



Designation: D8236 – 18

Standard Practice for Preparing an Equilibrium Liquid/Vapor Sample of Live Crude Oil, Condensates, or Liquid Petroleum Products Using a Manual Piston Cylinder for Subsequent Liquid Analysis or Gas Analysis¹

This standard is issued under the fixed designation D8236; 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 practice covers the preparation of an equilibrium gas sample of live crude oil, condensate, or liquid petroleum products, using a Practice D8009 manual piston cylinder (MPC) as a vapor tight expansion chamber to generate an equilibrium vapor/liquid pair at a known temperature and vapor/liquid ratio (V/L). Inert gas such as helium or argon is injected to the equilibrium vapor space of the MPC to provide an equilibrium vapor sample sufficiently above atmospheric pressure for subsequent analysis using a standard refinery gas analyzer (RGA) such as described in Test Method D7833. Other gas analysis methods may be used provided they meet the minimum performance criteria stated in 7.4.1.

1.2 This practice is suitable for UN Class 3 Liquid samples having vapor pressures between 0 kPa and 300 kPa at 50.0 °C, and 0.1:1 to 4:1 vapor/liquid ratio, spanning the nominal range near bubble point (Test Method D6377 VPCr,0.1) to Test Methods D323 (RVP), D4953, and D5191 (V/L=4). The temperature may vary over a wide range, provided that the cylinder is maintained at isothermal and isobaric conditions to prevent condensation of equilibrium vapor upon cooling either in the cylinder or in the injection system of the Refinery Gas Analyzer (RGA, Test Method D7833). The method is best suited for preparation of an equilibrium gas/liquid pair near ambient conditions, typical of routine daily operations in a typical refinery quality assurance or marine terminal laboratory, to routinely monitor the light ends content of crude oil receipts.

1.3 This practice is suitable to prepare an equilibrium liquid/vapor sample pair in a sealed sampling system (no light ends loss from either phase). The equilibrium gas phase is suitable for subsequent gas analysis of both hydrocarbon and fixed/inert gases in the sample, including: hydrogen, oxygen,

nitrogen, carbon dioxide, carbon monoxide, hydrogen sulfide, C1 to C7 hydrocarbons at levels consistent with the Test Method D7833 method used. The equilibrium liquid phase can be subsequently analyzed by Test Method D8003 to obtain paired analytical results on both the equilibrium liquid and vapor pair with a sealed sample system.

1.4 Addition of the diluent gas provides a positive pressure sample to allow the use of a typical RGA-type gas injection system that operates only slightly above barometric pressure. The preferred diluent gas shall be the same as the carrier gas used in the RGA (typically helium or argon). Choice of diluent or carrier gas may affect the ability to detect some inert gases (especially O₂ or H₂) in some RGA configurations conforming to Test Method D7833.

1.5 The VLE gas generation and subsequent RGA output is used as a screening method to identify gas components that can be present in the crude oil affecting the total vapor pressure. The RGA output only represents the equilibrium vapor components present and relative to one another. Due to dilution of the VLE gas with inert gas, the RGA output does not purport to accurately provide the actual vapor composition at VLE conditions and is definitely not representative of the composition of the whole sample.

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.*

1.8 *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.*

¹ This practice 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. Referenced Documents

2.1 ASTM Standards:²

- D323** Test Method for Vapor Pressure of Petroleum Products (Reid Method)
- D4057** Practice for Manual Sampling of Petroleum and Petroleum Products
- D4953** Test Method for Vapor Pressure of Gasoline and Gasoline-Oxygenate Blends (Dry Method)
- D5191** Test Method for Vapor Pressure of Petroleum Products and Liquid Fuels (Mini Method)
- D6377** Test Method for Determination of Vapor Pressure of Crude Oil: VPCR_x (Expansion Method)
- D7833** Test Method for Determination of Hydrocarbons and Non-Hydrocarbon Gases in Gaseous Mixtures by Gas Chromatography
- D7975** Test Method for Determination of Vapor Pressure of Crude Oil: VPCR_x-F(Tm°C) (Manual Expansion Field Method)
- D8003** Test Method for Determination of Light Hydrocarbons and Cut Point Intervals in Live Crude Oils and Condensates by Gas Chromatography
- D8009** Practice for Manual Piston Cylinder Sampling for Volatile Crude Oils, Condensates, and Liquid Petroleum Products

3. Terminology

3.1 Definitions:

3.1.1 *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.

