



Designation: D7975 – 22

Standard Test Method for Determination of Vapor Pressure of Crude Oil: VPCR_x-F(Tm°C) (Manual Expansion Field Method)¹

This standard is issued under the fixed designation D7975; 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 manual vapor pressure instruments to determine the vapor pressure of crude oils exerted in a vacuum. 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 0.25:1 ($X = 4$ to 0.25, see 3.2.4).

NOTE 1—This test method is suitable for the determination of the vapor pressure of crude oils at temperatures from 0 °C to 60 °C and pressures up to 500 kPa, but the precision and bias statements (see Section 15) may not be applicable.

1.2 This test method is meant primarily for use under field conditions for immediate evaluation of vapor pressure for storage, transport, or operational uses.

1.3 This test method is not intended for use in custody transfer applications. Test Method D6377 shall be used for custody transfer applications.

1.4 This test method provides a reasonable confirmation for the presence of light ends in the source material given that the partial pressure of low boiling components contribute significantly to total vapor pressure.

1.5 This test method allows both sample collection and subsequent vapor pressure measurement of crude oil samples directly from the sample source. The collected sample may also be transferred to an automated vapor pressure instrument such as used for Test Method D6377. The field test apparatus is suitable for transportation provided suitable over-pack is used to meet the regulations for the transportation of dangerous goods in the transportation jurisdiction(s).

1.6 This test method allows the determination of vapor pressure for crude oil samples having pour points below 0 °C and flow at the sampling conditions to allow inlet to the apparatus.

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

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 Dec. 1, 2022. Published January 2023. Originally approved in 2014. Last previous edition approved in 2014 as D7975 – 14. DOI: 10.1520/D7975-22.

1.7.1 *Exception*—Other units of measurement are included in this standard for ease of use in multiple jurisdictions.

1.8 *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.*

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

D323 Test Method for Vapor Pressure of Petroleum Products (Reid Method)

D1193 Specification for Reagent Water

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D5191 Test Method for Vapor Pressure of Petroleum Products and Liquid Fuels (Mini Method)

D5853 Test Method for Pour Point of Crude Oils

D6377 Test Method for Determination of Vapor Pressure of Crude Oil: VPCR_x (Expansion Method)

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

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:*

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

3.2.1 *dead crude oil, n*—a term usually employed for crude oils that, when exposed to normal atmospheric pressure at room temperature, will *not* result in actual boiling of the sample.

3.2.1.1 *Discussion*—These crudes will have vapor pressures below atmospheric pressure at room temperature.

3.2.1.2 *Discussion*—This method will provide vapor pressure information that will allow the user to determine if a crude oil is considered “dead” or “live.” A crude oil shall always be considered “live” until the vapor pressure can be established. Sampling and handling of dead crude oils can usually be done without concern in normal, non-pressurized sample containers, such as cans and other atmospheric containers.

3.2.2 *light ends, n*—components that cannot be maintained as a liquid at atmospheric pressure at temperatures greater than 0 °C.

3.2.2.1 *Discussion*—This includes any materials that have atmospheric boiling points below 0 °C including methane, ethane, propane, butane, and fixed gases such as H₂S, CO₂, N₂, O₂, H₂, CO.

3.2.3 *live crude oil, n*—a term usually employed for crude oils contained in pressurized systems that, when brought to normal atmospheric pressure at room temperature, will result in actual boiling of the sample.

3.2.3.1 *Discussion*—Sampling and handling of samples of live crude oils will necessitate the use of the field test apparatus and preclude the use of normal sample containers, such as cans and other atmospheric containers.

3.2.4 *vapor pressure of crude oil field measurement (VPCR_{X-F}), 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 0.25.

3.2.4.1 *Discussion*—This measurement is performed using the field test apparatus described herein.

3.3 Abbreviations:

3.3.1 *ARV, n*—accepted reference value.

3.3.2 *RVP, n*—Reid Vapor Pressure.

