Designation: D8009 – 15

Manual of Petroleum Measurement Standards (MPMS), Chapter 8.5

Standard Practice for Manual Piston Cylinder Sampling for Volatile Crude Oils, Condensates, and Liquid Petroleum Products[1]

This standard is issued under the fixed designation D8009; 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 practice includes the equipment and procedures for obtaining a representative sample of "live" or high vapor pressure crude oils, condensates, and/or liquid petroleum products from low pressure sample points, where there is insufficient sample point pressure to use a Floating Piston Cylinder (FPC) as described in Practice D3700.

1.2 This practice is intended for use with sample types, such as UN Class 3 Flammable Liquids, that might have been collected and transported using open containers. The use of a manual piston cylinder in place of open containers is intended to prevent the loss of volatile (light end) components, which can impact subsequent test results.

1.3 This practice is suitable for sampling crude oils, condensates, and/or liquid petroleum products having true vapor pressures less than 300 kPa (43 psia nominal) at 50 °C. This practice applies to samples that will typically fall between Practices D4057 (API *MPMS* Chapter 8.1) and D3700. This practice shall not be used for materials classified as UN Class 2 Gases[2] ("…having a vapor pressure greater than 300 kPa at 50 °C or is completely gaseous at 20 °C at 101.3 kPa.").

1.4 This practice allows for sampling of crude oils that flow freely at the conditions of sampling.

1.5 It is the responsibility of the user to ensure that the sampling point is located so as to obtain a representative sample.

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

1.6.1 *Exception*—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.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

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

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

D6378 Test Method for Determination of Vapor Pressure (VP_X) of Petroleum Products, Hydrocarbons, and Hydrocarbon-Oxygenate Mixtures (Triple Expansion Method)

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

2.2 *API Standards:*[4]

MPMS Chapter 8.1 Manual Sampling of Petroleum and Petroleum Products

MPMS Chapter 8.2 Automatic Sampling of Petroleum and Petroleum Products

3. Terminology

3.1 *Definitions:*

[1] This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and the API Committee on Petroleum Measurement, and is the direct responsibility of Subcommittee D02.02 /COMQ the joint ASTM-API Committee on Hydrocarbon Measurement for Custody Transfer (Joint ASTM-API). This practice has been approved by the sponsoring committees and accepted by the Cooperating Societies in accordance with established procedures. This practice was issued as a joint ASTM-API standard in 2015.

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[2] UN Recommendations of the Transportation of Dangerous Goods, Chapter 2.2.1.1.

[3] 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.

[4] Available from American Petroleum Institute (API), 1220 L. St., NW, Washington, DC 20005-4070, http://www.api.org.

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 Methods D6377, D6378, 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 (API *MPMS* Chapter 8.1).

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

3.1.2.1 *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.

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

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

3.1.3.1 *Discussion*—This includes any materials that have atmospheric boiling points below 0 °C including methane, ethane, propane, butane.

3.1.3.2 *Discussion*—Fixed gases, such as CO, CO_2, H_2, H_2S, N_2, and O_2, will also contribute to the composition and vapor pressure of the sample.

3.1.4 *maximum fill volume (reduced fill volume), n*—the volume of a container occupied by the sample, usually expressed as a percentage of the total capacity.

3.1.4.1 *Discussion*—Some regulatory agencies use the expressions "maximum fill density" and "reduced fill density."

3.1.5 *open container, n*—a container designed for use with samples at atmospheric pressure conditions.

3.1.5.1 *Discussion*—This includes glass and plastic bottles. These containers are not suitable for samples expected to have vapor pressures above atmospheric pressure.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *dead volume, n*—the fixed volume required to fill the void spaces in the manual piston cylinder when the piston is pushed firmly against the sample chamber end cap.

3.2.1.1 *Discussion*—The dead volume includes the annular volume around the piston, channel volume within the end caps, and volume within the pressure relief device and valves.

3.2.2 *manual piston cylinder (MPC), n*—a pressurized sample 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.

3.2.2.1 *Discussion*—A manual piston cylinder (Fig. 1) is used to collect a sample of liquid with a vapor pressure of less than 300 kPa (43 psia nominal), without the formation of a gaseous phase, which can result in changes in the composition of the liquid sample.

3.2.3 *single-phase fluid, n*—a liquid that has no separate vapor and liquid phases.

3.2.4 *true vapor pressure (TVP), n*—the total pressure generated by at fluid at a 0:1 vapor:liquid ratio at 50 °C.

3.2.4.1 *Discussion*—50 °C is the prescribed temperature for vapor pressure for distinguishing between UN Class 2 Gases and Class 3 Flammable Liquids.

3.2.4.2 *Discussion*—True vapor pressure is the sum of the partial pressures of all the components within a fluid including dissolved fixed gases such as CO, CO_2, H_2, H_2S, N_2, and O_2.

