



Designation: **D6377—16 D6377 – 20**

Standard Test Method for Determination of Vapor Pressure of Crude Oil: VPCR_x (Expansion Method)¹

This standard is issued under the fixed designation D6377; 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 use of automated vapor pressure instruments to determine the vapor pressure exerted in vacuum of crude oils. This test method is suitable for testing samples that exert a vapor pressure between 25 kPa and 180 kPa at 37.8 °C at vapor-liquid ratios from 4:1 to 0.02:1 ($X = 4$ to 0.02).

NOTE 1—This test method is suitable for the determination of the vapor pressure of crude oils at temperatures from 0 °C to 100 °C and pressures up to 500 kPa, but the precision and bias statements (see Section 14) may not be applicable. The current precision of the method is limited at vapor-liquid ratios of 0.02 and 4. (Section 14 is inclusive of vapor-liquid ratios of 0.02 and 4).

1.2 This test method also allows the determination of vapor pressure for crude oil samples having pour points above 15 °C provided the proper sample handling, transfer, and analysis procedures are followed.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific warning statements, see 7.2.1 – 7.3.2.

1.5 *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

[D323 Test Method for Vapor Pressure of Petroleum Products \(Reid Method\)](#)

[D2892 Test Method for Distillation of Crude Petroleum \(15-Theoretical Plate Column\)](#)

[D3700 Practice for Obtaining LPG Samples Using a Floating Piston Cylinder](#)

[D4057 Practice for Manual Sampling of Petroleum and Petroleum Products](#)

[D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products](#)

[D5191 Test Method for Vapor Pressure of Petroleum Products and Liquid Fuels \(Mini Method\)](#)

[D5842 Practice for Sampling and Handling of Fuels for Volatility Measurement](#)

[D5853 Test Method for Pour Point of Crude Oils](#)

[D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material](#)

[D7975 Test Method for Determination of Vapor Pressure of Crude Oil: VPCR_x-F\(Tm°C\) \(Manual Expansion Field Method\)](#)

[D8009 Practice for Manual Piston Cylinder Sampling for Volatile Crude Oils, Condensates, and Liquid Petroleum Products](#)

3. Terminology

3.1 *Definitions:*

3.1.1 *platinum resistance thermometer, n*—temperature measuring device constructed with a length of platinum wire, whose electrical resistance changes in relation to temperature.

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

3.1.2 *vapor-liquid ratio (V/L), n*—the ratio of the vapor volume to the liquid volume of specimen, in equilibrium, under specified conditions.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *dead crude oil, n*—crude oil with sufficiently low vapor pressure that, when exposed to normal atmospheric pressure at room temperature, does not result in boiling of the sample.

3.2.1.1 *Discussion*—

Sampling and handling of dead crude oils can usually be done without loss of sample integrity or other problems by using normal, non-pressurized sample containers such as cans.

3.2.2 *live crude oil, n*—crude oil with sufficiently high vapor pressure that it would boil if exposed to normal atmospheric pressure at room temperature.

3.2.2.1 *Discussion*—

Sampling and handling live crude oils requires a pressurized sample system and pressurized sample containers to ensure sample integrity and prevent loss of volatile components.

3.2.3 *Reid vapor pressure equivalent (RVPE), n*—a value calculated by a defined correlation equation (see Eq X1.1) from $VPCR_4$ at 37.8 °C that is expected to be equivalent to the vapor pressure value obtained by Test Method D323.

3.2.3.1 *Discussion*—

As noted in 14.3 and Appendix X1, subsection X1.2, since Eq X1.1 for estimating RVPE is not universally applicable to all crude oils, it is recommended to report the $VPCR_4$ (37.8 °C) result for a crude oil sample.

3.2.4 *vapor pressure of crude oil ($VPCR_x$), n*—the pressure exerted in an evacuated chamber at a vapor-liquid ratio of X:1 by conditioned or unconditioned crude oil, which may contain gas, air or water, or a combination thereof, where X may vary from 4 to 0.02.