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

3.1.1.2 *Discussion*—A crude oil is normally considered “live” until the vapor pressure can be established using Test Method **D6377** or **D7975**. Sampling and handling of dead crude oils can usually be performed without concern in open, non-pressurized sample containers, such as cans, bottles, and other atmospheric containers as described in Practice **D4057**.

3.1.2 *dead volume, n*—the dead volume includes any incidental volume from fittings, valves, threaded port connections, the annular volume around the piston, channel volume within the end caps, and volume within the pressure relief device and valves that can affect the actual V/L versus V/L calculated from the linear displacement of the cylinder rod used to calculate the volume of the cylindrical sample chamber.

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

3.1.3.1 *Discussion*—Sampling and handling of samples of live crude oils will necessitate the use of the closed sample container to maintain sample integrity and preclude the use of

open sample containers, such as cans, bottles, and other atmospheric containers.

3.1.3.2 *Discussion*—Samples and bulk storage (tank) liquids may or may not appear to boil visibly (rolling) but vaporization (off-gassing) is occurring.

3.1.4 *manual piston cylinder (MPC), n*—a Practice **D8009** vapor and liquid tight (“sealed”) pressurized sample cylinder container with an internal piston that effectively divides the container into two separate compartments and that is attached to a rod which allows the user to manually move the piston in order to collect volatile liquid samples at low sample point pressures.

3.1.5 *single-phase fluid, n*—a fluid (liquid or gas) that has no separate vapor and liquid phases.

3.1.6 *vapor liquid ratio, n*—the volume of the vapor space formed above a liquid sample in a piston sampling cylinder or test apparatus divided by the original sample volume (not corrected for the small volume change associated with generating the vapor phase).

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *combustion gas, n*—gas generated as a result of hydrocarbon combustion.

3.2.1.1 *Discussion*—Commonly used as pad gas in tank ship cargo tanks.

3.2.2 *isobaric, adj*—having a constant pressure throughout.

3.2.3 *isothermal, adj*—having a constant temperature throughout.

3.2.4 *pad gas, n*—gas used to prevent the ingress of atmospheric gases (predominately O₂, N₂) into a tank, or storage container/vessel as well as prevent explosive gas mixtures from forming in the vapor space above a flammable liquid, or a gas used to pressurize a tank temporarily to facilitate a liquid transfer or maintain pump suction pressure.

3.2.4.1 *Discussion*—Commonly used pad gases include but are not limited to: nitrogen, carbon dioxide, and methane.

3.3 Abbreviations:

3.3.1 *CV*—charge valve

3.3.2 *FPC*—floating piston cylinder

3.3.3 *MPC*—manual piston cylinder

3.3.4 *psia*—pounds per square inch absolute (psia = psig + barometric pressure)

3.3.5 *psig*—pounds per square inch gauge (psig = psia – barometric pressure)

3.3.6 *PRV*—pressure relief valve

3.3.7 *PV*—purge valve

3.3.8 *RGA*—refinery gas analyzer

3.3.9 *SV*—sample valve

3.3.10 *VLE*—vapor-liquid equilibrium

3.3.11 *V/L*—vapor/liquid volume ratio

4. Summary of Practice

4.1 A live crude oil or condensate sample is obtained using an MPC, as described in Practice **D8009**, to avoid loss of any light hydrocarbon or dissolved gases. After any other desired

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.

tests are completed, the remaining single-phase sample volume within the MPC is reduced to 20 % of MPC volume by iso-barically purging the excess sample. The piston is then extended to 100 % by injecting an inert gas such as helium or argon, at nominally 200 kPa (14.3 psig), creating a 4:1 vapor/liquid ratio.

4.2 Other V/Ls or inert gas pressures can be used, but V/L=4 and 200 kPa will ensure that the majority of the dissolved gases such as oxygen, nitrogen, and CO₂ will be in the vapor phase at sufficiently low pressure that the system will behave ideally, and that a sufficient volume of vapor is available to properly flush the GC inlet system, and that the liquid will not release or absorb large quantities of gas with minor variations of temperature or pressure.

4.3 The cylinder is agitated for 2 min to ensure a close approach to equilibrium.

4.4 After the equilibration period the cylinder is oriented vertically vapor side up and sample valve connection point to the sample chamber at the highest point, to allow the liquid to settle to the bottom of the sample chamber. The equilibrium vapor phase is then transferred to a refinery gas analyzer to determine the equilibrium gas composition.

