.

A5.5. Precision and Bias

A5.5.1 No statement is made concerning either the precision or bias of this annex for measuring wettage because the result is used solely within the context of Test Method D5236.

SUMMARY OF CHANGES

Subcommittee D02.08 has identified the location of selected changes to this standard since the last issue (D5236 – 13) that may impact the use of this standard. (Approved May 1, 2017.)

(1) Corrected Eq A4.1 and Eq A4.6.

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