3.3.3 *V/L, n*—vapor/liquid ratio.

3.3.4 *VPCR_{X-F}(T_m°C), n*—vapor pressure of crude oil field (-F) measurement at X vapor/liquid ratio at (T_m°C) measured temperature.

3.3.5 *STP, n*—Standard Pressure (101.325 kPa) and Temperature (0 °C)

4. Summary of Test Method

4.1 Using a portable variable volume measurement chamber with a built-in piston, a sample of crude oil of known volume is drawn from the sample point or sample container into the field test apparatus (see 7.1). 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 a specific measuring temperature if required (see 7.2).

4.2 After temperature and pressure equilibrium, the measured pressure is recorded as the VPCR_{X-F}(T_m°C) of the

sample. The test specimen is mixed during the measuring procedure by shaking the measuring chamber to achieve pressure equilibrium in a reasonable time of 1 min to 5 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 (4:1 V/L ratio) and the measuring temperature shall be 37.8 °C. The 4:1 V/L ratio is based on filling the measurement chamber 20 % full with liquid with vapor expansion to 100 %.

4.4 For the purpose of transportation, a lower V/L ratio is desirable to simulate the vapor pressure in vessel with 20 % outage. Filling the measurement chamber with 80 % liquid and expanding to 100 % will provide a 0.25:1 V/L ratio.

5. Significance and Use

5.1 Vapor pressure of crude oil at various vapor/liquid ratios is an important physical property for transport, storage, and refinery operations.

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

5.3 The direct sample collection and subsequent, in place, vapor pressure measurement without the need for further sample handling eliminates the potential loss of light hydrocarbon material from the sample. The combination of sampling and testing may produce higher results than Test Method D323.

5.4 Chilling and air saturation of the sample prior to the vapor pressure measurement (as required in Test Method D323) is not required in this test method.

6. Interferences

6.1 Exposure of a sample to the atmosphere will result in air saturation of the sample and loss of volatile material. At low V/L ratios, the pressure exerted by air saturation can have a material effect on the vapor pressure measurement. Sample exposure to the atmosphere is avoided by sampling directly into the field test apparatus (see 7.1) from the sample source. The sample source shall not be an atmospheric container, such as a can or bottle, since vapor pressure measurement results can be impacted by air saturation and loss of volatile material.

7. Apparatus

7.1 *Field Test Apparatus*³—The apparatus is shown in Fig. 1 and described in detail in Annex A1.

7.1.1 The measuring chamber shall be designed to have a total volume of 100 mL to 120 mL and shall be capable of maintaining a V/L of 4:1 to 0.25:1. The accuracy of the adjusted V/L shall be within ±0.01.

NOTE 2—The measuring chambers employed by the instruments used in generating the precision and bias statements were constructed as per A1.1,

³ The sole source of supply of the apparatus known to the committee at this time is Parkes Scientific Inc. 17360-108 Ave NW, Edmonton, AB, Canada T5S 1E8, (780) 484-1849. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.



FIG. 1 Field Test Apparatus

with a total volume of 100 mL to 120 mL. Measuring chambers exceeding a 120 mL capacity and different design can be used, but the precision and bias statement (see Section 15) may not be applicable.

7.1.2 The vapor pressure apparatus shall have provisions to measure the sample vapor pressure (vapor pressure measurement gauge) and shall have a minimum operational range from 0 kPa to 200 kPa with a minimum resolution of 5 kPa and a minimum accuracy of ± 2 kPa (see A1.1.1.3).

NOTE 3—Many gauges will provide a positive and negative scale with zero considered barometric pressure (101.325 kPa at STP). For example: Gauge scales will read -100 kPa to 100 kPa but the overall operating range is 200 kPa.

7.1.3 The vapor pressure apparatus shall have provisions to measure the sample source pressure (source pressure measurement gauge) and shall have a minimum operational range from 0 kPa to 400 kPa with a minimum resolution of 20 kPa and a minimum accuracy of ± 10 kPa.