4.4.1 This practice is intended for use in screening the bulk of gaseous components present in the equilibrium vapor. This practice is not intended to measure or report trace quantities (<0.1 % by mass) although some RGA test methods may have lower quantitation limits for some or all components listed.

4.5 After transferring and testing the vapor phase with the RGA the cylinder may be oriented with the sampling valve facing vertically downward to obtain an equilibrium liquid sample for other analysis not included in this practice.

4.6 Isobaric operation during sample withdrawal can be obtained by applying a constant pressure to the handle (pre-charge) side of the MPC. The pre-charge side of the cylinder may also be pressurized with inert gas if the user is unable to put sufficient force on the piston handle to maintain the pressure.

4.6.1 For Test Method D8003 or other similar methods, the liquid phase must be transferred into a separate small cylinder that meets the minimum 300 psig injection pressure requirements for Test Method D8003, or an inert gas sampling bag for gaseous GC injection, provided that all connections are evacuated prior to transfer.

4.6.2 It is recommended that for direct connection of the MPC to the RGA, vapor transfers be through a small glass impinge (liquid knock-out) or coil of small internal diameter PTFE tubing, or combination thereof, so that any inadvertent entrainment of crude oil liquid is visible to the operator sufficiently quickly that the sample loop flow can be stopped before the RGA inlet is contaminated by liquid crude oil. All transfer lines must be evacuated or sufficiently flushed to ensure that a representative sample is injected into the GC.

4.6.3 It is recommended that the 20 % cylinder rod spacer be put in place during vapor transfers from the cylinder as an additional precaution against inadvertent liquid crude transfer.

5. Significance and Use

5.1 This practice allows for compositional analysis of the gases in equilibrium with crude oil, condensate, and liquid petroleum products at a 4:1 vapor/liquid ratio at ambient temperature for analysis using typical instrumentation (RGA) already available in typical refinery laboratories. These highly volatile components can result in vapor pressure conditions above atmospheric pressure, so this mechanically simple system is easily adaptable to day-to-day application at low cost/effort using existing analytical equipment.

5.2 This practice allows for compositional analysis and day-to-day tracking or trending of the light hydrocarbons in crude oil for the purpose of identifying unusual blending of NGL, LPG, butane etc. into individual crude oil batch receipts.

5.3 This practice allows identification of gases: including: CO, CO₂, H₂, H₂S, N₂, O₂, CH₄, C₂H₆, C₃H₈, etc. that can contribute to vapor pressure by Test Method D6377, but are not identified using Test Method D8003 (see Note 1). These components can originate from production or can be the result of the use of pad gas and may not be native to the original product. Significant difference in Test Method D6377 vapor pressure measurements at low V/L (for example, 0.1:1) versus high V/L (for example, 4:1) indicate the contribution of high vapor pressure gases such as those in 5.2.

NOTE 1—Test Method D8003 does identify: CH₄, C₂H₆, and C₃H₈. Test Method D8003 does not identify: CO, CO₂, H₂, H₂S, N₂, and O₂.

5.4 Nitrogen and combustion gases (mostly nitrogen and CO₂ with minor concentrations of air) at positive pressures up to 2500 mm water column (nominal 4 psig) is required by International Marine Organization (IMO) Marine Pollution (MARPOL) and Safety of Life at Sea (SOLAS) regulations for the marine transport of crude oil. Analysis of the equilibrium vapor may be required to determine the contribution of inert gases to the total vapor pressure of the crude oil on receipt at the discharge port or refinery.

6. Interferences

6.1 Interference in a sampling procedure is anything that compromises the integrity of the sample.

6.2 Incorrect choice of a sample point location can result in a non-representative sample due to solid or liquid contaminants, separate phases, storage tank stratification, etc.

6.3 Reactivity of steel surfaces can result in the chemical alteration of trace reactive components such as H₂S, COS, and mercaptans.

6.4 A lubricant, used on the piston or other internal wetted parts, that is soluble in hydrocarbon can contaminate the sample and analytical equipment.

6.5 Leakage can result in loss of sample. Consult the manufacturer's guidelines for suitable procedures to verify a leak-free cylinder, such as vacuum or pressure testing.