3.3 *Abbreviations:*

3.3.1 *ARV, n*—accepted reference value

3.3.2 *RVPE, n*—Reid vapor pressure equivalent

3.3.3 *V/L, n*—vapor liquid ratio

3.3.4 *$VPCR_x$, n*—vapor pressure of crude oil at x vapor liquid ratio

4. Summary of Test Method

4.1 Employing a measuring chamber with a built-in piston, a sample of known volume is drawn from the sample container into the temperature controlled chamber at 20 °C or higher. After sealing the chamber, the volume is expanded by moving the piston until the final volume produces the desired V/L value. The temperature of the measuring chamber is then regulated to the measuring temperature.

4.2 After temperature and pressure equilibrium, the measured pressure is recorded as the $VPCR_x$ of the sample. The test specimen shall be mixed during the measuring procedure by shaking the measuring chamber to achieve pressure equilibrium in a reasonable time of 5 min to 30 min.

4.3 For results related to Test Method D323, the final volume of the measuring chamber shall be five times the test specimen volume and the measuring temperature shall be 37.8 °C.

5. Significance and Use

5.1 Vapor pressure of crude oil at various V/Ls is an important physical property for shipping and storage.

NOTE 2—A vapor-liquid ratio of 0.02:1 ($X = 0.02$) mimics closely the situation of an oil tanker.

5.2 Vapor pressure of crude oil is important to crude oil producers and refiners for general handling and initial refinery treatment.

5.3 The vapor pressure determined by this test method at a vapor-liquid ratio of 4:1 ($VPCR_4$) of crude oil at 37.8 °C can be related to the vapor pressure value determined on the same material when tested by Test Method D323 (see Appendix X1).

5.4 Air saturation of crude oil shall not be done to avoid potential vapor loss. However air saturation of the chilled verification fluid is mandatory (see 7.2 and Section 11).

5.5 This test method can also be applied in online applications.

6. Apparatus

6.1 The apparatus suitable for this test method employs a small volume, cylindrically shaped measuring chamber with associated equipment to control the chamber temperature within the range from 0 °C to 100 °C. The measuring chamber shall contain a movable piston with a minimum dead volume of less than 1 % of the total volume at the lowest position to allow sample introduction into the measuring chamber and expansion to the desired V/L. A static pressure transducer shall be incorporated in the piston. The measuring chamber shall contain an inlet/outlet valve combination for sample introduction and expulsion. The piston and the valve combination shall be at the same temperature as the measuring chamber to avoid any condensation or excessive evaporation.

6.1.1 The measuring chamber shall be designed to have a total volume of 5 mL to 15 mL and shall be capable of maintaining a V/L of 4:1 to 0.02:1. The accuracy of the adjusted V/L shall be within 0.01.

NOTE 3—The measuring chambers employed by the instruments used in generating the precision and bias statements were constructed of nickel plated aluminium, stainless steel and brass with a total volume of 5 mL. Measuring chambers exceeding a 5 mL capacity and different design can be used, but the precision and bias statement (see Section 14) may not be applicable.

6.1.2 The pressure transducer shall have a minimum operational range from 0 kPa to 500 kPa with a minimum resolution of 0.1 kPa and a minimum accuracy of ± 0.5 kPa. The pressure measurement system shall include associated electronics and readout devices to display the resulting pressure reading.

6.1.3 Electronic temperature control shall be used to maintain the measuring chamber at the prescribed temperature within ± 0.1 °C for the duration of the test.

6.1.4 A platinum resistance thermometer shall be used for measuring the temperature of the measuring chamber. The minimum temperature range of the measuring device shall be from 0 °C to 100 °C with a resolution of 0.1 °C and an accuracy of ± 0.1 °C.

6.1.5 The vapor pressure apparatus shall have provisions for rinsing the measuring chamber with the next sample to be tested or with a solvent of low vapor pressure.