7.1.4 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.

7.1.5 The vapor pressure apparatus shall be of such configuration as to be easily shaken during the measurement procedure with a minimum frequency of 1 cycle per second. Shaking is intended to be performed by hand or could be performed by mechanical shaker or equivalent.

7.1.6 The vapor pressure apparatus shall be equipped with a pressure relief device set to a maximum pressure of 700 kPa (nominal 100 psi).

7.2 *Temperature Controlled Bath (Optional)*—A water or other suitable liquid bath capable of maintaining the desired test temperature between 0°C and 60°C . Temperature controlled bath shall be suitable in length, width and depth to completely immerse the measurement chamber while it is in a horizontal position. Other suitable heating devices may be used provided the test temperatures are within the manufacturer's specification for the measurement gauges. Examples of other suitable heating devices may include: ovens, air baths, jacket or hot-block type heaters.

7.3 *Temperature Controlled Bath Temperature Measuring Device (Optional)*—An electronic measuring device such as a resistance device or thermocouple capable of measuring the temperature of the temperature control bath with a resolution of 0.5°C and an accuracy of $\pm 2^{\circ}\text{C}$.

7.4 *Vapor Pressure Apparatus Temperature Measuring Devices (Acceptable Devices)*:

7.4.1 A surface temperature measuring device, such as an IR thermal gun or surface mounted liquid crystal thermometer, capable of measuring the temperature of the measurement cylinder with a resolution of 0.5°C and an accuracy of $\pm 2^{\circ}\text{C}$.

7.4.2 An electronic measuring device such as a resistance device or thermocouple inserted into a thermal well within the piston rod capable of measuring the temperature of the measurement cylinder with a resolution of 0.5°C and an accuracy of $\pm 2^{\circ}\text{C}$.

7.5 *Portable Barometer*—A barometer with minimum pressure range of 95 kPa and 105 kPa with an accuracy of $\pm 0.5\%$ of reading and a resolution of 0.05 kPa.

7.6 *Top Loading Balance*—A top loading balance with a capacity of 500 g to 3 digits accuracy ± 0.001 g.

7.7 *Vacuum Pump*—A vacuum pump capable of generating a minimum vacuum of 1.3 kPa.

7.8 *Low Pressure Tubing*—6.35 mm ($1/4$ in.) inert flexible tubing for transferring fluids at atmospheric pressure.

7.9 *Low Pressure Tubing*—3.175 mm ($1/8$ in.) inert flexible tubing for transferring fluids at atmospheric pressure.

7.10 *High Pressure Tubing*—6.35 mm ($1/4$ in.) inert flexible tubing for transferring fluids up to 689 kPa (100 psi).

7.11 *Purge Container*—A container suitable for purging fluids during sampling operations.

8. Reagents and Materials

8.1 *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.⁴ 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.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II in Specification **D1193**.

8.2.1 The chemicals in 8.3.1, 8.3.2, and 8.3.3 are suggested for verification of instrument performance (see Section 11), based on the air saturated reference fuels analyzed in the Test Method **D5191** 2003 inter-laboratory study (ILS)⁵ (see **Table 1**). P_{tot} used in Test Method **D5191** is equivalent to VPCR4-F(37.8 °C) used in this test method. **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.

8.2.2 The chemicals in 8.4.1 and 8.4.2 are suggested for use as rinsing solvents capable of cleaning the measuring chamber, the valves and the inlet and outlet tubes.

8.3 Verification Fluids:

8.3.1 *2,2-Dimethylbutane*, (**Warning**—2,2-dimethylbutane is flammable and a health hazard.)

8.3.2 *2,3-Dimethylbutane*, (**Warning**—2,3-dimethylbutane is flammable and a health hazard.)