3.2.4.3 *Discussion*—True vapor pressure is equivalent to the bubble point pressure at a prescribed temperature. Fluids above their bubble point pressure are also referred to as single-phase fluids.

3.3 *Abbreviations:*

3.3.1 *BPR*—back pressure regulator

3.3.2 *CVC*—constant volume cylinder

3.3.3 *CV*—charge valve

3.3.4 *FPC*—floating piston cylinder

3.3.5 *MPC*—manual piston cylinder

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

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

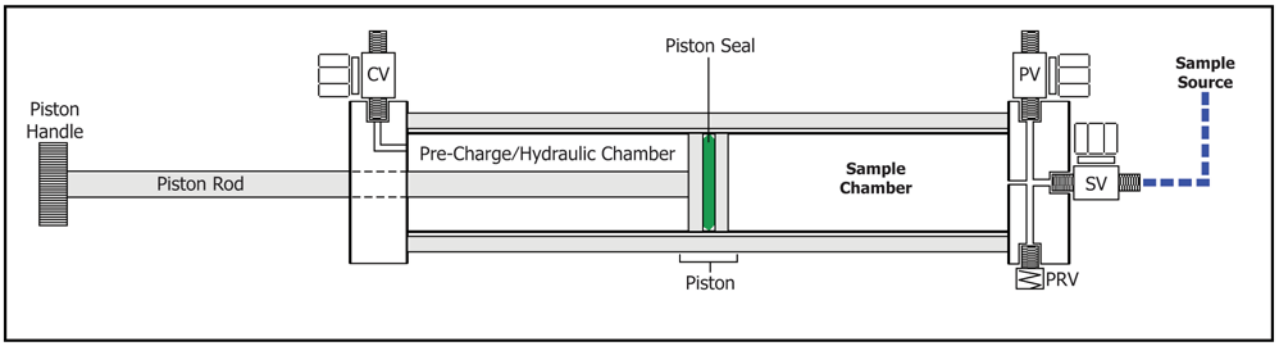

FIG. 1 Manual Piston Cylinder Schematic (Example)

3.3.8 *PRV*—pressure relief valve

3.3.9 *PSV*—pressure safety valve

3.3.10 *PV*—purge valve

3.3.11 *PTFE* —polytetrafluoroethylene

3.3.12 *SV*—sampling valve

3.3.13 *TVP*—True Vapor Pressure (0:1 vapor/liquid ratio at 50 °C)

4. Summary of Practice

4.1 A crude oil or condensate sample is transferred as a single-phase liquid under pressure from a sample point into a manual piston cylinder. The manual piston cylinder (MPC) is designed to collect liquid samples with no vaporization or loss of volatile components by displacing a piston against the mechanical backpressure of the user. The piston serves as a physical barrier between the sample and the atmosphere. The manual movement of the piston allows the user to pull sample into the cylinder as well as compress the sample for injection into an instrument for analysis. The position of the piston at the end of sampling indicates the percent fill of the sample cylinder.

4.2 It is the responsibility of the user of this practice to locate the sample point at a suitable location and orientation where the product being sampled is a representative, single phase, homogeneous liquid.

5. Significance and Use

5.1 This practice allows the collection of a representative sample of crude oil and/or condensate that may contain trace volatile dissolved components such as methane, ethane, propane, and fixed gases that would normally be lost using conventional atmospheric sampling methods. These highly volatile components can result in vapor pressure conditions above atmospheric pressure. This practice is recommended whenever accurate determination of vapor pressure, flash point, or other properties are required and where loss of volatile components can affect the test results.

5.2 This practice is intended for capturing samples of crude oil and/or condensate for testing for the purpose of classification for transportation of dangerous goods as UN Class 3 Flammable Liquids, but is not limited to classification testing. Other test methods with sensitivities to light end loss may also utilize this sampling practice.

5.3 Practice D3700 using a floating piston cylinder is recommended whenever true vapor pressures greater than 300 kPa at 50 °C are anticipated.

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, and so forth.

6.3 Reactivity of steel surfaces can result in the chemical alteration of trace reactive components such as H_2S, 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.

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

6.8 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):*

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 Users should consult with the manufacturer of the MPC and sample collection systems any time ambient or product temperatures, or both, exceed the range of –30 °C to 60 °C (–22°F to 140 °F). Extreme temperature effects upon metal, O-rings, valve seats, seals, gauges, relief devices, sample pump components, and other devices and components in the system should be assessed in a hazards analysis before any sampling takes place.

7.1.3 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.

7.1.4 *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 to comply with the maximum percent fill (maximum fill volume) allowed for storage and transportation. A volumetric guide inserted over the piston rod may also be used (see Fig. 2).