6.1.6 The vapor pressure apparatus shall have provisions for shaking the sample during the measuring procedure with a minimum frequency of 1.5 cycles per second.

6.2 *Vacuum Pump for Calibration*, capable of reducing the pressure in the measuring chamber to less than 0.01 kPa absolute.

6.3 *McLeod Vacuum Gage or Calibrated Electronic Vacuum Measuring Device for Calibration*, to cover at least the range of 0.01 kPa to 0.67 kPa. The calibration of the electronic measuring device shall be regularly verified in accordance with Annex A of Test Method **D2892**.

6.4 *Pressure Measuring Device for Calibration*, capable of measuring local station pressure with an accuracy and a resolution of 0.1 kPa or better, at the same elevation relative to sea level as the apparatus in the laboratory.

NOTE 4—This standard does not give full details of instruments suitable for carrying out this test. Details on the installation, operation and maintenance of each instrument may be found in the manufacturer's manual.

7. Reagents and Materials

7.1 *Purity of Reagents*—Use chemicals of at least 99 % purity for verification of instrument performance (see Section 11). Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

7.1.1 The chemicals in 7.2.1, 7.2.2, and 7.2.3 are suggested for verification of instrument performance (see Section 11), based on the air saturated reference fuels analyzed in the Test Method **D5191** 2003 interlaboratory study (ILS)⁴ (see Table 1). P_{tot} used

TABLE 1 Accepted Reference Value (ARV) and Acceptable Testing Range for Air Saturated Reference Fluids^A

Reference Fluid	ARV [P_{tot}] \pm Uncertainty, (kPa)	Recommended Instrument Manufacturer Tolerance, (kPa)	Acceptable Testing Range for Reference Fuel [P_{tot}], (kPa)
Pentane	112.8 \pm 0.2	\pm 1.0	112.8 \pm 1.2 (111.6 to 114.0)
2,2 Dimethylbutane	74.1 \pm 0.2	\pm 1.0	74.1 \pm 1.2 (72.9 to 75.3)
2,3 Dimethylbutane	57.1 \pm 0.2	\pm 1.0	57.1 \pm 1.2 (55.9 to 58.3)

^A Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1619.

³ *Reagent Chemicals, American Chemical Society Specifications* ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

⁴ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1619. Contact ASTM Customer Service at service@astm.org.

in Test Method **D5191** is equivalent to VPCR₄ used in this test method). Such reference fuels are not to be used for instrument calibration. **Table 1** identifies the accepted reference value (ARV) and uncertainty limits, as well as the acceptable testing range for each of the reference fuels listed.

7.1.2 The chemicals in **7.3.1** and **7.3.2** are suggested for use as rinsing solvents capable of cleaning the measuring chamber, the valves and the inlet and outlet tubes.

7.2 Verification Fluids:

7.2.1 *2,2-Dimethylbutane*, (**Warning**—2,2-dimethylbutane is flammable and a health hazard.)

7.2.2 *2,3-Dimethylbutane*, (**Warning**—2,3-dimethylbutane is flammable and a health hazard.)

7.2.3 *Pentane*, (**Warning**—Pentane is flammable and a health hazard.)

7.3 Solvents:

7.3.1 *Toluene*, (**Warning**—Toluene is flammable and a health hazard.)

7.3.2 *Acetone*, (**Warning**—Acetone is flammable and a health hazard.)

8. Sampling and Sample Introduction

8.1 General Requirements:

8.1.1 The extreme sensitivity of vapor pressure measurements to losses through evaporation and the resulting changes in composition requires the utmost precaution and the most meticulous care in the drawing and handling of samples. Sampling of live crude oil shall be performed in accordance with Practice **D3700** or Practice **D8009**. Sampling in accordance with Practice **D4057** shall only be used for dead crude oil and if Practice **D3700** or Practice **D8009** is impractical.