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

8.4 Solvents:

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

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

8.5 Silicon-based or perfluorocarbon based lubricant is recommended for hydraulic cylinder piston lubrication.

8.6 Water.

9. Preparation of Apparatus

9.1 Rinse the measuring chamber, if necessary, with a solvent (see 8.4). Toluene has a low vapor pressure and has been used successfully. The measuring chamber is rinsed by drawing the solvent into the chamber by extension of the piston and expulsion of the solvent into the waste container by compressing the piston (see 12.2 Sampling Procedure).

9.2 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 sampling and measuring procedure (see section 12.3).

10. Calibration and Standardization

10.1 Cylinder Dead Volume Calibration:

10.1.1 CLOSE valves A and B and compress the cylinder piston into the cylinder body completely. See **Fig. 2-A**.

10.1.2 CONNECT valve A to a vacuum source.

10.1.3 OPEN valve A to begin evacuation of the apparatus.

10.1.4 OPEN valve B and confirm vacuum pressure is greater than 85 kPa (25 inHg) below atmospheric pressure.

NOTE 4—Some vacuum gauges read in negative values and use atmospheric pressure as zero.

10.1.5 Allow 5 min to evacuate.

10.1.6 CLOSE valve A and monitor the pressure for a further 2 min.

10.1.6.1 If there is no change in vacuum pressure, then proceed to 10.1.7.

10.1.6.2 If there is a loss of vacuum pressure, then the system has a leak that shall be resolved prior to proceeding. Follow procedures in **Annex A1** regarding the apparatus.

10.1.7 DISCONNECT vacuum source from valve A.

10.1.8 CONNECT a short length of 3.175 mm (1/8 in.) tubing to valve A (copper, vinyl, or TFE-fluorocarbon tubing are commonly available). This length should be kept as short as possible.

10.1.9 FILL a 500 mL beaker with approximately 300 mL water.

10.1.10 PLACE the beaker on the top loading balance.

10.1.11 INSERT the tubing from the cylinder in the water filled beaker without touching the sides or bottom of the beaker. Suspending the tubing using a ring stand or other means will allow the tubing to be in the beaker but not touching it. See **Fig. 2-B**.

⁴ 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.

⁵ 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.

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

Reference Fluid	ARV [P _{tot}] ± Uncertainty, (kPa) @ V/L 4:1 (37.8 °C)	Acceptable Testing Range for Reference Material [P _{tot}], (kPa)
Pentane	112.8 ± 0.2	112.8 ± 1.2 (111.6 to 114.0)
2,2 Dimethylbutane	74.1 ± 0.2	74.1 ± 1.2 (72.9 to 75.3)
2,3 Dimethylbutane	57.1 ± 0.2	57.1 ± 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.