7.1.4.1 *Volumetric Fill Guide*—If used, shall be made of brass, aluminum, or other suitable material that will perform without deforming over time or damaging the piston rod. Guides shall be "C-Channel" type to allow insertion over the

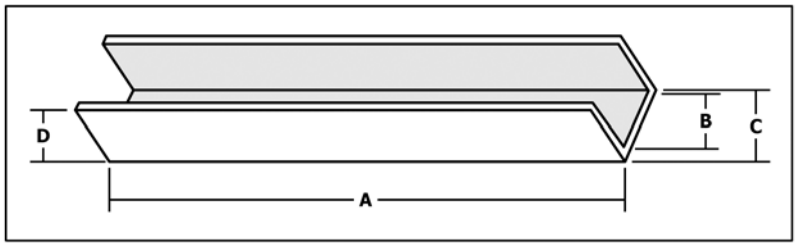

FIG. 2 Volumetric Fill Guide (Example)

piston rod (see Fig. 2). Dimension A will determine the volume and will be dependent on the piston stroke length and the required fill density. 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. Appropriate piston stroke length measurement adjustment is required for reduced filled density.

7.1.4.2 Manual piston cylinders that are not equipped with a piston position indicator shall not be used without a procedure to allow the operator to verify fill volume immediately after sampling and prior to transport. Consult the authority having jurisdiction for acceptable procedures.

7.1.5 The cylinder sample chamber end cap shall have provision for a safety relief device to protect the user from accidental over-pressure by connection to a sample point pressurized beyond the maximum working pressure of the manual piston cylinder, and to prevent over-pressure in the event that a cylinder becomes fully liquid filled (hydraulically locked) from either overfilling or liquid thermal expansion from excessive temperature increase.

7.1.6 A rupture disk or a self-resetting pressure relief valve (PRV) shall be fitted to the cylinder. PRV relief pressure shall be less than the maximum working pressure of the cylinder.

7.1.6.1 In the event that a self-resetting PRV is activated due to an overpressure condition, sample integrity can be compromised without alerting the user that a release has occurred. If a self-resetting PRV is used, a release indicator is recommended to alert the user that the sample has been compromised and to capture a new sample or use testing results with caution.

NOTE 1—Pressure relief valve (PRV) may also be referred to as a pressure safety valve (PSV).

7.1.7 Users should not alter the release pressure of safety relief devices.

7.2 *Vacuum Pump*—Capable of maintaining 13 kPa (100 mm Hg nominal).

7.3 *Sampling System*—It is not possible to provide a single procedure that will be applicable for all sampling situations. Different procedures and fittings may be required for sampling pipes, storage tanks, rail cars, trucks, and smaller storage vessels in order to obtain a representative sample (see 4.2). Refer to Practices D4057 (API *MPMS* Chapter 8.1) and D4177 (API *MPMS* Chapter 8.2) for recommended sample point selection.

7.3.1 Sampling procedures shall be designed and used to obtain representative samples of a product, and to maintain sample integrity for the tests to be performed.

7.3.2 Sampling system shall have a pressure control device to maintain the outlet pressure to the sampling cylinder below manufacturer maximum working pressure and release pressure of the pressure relief device.

7.3.3 Sampling system shall have a pressure gauge to confirm the source pressure does not exceed the pressure relief device set point pressure and maximum working pressure of the cylinder.

7.4 *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 transfer lines should be as short as practical to minimize line blockage or sample vaporization, or both. The use of filters, dryers, needle valves, and so forth are not recommended, unless provisions are made to prevent excessive flow restriction and pressure drop. A "T" junction with a purge valve at the sample connection point is recommended to allow purging of the dead volume at the sampler connection. Flexible hose or tubing with adequate pressure rating may be used.

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 H_2S and water.

7.5 Sampling pumps or other means of controlling pressures higher than the vapor pressure of the sample may be acceptable, and may be used to flush the lines or the cylinder dead volume, or both, if any, prior to sample collection. The cylinder may be partially filled and then emptied prior to collection of the sample as an alternative to venting hydrocarbon to flush lines.

8. Reagents and Materials

8.1 *Seal Lubricants:*

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

8.1.2 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 sample chamber side of the piston seal can result in contamination of the sample, which can lead to contamination of analytical instruments with lubricant. Excess lubricant may be used in the pre-charge/hydraulic chamber only to replace lubricant lost by wall coating during piston movement.

8.1.3 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, 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 millilitres may be required).

9.3 *Sampling Procedure A—Manual Piston Cylinder (MPC) without Pre-Charge Gas*—This procedure is applicable to sample points that have sufficiently low pressure to allow the manual movement of the piston by the user during sampling operations. If the sample point pressure is high enough that a user is unable to control the movement of the piston manually, then Procedure B should be used.