NOTE 5—Sampling in accordance with Practice **D4177** may also be used instead of Practice **D4057**.

8.1.2 For sampling in accordance with Practice **D3700**, a floating piston cylinder with a minimum sample volume of 200 mL shall be used if the overall volume of the test specimen required for the vapor pressure determination, including the rinsing procedure is not larger than 20 mL. Larger floating piston cylinders can be used. The minimum piston back-pressure shall be higher than the sample vapor pressure at the introduction temperature of the measuring chamber plus 100 kPa for the shifting of the piston. The maximum back-pressure shall not exceed the maximum measurement pressure of the apparatus pressure transducer. Compressed air, or any other, non-flammable compressed gas, can be used as the back-pressuring agent. The floating piston cylinder shall have provisions for mechanical stirring of the sample and a second valve at the inlet for rinsing.

8.1.3 Do not unnecessarily expose the samples to temperatures exceeding 30 °C during sampling and storage. For prolonged storage, store the samples in an appropriate room or refrigerator.

8.1.4 Perform the vapor pressure determination on the first test specimen withdrawn from the cylinder after the rinsing step in **9.3**. Do not use the remaining sample in the floating piston cylinder for more than three repeat vapor pressure determinations.

8.1.5 In the case of quality control checks with pure compounds (see **11.1**), smaller sample containers without applied pressure can be used.

8.2 Sampling Procedures:

8.2.1 If the sample is contained in a pressurized source like a pipeline, use a floating piston cylinder and obtain the sample directly from the source under pressure. Rinse the cylinder by opening the rinsing valve until the crude oil emerges at the second inlet. Close the rinsing valve and let the piston move slowly until at least 200 mL of sample has entered the cylinder. Close the inlet valve and apply the back pressure immediately. Check the filling of the cylinder to be at least 200 mL.

NOTE 6—The current precision statements were derived from the 2005 ILS using samples in 250 mL floating piston cylinders (see **14.3**).

8.2.2 If the sample is taken from a non-pressurized source like a storage tank, oil tanker, drum or other container, obtain a sample and test specimen in accordance with Practice **D4057**. Use either a 250 mL or 1 L sized container filled between 70 % and 80 % with sample. For best testing precision (reproducibility), it is recommended that a 1 L sized container be used.

NOTE 7—The current precision statements were derived from the 2005 ILS using samples in 1 L amber glass containers (see **14.3**). However, samples in containers of other sizes, as prescribed in Practice **D4057**, may be used with the same ullage requirement if it is recognized that the precision can be affected.

8.3 Sample Transfer:

8.3.1 Transfer the sample at room temperature but at least 5 °C above the pour point (as determined by Test Method **D5853**) of the sample from the container into the measuring chamber.

8.3.2 If the sample is contained in a pressurized floating piston cylinder, apply a back-pressure which is higher than the vapor pressure of the sample at the introduction temperature plus a minimum of 100 kPa for the piston movement. The applied back-pressure shall not exceed the maximum limit of the pressure transducer used in the vapor pressure apparatus.

8.3.3 If the sample is contained in a nonpressurized container, transfer the sample into the measuring chamber using a transfer tube or a syringe. **Warning**—If the sample flashes or outgases in the inlet line, either before or after any inlet filter or regulator, then liquid volume to the cell is reduced and measured vapor pressure is falsely low. If flashing or outgassing occurs, results are no longer valid under **D6377**.

9. Preparation of Apparatus

9.1 Prepare the instrument for operation in accordance with the manufacturer's instructions.

9.2 Rinse the measuring chamber, if necessary with a solvent. Toluene has a low vapor pressure and can be used successfully. Rinsing is performed by drawing the solvent into the chamber by the piston and expelling the solvent into the waste container.

9.3 To avoid contamination of the test specimen with the previous sample or the solvent, rinse the measuring chamber a minimum of three times with the sample to be tested. Fill the measuring chamber with sample to at least half the total volume of the chamber for each rinse. This rinsing procedure shall always be carried out immediately before the measuring procedure (see Section 12).