6.6 Failure to flush sample lines and dead volumes can result in contaminated and non-representative samples that cause a high bias when vapor pressure is measured at V/L < 1.

6.7 Fully air-saturated samples will report barometric pressure at the time at V/L approaching 0.

6.8 Sampling from stratified tanks, dead zones in flowing systems, or inappropriate time periods can result in non-representative samples.

6.9 Any material that can create carryover contamination from one sample to the next shall be removed from the cylinder, and the cylinder thoroughly cleaned before collection of subsequent samples. In addition to cleaning the interior metal surfaces and cleaning the soft parts (O-rings, for example), consideration should be given to replacing the soft parts if they might have absorbed any contamination. Examples of contaminants include glycol, amine, lubricants, sulfur species, solvents, methanol, etc.

7. Apparatus

7.1 *Manual Piston Cylinder (MPC) as described in Practice D8009:*

7.1.1 *Construction*, typically fabricated from corrosion-resistant material such as 316 stainless steel or aluminum. Protective internal coatings or surface treatments are acceptable provided that they do not adversely affect the free movement of the piston, or effectiveness of the seals (see Fig. 1).

7.1.2 The cylinder shall have provision for moving the piston, both in and out, by means of a rod connected directly to the piston. In some instances an FPC may be equipped with a mixing rod that can be fixed to the piston to meet the movement criteria and therefore such an FPC may also be used as an MPC.

7.1.3 *Piston Position Indicator*—The MPC shall be equipped with a piston position indicator such as a marking on the piston rod or equivalent mechanism that indicates the sample volume. A volumetric guide inserted over the piston rod may also be used (see Fig. 2).

7.1.3.1 *Volumetric Fill Guide*—If used, shall be made of brass, aluminum, or other suitable material that will perform without deforming over time or damage the piston rod. Guides shall be “C-Channel” type to allow insertion over the piston rod (see Fig. 2). Dimension A will determine the volume and will be dependent on the piston stroke length and the required sample volume. Multiple guides may be cut to provide varying volume requirements. Dimension B (internal diameter) shall be slightly greater than the piston rod diameter to allow the guide to be inserted easily. Dimension D shall be slightly greater than

the piston rod diameter plus the material thickness. For example: an 80 % guide length is based on 80 % of the length of the piston stroke. A cylinder with a 20.3 cm (nominal 8 in.) piston stroke length will have a maximum 16.2 cm (nominal 6.4 in.) length guide.

7.1.3.2 Volumetric guides for 20 %, 40 %, and 100 % are required for this test method.

7.1.3.3 Manual piston cylinders that are not equipped with a piston position indicator shall not be used.

7.2 *Transfer lines, valves, pressure gauges and related equipment* in the transfer system shall be corrosion resistant (typically stainless steel) and designed consistent with maximum anticipated pressure. The equilibrium gas transfer lines should be as short as practical to prevent contamination with atmospheric air by preventing appropriate equilibrium gas purging or evacuation prior to analysis.

NOTE 2—While not required by this practice, the use of non-reactive and non-absorptive materials is recommended, especially when sampling to determine trace levels of reactive or polar materials such as H₂S and water.

7.3 *Vapor transfer line* 3.175 mm (1/8 in.) PTFE (Teflon™) translucent tubing or equivalent with a pressure rating exceeding 400 kPa, or glass impinger type liquid trap with sufficient pressure rating for the GC inlet system, or combination thereof.

7.4 *Refinery Gas Analyzer.*

7.4.1 *Performance Criteria*—Table 1 lists the minimum acceptable performance criteria.