9.3.1 CLOSE valve (SV).

9.3.2 OPEN valve (PV).

9.3.3 OPEN valve (CV) to allow air to move in and out of the pre-charge/hydraulic chamber.

9.3.4 PUSH the piston fully into cylinder to expel any residual air. See Fig. 3-A.

9.3.5 CLOSE valve (PV).

9.3.6 Cylinder Leak Test (Vacuum Test).

9.3.6.1 PULL the piston a far as possible to create a vacuum condition within the sample chamber.

9.3.6.2 Slowly allow the piston to self-retract into the cylinder.

(1) The piston shall self-retract fully into the cylinder with the piston firmly against the sample chamber end cap. This position may be confirmed by gently pushing on the piston to confirm there is no inward movement, indicating the piston is seated against the end cap. This confirms the apparatus is properly sealed and that no high volatility material remains.

(2) If the piston does not self-retract fully, which will be indicated by either the visual position of the piston or the ability to push the piston further into the cylinder, clean the apparatus and repeat 9.3.1 to 9.3.6. Both scenarios indicate a possible leak or that high volatility material remains in the apparatus. If the check fails again, then the apparatus needs to be disassembled to confirm the seals are intact and appropriately lubricated.

9.3.7 CONFIRM the sample source pressure does not exceed the pressure relief valve set point pressure and the maximum working pressure of the MPC.

9.3.8 CLOSE valve (CV).

9.3.9 CONNECT valve (SV) to the source valve using low volume, high-pressure, flexible tubing.

9.3.10 SLOWLY OPEN the source valve to pressurize the tubing to valve (SV).

9.3.11 POSITION the apparatus to ensure the piston is faced away from the user. Make note of the pressure relief device orientation and ensure the PSV outlet is facing away from the user in the event of release.

9.3.12 While holding the piston firmly in place SLOWLY OPEN valve (SV) to pressurize the apparatus and fill the dead volume.

NOTE 5—Use caution as the piston is now under source pressure and can be forced out to its full extension if not handled carefully. DO NOT pressurize the apparatus with the piston handle facing directly towards the operator.

9.3.13 PUSH the piston tightly against the sample chamber end cap.

9.3.14 While still holding the piston firmly in place, SLOWLY OPEN valve (PV) to purge the sample fluid through the cylinder end cap (see Fig. 3-B). PURGE an equivalent of three (3) times the line volume connected to the sample source then CLOSE valve (PV). Line volume can be calculated based on the tubing internal diameter and length of tubing using Eq 1.

9.3.14.1 Purging of fluids shall follow all site-specific and jurisdictional requirements for fluid release. If necessary, connect valve (PV) to a flare line or alternate container for venting/capture of purged material.

$$V = \left[\pi \times \left(\frac{ID}{2} \right)^2 \times L \right] \times 3 \quad (1)$$

where:

V = purge volume (mL),
ID = tubing internal diameter (cm), and
L = length of tubing (cm).

9.3.15 Allow the piston handle to move out SLOWLY using the sample point pressure to drive the piston so air in the pre-charge/hydraulic chamber is compressed until it equals the