10. Calibration

10.1 Pressure Transducer:

10.1.1 Perform a calibration check of the transducer as indicated from the quality control checks (see Section 11). The calibration of the transducer is checked using two reference points, zero pressure (<0.1 kPa) and the ambient barometric pressure.

NOTE 8—Calibration frequency of the pressure transducer may vary with instrument type and frequency of use. A calibration check of the pressure transducer at least once every six months is recommended.

10.1.2 Connect a McLeod gage or a calibrated electronic vacuum measuring device to the vacuum source in line with the measuring chamber (see Note 7). Apply vacuum to the measuring chamber (see Note 9). When the vacuum measuring device registers a pressure less than 0.1 kPa, adjust the transducer control to zero or to the actual reading on the vacuum measuring device as dictated by the instrument design or manufacturer's instructions.

NOTE 9—Refer to Annex A6.3 of Test Method D2892 for further details concerning the calibration of electronic vacuum measuring devices and proper maintenance of McLeod gages.

10.1.3 Open the measuring chamber of the apparatus to atmospheric pressure, and observe the corresponding pressure value of the transducer. Ensure that the apparatus is set to display the total pressure and not a calculated or corrected value. Compare this pressure value with the pressure obtained from a pressure measuring device (see 6.4), as the pressure reference standard. The pressure measuring device shall measure the local station pressure at the same elevation as the apparatus in the laboratory at the time of pressure comparison. When the instrument is used over the full pressure range, a calibration with a dead weight balance shall be carried out. (**Warning**—Many aneroid barometers, such as those used at weather stations and airports, are pre-corrected to give sea level readings; these shall not be used for calibration of the apparatus.)

10.1.4 Repeat 10.1.2 and 10.1.3 until the zero and barometric pressures read correctly without further adjustments.

10.2 *Temperature Sensor*—Verify the calibration of the platinum resistance thermometer used to monitor the measuring chamber temperature at least every six months against a thermometer which is traceable to National Institute of Standards and Technology (NIST) or to national authorities in the country the equipment is used.

11. Quality Control Checks

11.1 Use a verification fluid (see 7.2) of known volatility as an independent check against the instrument calibration each day the instrument is in use. For pure compounds, multiple test specimens may be taken from the same container over time. Air saturate the verification fluid at temperatures between 0 °C to 1 °C as described in Test Method D5191 (see 8.4). Transfer the verification fluid into the measuring chamber using a transfer tube or a syringe. The temperature of the verification fluid shall be at 0 °C to 3 °C during the sample introduction, and the measuring procedure shall be in accordance with Section 12 with a V/L ratio of 4:1 and a measuring temperature of 37.8 °C.

11.2 Table 1 provides the accepted reference value (ARV) [P_{tot}] and uncertainty limits (at least 95 % confidence interval) of reference fluids tested in the 2003 D5191 ILS.⁴ As stated in 7.1.1, the " P_{tot} " reference used in Test Method D5191 (and Table 1 of this test method) is equivalent to VPCR₄ (37.8 °C) in this test method. This information, combined with the tolerance value recommended by instrument manufacturers, was used to establish the acceptable testing range for the reference fuels to verify instrument performance.

11.3 Values obtained within the acceptable testing range intervals in Table 1 indicate that the instrument is performing at the level deemed acceptable by this standard. If values outside the acceptable testing range intervals are obtained, verify the quality of the pure compound(s) and re-check the calibration of the instrument (see Section 10). (**Warning**—The use of single component verification materials such as those listed in Table 1 will only prove the calibration of the equipment. It will not check the accuracy of the entire test method, including sample handling, because losses due to evaporation will not decrease the sample vapor pressure as happens with losses of light ends in multi component mixtures. The vapor pressure measurement process (including operator technique) can be checked periodically by performing this test method on previously prepared samples from one batch of product,