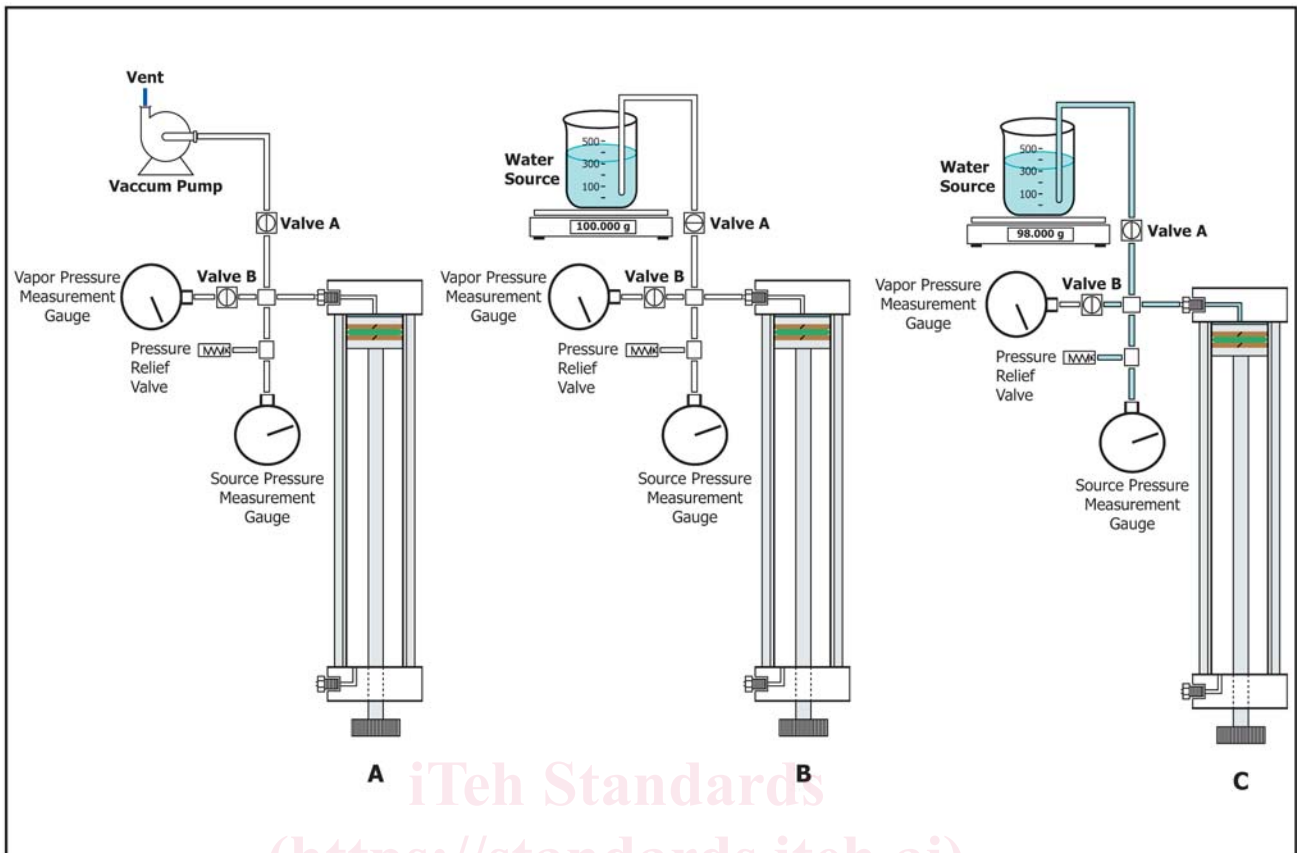


FIG. 2 Dead Volume Calibration

10.1.12 CLOSE valve B to avoid filling the vapor pressure measurement gauge with water.

10.1.13 RECORD the mass of water in the beaker as the initial mass of water (W1).

10.1.14 OPEN valve A to allow water to be displaced into the apparatus filling the dead volume. See Fig. 2-C.

10.1.15 RECORD the mass of water in the beaker as the final mass of water (W2).

10.1.16 RECORD the water temperature and locate the water density at that temperature.

10.1.17 CALCULATE the dead volume using Eq 1.

$$DV = \left(\frac{W1 - W2}{DW} \right) \quad (1)$$

where:

- W1 = initial mass of water in the beaker (g),
- W2 = final mass of water in the beaker (g),
- DW = density of water at the calibration temperature (g/mL), and
- DV = dead volume (mL).

10.1.18 Continue directly to 10.2 to proceed with the cylinder volume guide calibration. DO NOT move the piston or add or remove water from the beaker.