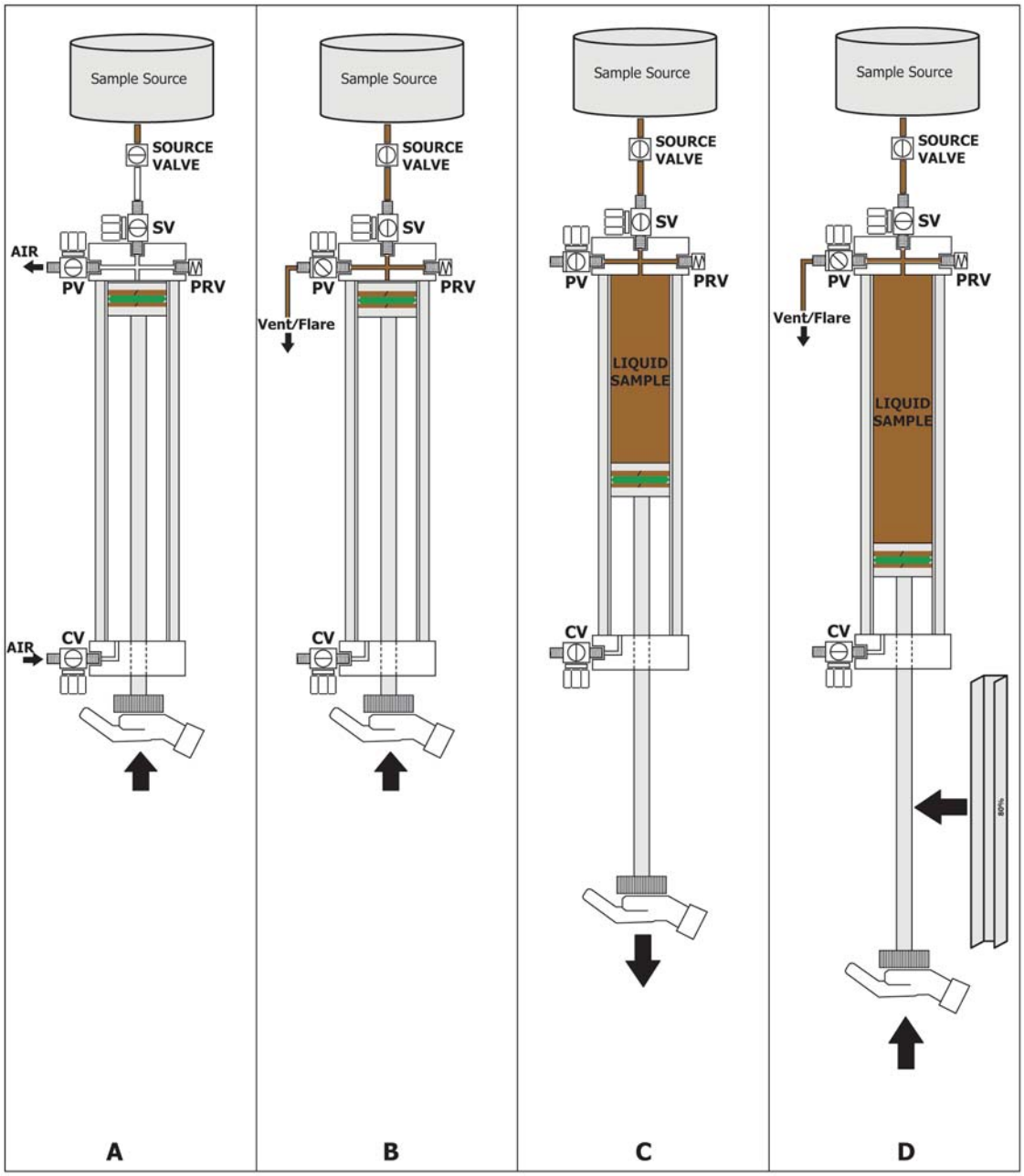

FIG. 3 Sampling Procedure A Images

sample point pressure, at which point the piston will no longer move. Exercise caution not to exceed the fill rate allowed by the size of the lines and valves, resulting in rapid depressurization of the sample and formation of a vapor phase during sampling. See Fig. 3-C.

9.3.15.1 If the desired fill volume has not been reached, SLOWLY OPEN valve (CV) to slowly release the compressed air, creating a differential pressure, which will allow the piston to continue to move until the cylinder is completely full. Proceed to 9.3.16.

9.3.15.2 If the piston will not move freely, OPEN valve (CV) and PULL piston handle back slowly and allow the cylinder to FILL completely. Proceed to 9.3.16.

NOTE 6—With experience, a user will be able to feel the sample conditions within the cylinder through the piston handle as sampling is occurring and will be able to adjust the fill rate accordingly to avoid depressurization. A handle pulled too quickly will have a tendency to retract if released, indicating a slight vacuum was present. It is recommended that users new to sampling be trained by experienced personnel.

9.3.16 CLOSE valve (SV) and push on the piston handle to confirm liquid fill.

9.3.16.1 If the cylinder is full of single-phase liquid (no vapor present), then no inward handle movement will be possible. Proceed to 9.3.17.

9.3.16.2 If the cylinder has vapor present, inward handle movement will be possible. Return to 9.3.12.

9.3.17 While compressing the piston, SLOWLY OPEN valve (PV) to purge sample fluid until the 80 % 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-D. Other means of identifying the percentage fill may be supplied by the manufacturer. In that event, follow manufacturer instructions. (**Warning—**When filling a manual piston cylinder below approximately –5 °C, the maximum fill density (volume) shall be reduced below 80 % to account for the additional thermal expansion and satisfy regulatory requirements for increased outage or reduced fill density.)

9.3.17.1 *Volume of Sample*—The minimum volume 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).

9.3.17.2 For safe handling of these cylinders under extremes of product or ambient temperatures, or both, the user shall consider the effects of thermal expansion on the volume of product in the cylinder. For example, if a product is sampled at –40 °C (–40 °F), the user shall plan for the cylinder and sample to warm considerably during transport and before analysis is performed in the laboratory. During summer months, the temperature of the cylinder and product could reasonably be expected to rise to as high as 46 °C (115 °F) in hot environments. A cylinder initially filled cold to 80 % of its capacity will, upon warming, be over-pressured and the relief device(s) will activate under these conditions. Hydrocarbon releases of this type are unexpected and dangerous. In such an extreme, but not uncommon case, the cylinder should not be filled more than approximately 60 % of its capacity during the initial fill. Users should review ASTM/IP/GPA volume correction factor calculations, or data from similar samples, or both, to determine the maximum fill for the product and conditions being sampled, but should always leave at least 10 % vapor space after allowing for the worst likely case of thermal expansion.