8. Reagents and Materials

8.1 *Inert Gases:*

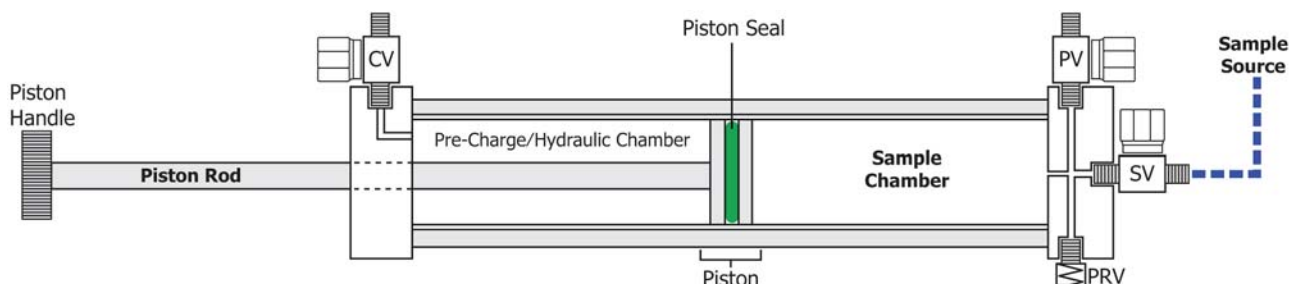
8.1.1 Helium, 99.999 %. (**Warning**—Compressed gas under high pressure.) This gas should not contain more than 5 mL/m³ of O₂.

8.1.2 Argon, 99.999 %. (**Warning**—Compressed gas under high pressure.) This gas should not contain more than 5 mL/m³ of O₂.

8.2 *Seal Lubricants:*

8.2.1 Lubricants used to lubricate or seal the piston, O-ring seals, and other components shall be inert and low soluble in crude oil or condensates.

8.2.2 PTFE lubricants have been found to be suitable in manual piston cylinders for most applications. These lubricants are insoluble in aliphatic/aromatic hydrocarbons, water, caustic, amines, and glycols. Use of excessive lubricant on the



NOTE 1—Example, location and orientation of valves and fittings may vary by vendor.

FIG. 1 Manual Piston Cylinder Schematic

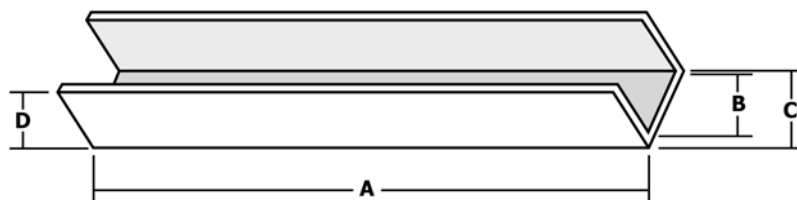


FIG. 2 Volumetric Fill Guide (Example)

TABLE 1 RGA Performance Criteria

Component	Limit of Quantitation % by mass
Carbon Monoxide	0.1
Carbon Dioxide	0.1
Hydrogen	0.1
Hydrogen sulfide	0.1
Nitrogen	0.1
Oxygen	0.1
C1-C4 Hydrocarbons	0.1

sample chamber side of the piston seal can result in contamination of the sample, which can lead to contamination of analytical instruments with PTFE lubricant that cannot be cleaned by solvent flushing alone. Excess lubricant may be used in the pre-charge/hydraulic chamber only to replace lubricant lost by wall coating during piston movement.

8.2.3 Some common grades of silicone-based O-ring lubricants are quickly removed by aromatic hydrocarbons and crude oils, and are not recommended for this service. If used, frequent re-lubrication will be required to maintain seal integrity.

NOTE 3—The use of lubricants that are soluble in hydrocarbon samples will result in contamination of the sample and loss of sealing integrity of the piston, requiring frequent re-lubrication.

9. Procedure

9.1 Preparation of the Manual Piston Cylinder:

9.1.1 Thoroughly clean the cylinder prior to use or after change of service or repair, with an appropriate cleaning agent, following the manufacturer’s recommendations. Remove any traces of cleaning agent by evacuation, inert gas purge, or solvent wash, as appropriate. The use of steam is not recommended for cleaning piston-type cylinders.

NOTE 4—Residual hydrocarbon-based cleaning agents, such as toluene and mineral spirits, can appear in compositional analysis.

9.2 Disassembly of Manual Piston Cylinders:

9.2.1 Consult the manufacturer’s instructions. (**Warning—**Disassembly of a piston cylinder for maintenance requires special precautions. User shall ensure both sample and pre-charge/hydraulic chambers are opened to the atmosphere to relieve any residual pressure prior to removing either end cap. Failure to do so could result in ejection of the piston with sufficient force to cause serious injury to personnel and damage to equipment.)

9.2.2 User shall lubricate the piston to ensure piston seal effectiveness. To ensure the piston is thoroughly lubricated, the pre-charge/hydraulic chamber dead volume (volume remaining

in the pre-charge/hydraulic chamber when the piston rod is fully extended) may be filled with lubricant (several milliliters may be required).