10.1.19 If any component of the apparatus is altered or replaced after calibration repeat 10.1.1 to 10.1.16.

10.2 Cylinder Volume Guide Calibration:

10.2.1 Starting from 10.1.16 with the dead volume full of water. See Fig. 3-A.

10.2.2 PULL the cylinder piston out enough to allow the insertion of the volume measurement 20 % guide. See Fig. 3-B.

10.2.3 With the guide in place and the piston firmly pressed to the guide, wait for the mass of water to stabilize.

10.2.4 Once the mass of water has been stable for 5 s, RECORD the mass of water in the beaker as W3.

10.2.5 REMOVE the 20 % guide and PULL the piston out enough to allow insertion of the 80 % guide. See Fig. 3-C.

10.2.6 With the guide in place and the piston firmly pressed to the guide, WAIT for the mass of water to stabilize.

10.2.7 Once the mass of water has been stable for 5 s, RECORD the mass of water in the beaker as W4.

10.2.8 REMOVE the 80 % guide and PULL the piston to the fully extended position.

10.2.9 With the piston in the fully extended position, RECORD the mass of water in the beaker as W5.

10.2.10 CALCULATE the cylinder volume, guide volume and guide volume percent as Eq 2 through Eq 5.

10.2.11 For other volume guides, see Annex A1.

$$GV1 = \left[\frac{(W2 - W3)}{DW} \right] + DV \quad (2)$$

$$GV2 = \left[\frac{(W2 - W4)}{DW} \right] + DV \quad (3)$$

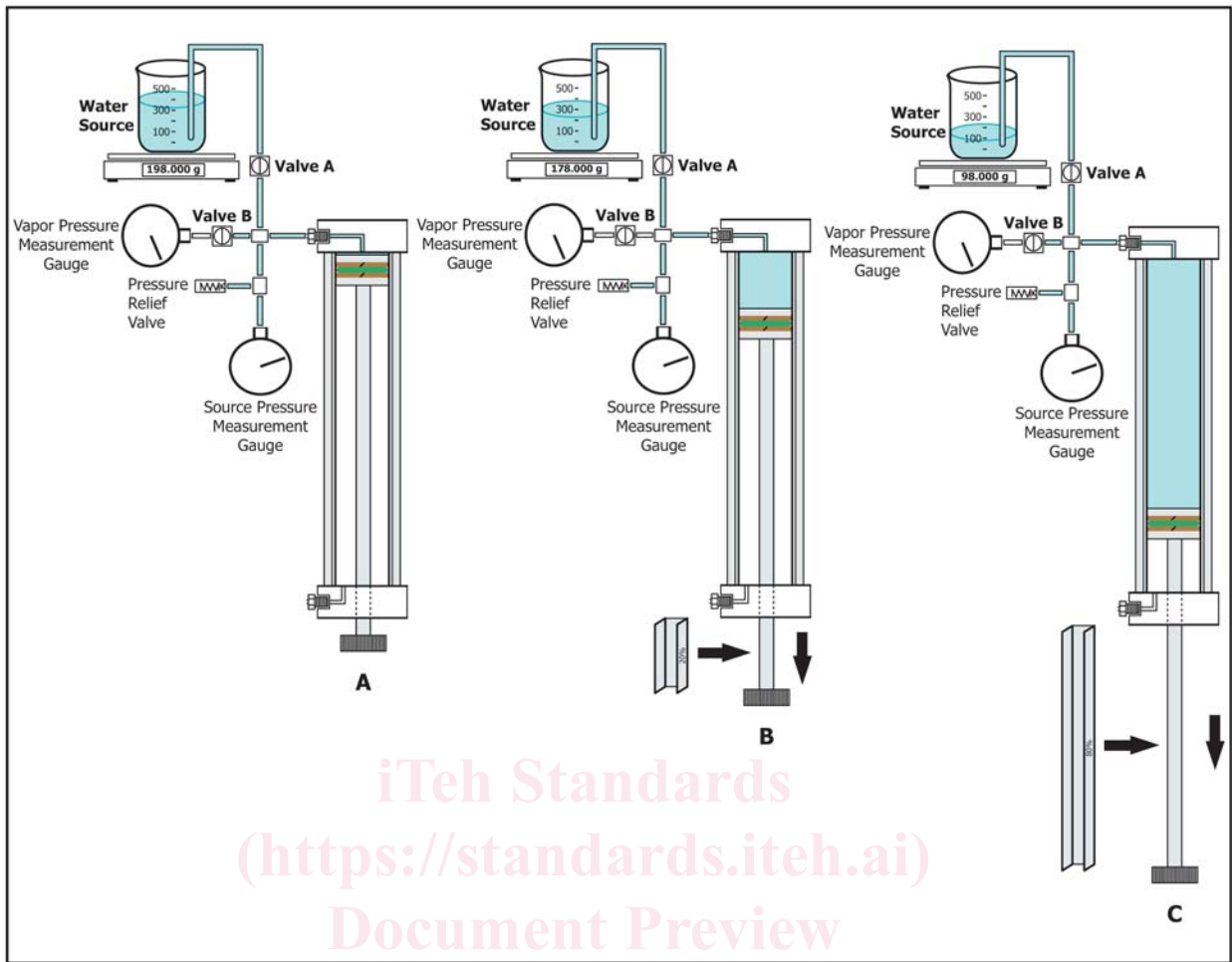


FIG. 3 Cylinder Volume Guide Calibration

iTeh Standards
 (https://standards.itih.ai)
 Document Preview

$$TV = \left[\frac{(W_2 - W_5)}{DW} \right] + DV \quad (4)$$

$$GP = \left[\frac{(GV_x)}{TV} \right] \times 100 \quad (5)$$

where:

- GV1 = 20 % Guide volume (mL),
- GV2 = 80 % Guide volume (mL),
- TV = total cylinder volume (mL),
- GP = guide volume percent (percent by volume),
- W2 = final mass of water in the beaker after dead volume calibration (g),
- W3 = mass of water in the beaker required to fill cylinder to the 20 % guide (g),
- W4 = mass of water in the beaker required to fill cylinder to the 80 % guide (g),
- W5 = mass of water in the beaker required to fill the fully extended cylinder (g),
- DW = density of water at the calibration temperature (g/mL),
- GV_x = guide volume with x as the specific guide (GV1 or GV2),