NOTE 7—Joint ASTM/IP/GPA volumetric temperature correction factors are available as GPA Technical Publication TP-27/API MPMS 11.2.4, and can be used to calculate maximum fill volume at low temperatures.

NOTE 8—The 80 % guide length is based on 80 % of the length of the piston stroke. For example: a cylinder with a 20.3 cm (nominal 8 in) piston stroke length will have a 16.2 cm (nominal 6.4 in) guide. Appropriate piston stroke length measurement adjustment is required for reduced filled density. Follow manufacturer's instructions for fill guide use.

9.3.18 With the volumetric guide held firmly in place, CLOSE valve (SV) to isolate the sample source from the apparatus.

9.3.19 SLOWLY release the piston handle and allow the piston to move freely.

NOTE 9—The cylinder is now 80 % full of liquid, allowing the remaining 20 % for liquid expansion volume. The piston may or may not extend fully unless sufficient vapor pressure exists to fill the remaining volume with vapor, however the piston will extend and retract with fluid expansion and contraction due to temperature changes.

9.3.20 CLOSE charge valve (CV).

9.3.21 CLOSE the source valve.

9.3.22 DISCONNECT valve (SV) from the source tubing and, if required, valve (PV) from the flare line or alternate container.

9.3.23 INSTALL caps on valves (SV), (PV), and (CV).

9.3.24 Cylinder is now prepared for transport with a minimum 20 % expansion volume.

9.3.25 Packaging for transport shall ensure that the piston handle has sufficient space to extend and contract with changes in temperature.

9.4 *Sampling Procedure B—Manual Piston Cylinder (MPC) with Pre-Charge Gas*—This procedure is applicable to sample points with pressures beyond that which would allow the manual movement of the piston by the user during sampling operations. Leak testing and filling with pre-charge gas (9.4.1 to 9.4.10) may be performed prior to bringing the MPC to the sampling location. If leak testing and pre-charge gas filling has already been performed, proceed directly to 9.4.11.

9.4.1 CLOSE valve (PV).

9.4.2 OPEN valve (SV).

9.4.3 OPEN valve (CV) to allow air to move in and out of the pre-charge/hydraulic chamber.

9.4.4 PUSH the piston fully into cylinder to expel any residual air. See Fig. 4-A.

9.4.5 CLOSE valve (SV).

9.4.6 Cylinder Leak Test (Vacuum Test).

9.4.6.1 PULL the piston as far as possible to create a vacuum condition within the sample chamber.

9.4.6.2 Slowly allow the piston to self-retract into the cylinder.

(1) The piston shall self-retract fully into the cylinder with the piston firmly against the sample chamber end cap. This position may be confirmed by gently pushing on the piston to confirm there is no inward movement, indicating the piston is seated against the end cap. This confirms the apparatus is properly sealed and that no high volatility material remains.

(2) If the piston does not self-retract fully, which will be indicated by either the visual position of the piston or the ability to push the piston further into the cylinder, clean the apparatus and repeat 9.4.1 to 9.4.6. Both scenarios indicate a possible leak or that high volatility material remains in the apparatus. If the check fails again, then the apparatus needs to be disassembled to confirm the seals are intact and appropriately lubricated.

9.4.7 CONFIRM the sample source pressure does not exceed the pressure relief valve set point pressure and the maximum working pressure of the MPC.

9.4.8 CONNECT valve (CV) to a compressed gas source at a higher pressure than the sample source pressure (for example, about 10 % higher than the sample source pressure).

9.4.9 OPEN valve (CV) to fill the pre-charge/hydraulic chamber with compressed gas. See Fig. 4-B.

9.4.10 CLOSE valve (CV) and DISCONNECT from compressed gas source.

9.4.11 CONNECT valve (SV) to the source valve using low volume, high-pressure, flexible tubing.

9.4.12 SLOWLY OPEN the source valve to pressurize the tubing to valve (SV).

9.4.13 POSITION the apparatus to ensure the piston is faced away from the user. Make note of the pressure relief device and ensure the outlet is facing away from the user in the event of release.