9.3 Sample Collection and Conditioning:

9.3.1 Collect samples in accordance with Practice D8009.

9.3.1.1 *Volume of Sample*—The minimum volume of sample for collection should be determined by the combined volumes required by each of the tests to be performed, typically 400 mL (that is, 80 % of a 500 mL sample cylinder at 15 °C).

9.3.2 Transport the samples to the laboratory.

9.3.3 Allow the cylinder to come to room temperature.

9.3.4 Compress the piston inward and shake the cylinder contents for 30 s to 60 s to agitate the sample to homogenize and allow any free vapor to re-dissolve into the liquid to re-establish the sample as single-phase. Exceeding the sample point pressure by 20 % or more, coupled with agitation, will accelerate re-dissolution of gases into the liquid phase.

9.3.4.1 If the user is unable to compress the piston manually, an external compressed inert gas source, such as compressed argon, helium, or nitrogen, may be used on the pre-charge/hydraulic chamber to compress the piston. The compressed gas source shall not exceed the pressure relief valve (PRV) set point pressure and maximum working pressure of the MPC. If available, the use of compressed gas is preferable to ensure constant pressure during sample conditioning and handling.

9.3.5 Verify the sample is single phase by confirming the piston will not compress further. Free vapor will allow the piston to compress greater than 1 mm to 3 mm but single-phase liquid will not allow compression. If free vapor is detected, repeat 9.3.4 until the piston will no longer compress.

9.3.6 Continue to provide compression on the piston manually or with the use of compressed gas throughout the sample transfer procedure(s) to maintain the sample under single-phase conditions. If the compression is released at any time during sample transfer, immediately stop transferring and repeat 9.3.4.

9.4 *Procedure A—Sample Expansion Without Pre-Charge Gas Available*—This procedure is applicable to samples that have sufficiently low pressure to allow the manual movement of the piston by the user during sample handling. If the sample pressure is high enough that a user is unable to control the movement of the piston manually then Procedure B should be used.

NOTE 5—This procedure is destructive to the sample. If other testing is required on the collected sample, it should be performed prior to beginning 9.4 as no additional testing can be performed after.

9.4.1 Position the MPC with the sample chamber in a vertical (upright) position and ensure all valves are closed. See Fig. 3-A.