- DV = dead volume of the apparatus from 10.1.17 (mL), and
- GP = guide volume percent (volume percent).

11. Quality Control Checks

11.1 Use a verification fluid (see 8.3) of known volatility as an independent check against the instrument calibration as required. 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 cell using a transfer tube or a syringe. The temperature of the verification fluid shall be at 0 °C to 1 °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.

11.1.1 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 Test Method D5191 ILS.⁵ As stated in 8.2.1, P_{tot} used in Test Method D5191 is equivalent to VPCR4-F(37.8 °C) used in this test method.

11.2 For the purpose of this field method, an acceptable tolerance value, based on the method precision, 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 2 indicate that the instrument is performing

TABLE 2 Acceptable Testing Range for Air Saturated Reference Fluids

Reference Fluid	ARV [P_{tot}] ± Acceptable Tolerance, (kPa) @ V/L 4:1 (37.8 °C)	Acceptable Testing Range for Reference Material [P_{tot}], (kPa)
Pentane	112.8 ± 8.1	112.8 ± 8.1 (104.7 to 120.9)
2,2 Dimethylbutane	74.1 ± 8.1	74.1 ± 8.1 (66.0 to 82.2)
2,3 Dimethylbutane	57.1 ± 8.1	57.1 ± 8.1 (49.0 to 65.2)

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). (Note that 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) may be checked periodically by performing this test method on previously prepared samples from one batch of product, as per procedure described in Section 12. Samples should be stored in an environment suitable for long-term

storage without sample degradation. Analysis of result(s) from these quality control samples may be carried out using control chart techniques.⁶⁾

12. Procedure

12.1 General Requirements:

12.1.1 Fig. 4 illustrates the basic steps required to obtain a sample for expansion to a 4:1 V/L ratio. Use of other combinations of volumetric guides will produce different V/L ratios depending on the desired application. Table 3 includes the most common guide combinations that could be used with the recommended guides.

⁶ MNL 7, Manual on Presentation of Data Control Chart Analysis, "Section 3: Control Charts for Individuals," 6th edition, ASTM International, W. Conshohocken, PA.