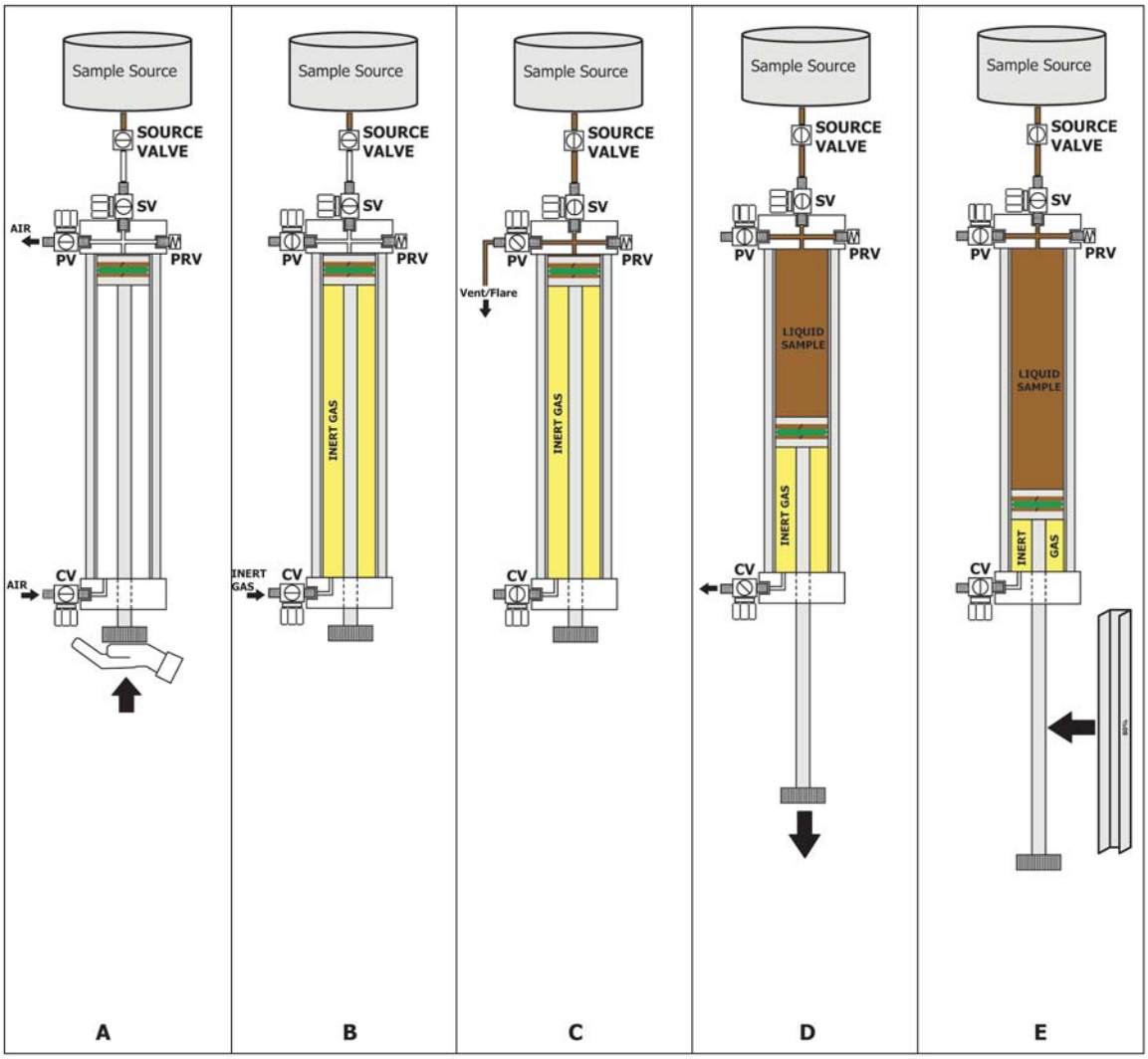

FIG. 4 Sampling Procedure B Images

9.4.14 While holding the piston firmly in place SLOWLY OPEN valve (SV) to pressurize the apparatus and fill the dead volume.

NOTE 10—Use caution as the piston is now under source pressure and can be forced out to its full extension if not handled carefully. DO NOT pressurize the apparatus with the piston handle facing directly towards the operator.

9.4.15 SLOWLY OPEN valve (PV) to purge the sample fluid through the cylinder end cap (see Fig. 4-C). PURGE an equivalent of three (3) times the line volume connected to the sample source then CLOSE valve (PV). Line volume can be calculated based on the tubing internal diameter and length of tubing using Eq 2.

9.4.15.1 Purging of fluids shall follow all site-specific and jurisdictional requirements for fluid release. If necessary, connect valve (PV) to a flare line or alternate container for vent/capture of purged material.

$$V = \left[\pi \times \left(\frac{ID}{2} \right)^2 \times L \right] \times 3 \qquad (2)$$

where:

V = purge volume (mL),
ID = tubing internal diameter (cm), and
L = length of tubing (cm).

