



Designation: D5236 – 18a

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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

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 Wet-tage.

1.6 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

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, health, and environmental 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 substance that can cause serious medical issues. Mercury, or its vapor, has been demonstrated to be hazardous to health and corrosive to materials. Use Caution when handling mercury and mercury-containing products. See the applicable product Safety Data Sheet (SDS) for additional information. The potential exists that selling mercury or mercury-containing products, or both, is prohibited by local or national law. Users must determine legality of sales in their location.

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

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.

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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.

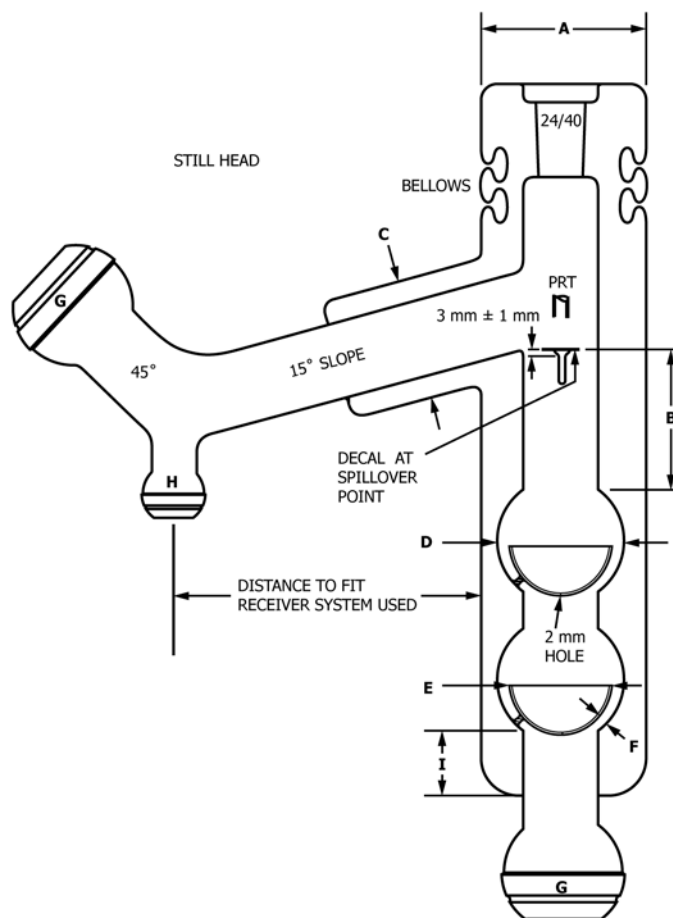
5.3 Residues to be used in the manufacture of asphalt can also be made but may not always be suitable. The long heat soaking that occurs in this test method may alter some of the properties.

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](#).

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

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