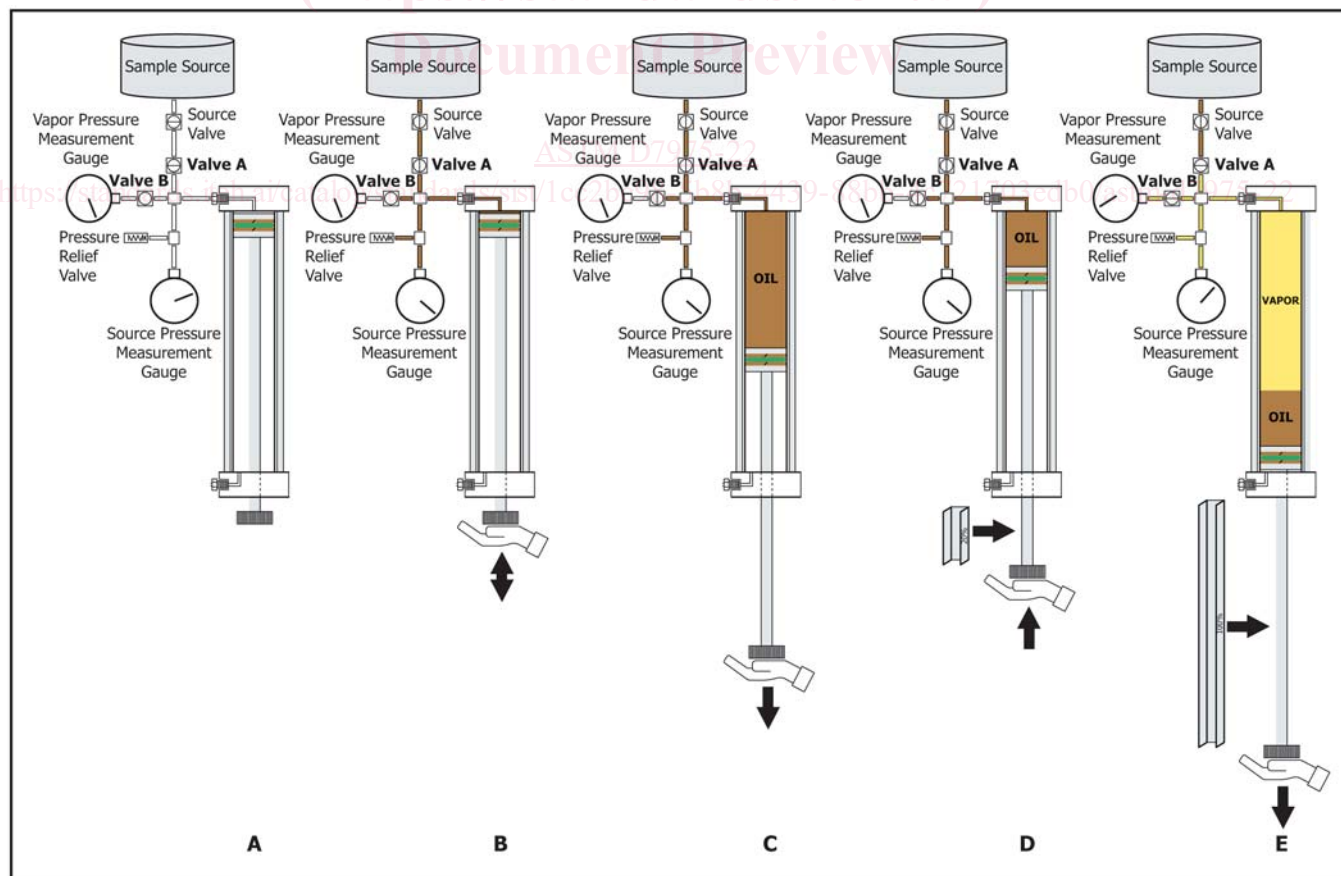


FIG. 4 Sampling Procedure (4:1 V/L Ratio)