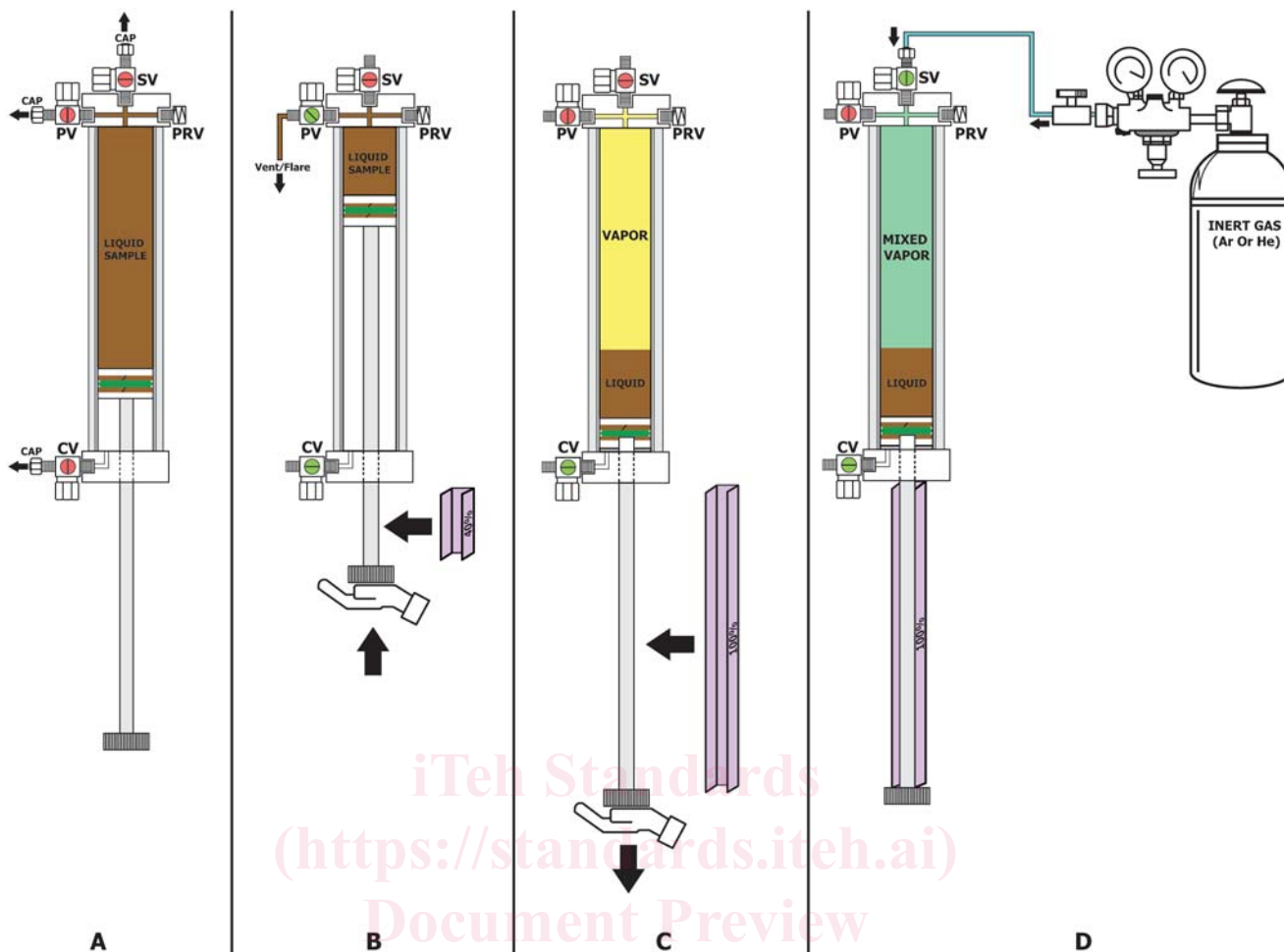


FIG. 3 Vapor Liquid Equilibrium Gas Preparation (no pre-charge gas)

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9.4.2 Remove the caps from valves SV, PV, and CV. See Fig. 3-A.

9.4.3 While holding the piston in place, open the pre-charge valve (CV). See Fig. 3-B.

9.4.4 While manually compressing the piston to maintain the sample in a single-phase condition during purge operations, slowly open valve (PV) to purge sample fluid until the 20 % guide fits on the piston rod with the base of the handle pressed firmly to the bottom of the guide, then close purge valve (PV). See Fig. 3-B. Other means of identifying the percentage fill may be supplied by the manufacturer. In that event, follow manufacturer instructions.

9.4.5 Slowly release the piston handle and allow the piston to move freely.

NOTE 6—The cylinder is now 20 % full of liquid, allowing the remaining 80 % for expansion volume. The piston may or may not extend fully on its own unless sufficient vapor pressure exists to fill the remaining volume with vapor.

9.4.6 Extend the piston by pulling on the piston handle until the 100 % guide fits on the piston rod with the base of the handle pressed firmly to the bottom of the guide. See Fig. 3-C. The sample is now expanded to a 4:1 vapor/liquid ratio.

9.4.7 Loosely connect the sample valve (SV) to the inert gas source using 3.175 mm (1/8 in.) PTFE tubing. The inert gas

source outlet shall be regulated to 200 kPa ± 10 kPa (14.3 psig nominal). The inert gas type and grade shall match the type and grade of carrier gas used in the refinery gas analyzer (RGA). See Fig. 3-D.

9.4.8 Open the inert gas source and allow gas to purge through the loosely connected tube fitting on the sample valve (SV). Allow gas to purge through the tubing and fitting for 5 s to 10 s to ensure the tubing from the inert gas source is sufficiently purged with inert gas prior to connecting to the sample valve (SV) to avoid contaminating the sample with atmospheric gases. Tighten the tube fitting connected to the sample valve (SV) and leak test the connection.

9.4.9 Open sample valve (SV) to allow the inert gas to enter the MPC sample chamber and allow 5 s for the pressure to stabilize.

9.4.10 Close sample valve (SV).

9.4.11 Close the inert gas source.

9.4.12 Slowly disconnect the tubing from the sample valve (SV) and allow the pressure to dissipate before completely disconnecting.

9.4.13 By hand, manually invert the MPC from upright to nominally ±180° approximately 30 cycles per minute for 2 min. See Fig. 4-A.