9.4.16 SLOWLY OPEN valve (CV) to SLOWLY vent pre-charge gas in order to create a slight differential pressure between the pre-charge and the sample point pressures. This will move the piston, allowing sample to flow into the sample chamber. See Fig. 4-D. Exercise caution not to create too large a differential pressure and exceed the fill rate resulting in rapid depressurization of the sample and formation of a vapor phase during sampling.

NOTE 11—It is recommended that users new to sampling be trained by experienced personnel.

9.4.17 When the cylinder is 80 % full CLOSE valve (CV) and then CLOSE valve (SV). Fill volume may be determined by use of a volumetric fill guide or piston position indicator.

See Fig. 4-E. (**Warning—**When filling a manual piston cylinder below approximately –5 °C, the maximum fill density (volume) shall be reduced below 80 % to account for the additional thermal expansion and satisfy regulatory requirements for increased outage or reduced fill density.)

9.4.17.1 *Volume of Sample*—The minimum volume 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).

9.4.17.2 For safe handling of these cylinders under extremes of product or ambient temperatures, or both, the user shall consider the effects of thermal expansion on the volume of product in the cylinder. For example, if a product is sampled at –40 °C (–40 °F), the user shall plan for the cylinder and sample to warm considerably during transport and before analysis is performed in the laboratory. During summer months, the temperature of the cylinder and product could reasonably be expected to rise to as high as 46 °C (115 °F) in hot environments. A cylinder initially filled cold to 80 % of its capacity will, upon warming, be over-pressured and the relief device(s) will activate under these conditions. Hydrocarbon releases of this type are unexpected and dangerous. In such an extreme but not uncommon case, the cylinder should not be filled more than approximately 60 % of its capacity during the initial fill. Users should review ASTM/IP/GPA volume correction factor calculations, or data from similar samples, or both, to determine the maximum fill for the product and conditions being sampled, but should always leave at least 10 % vapor space after allowing for the worst likely case of thermal expansion.

NOTE 12—Joint ASTM/IP/GPA volumetric temperature correction factors are available as GPA Technical Publication TP-27/API MPMS 11.2.4, and can be used to calculate maximum fill volume at low temperatures.

NOTE 13—The 80 % guide length is based on 80 % of the length of the piston stroke. For example: a cylinder with a 20.3 cm (nominal 8 in) piston stroke length will have a 16.2 cm (nominal 6.4 in) guide. Appropriate piston stroke length measurement adjustment is required for reduced filled density. Follow manufacturer's instructions for fill guide use.

9.4.18 CLOSE the source valve.

9.4.19 DISCONNECT valve (SV) from the source tubing and, if required, valve (PV) from the flare line or alternate container.

9.4.20 INSTALL caps on valves (SV), (PV) and (CV).

9.4.21 Cylinder is now prepared for transport with a minimum 20 % expansion volume.

9.4.22 Packaging for transport shall ensure that the piston handle has sufficient space to extend and contract with changes in temperature.

10. Sample Handling for Analysis Using a Manual Piston Cylinder

10.1 Samples of "live" or high vapor pressure crude oils or condensates captured in a manual piston cylinder require specific handling to ensure the integrity of the sample is maintained for analysis. This section gives general guidance for returning the sample to single-phase source conditions followed by single-phase transfer to either a pressurized instrument or atmospheric test vessel. Sample handling and transfers to additional containers should be minimized to the extent practicable. Each transfer from one vessel to another has the potential to alter the representative nature of the sample. The best practice is transfer directly from the MPC to a pressurized instrument. Appendix X1 provides instruction on transfer between the MPC and other sample containers if required.

10.2 *Sample Conditioning—Procedure A only:*

10.2.1 Allow the cylinder to come to room temperature.

10.2.2 COMPRESS the piston inward and shake the cylinder contents for 30 s to 60 s to agitate the sample to mix 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.

10.2.2.1 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 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.

10.2.3 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 10.2.2 until the piston will no longer compress.

10.2.4 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 anytime during sample transfer, immediately stop transferring and repeat 10.2.2.

10.3 *Sample Transfer for Analysis Procedure:*

10.3.1 *Transfer Directly to a Pressurized Instrument*—It is not possible to consider all instrument configurations and connections. The following is a general instruction meant as a guide for sample transfer. Follow instrument manufacturer instructions for sample injection and loading.

10.3.1.1 CONNECT a flow control valve, such as a needle or ball valve, to the instrument outlet to allow flow control when purging the sample.

10.3.1.2 CLOSE the flow control valve.

10.3.1.3 CONNECT sample cylinder valve (SV) to the inlet of the instrument.

10.3.1.4 While compressing the piston to maintain the sample in a single-phase state, slowly OPEN sample cylinder Valve (SV) to begin filling the transfer line and instrument. When filling is complete OPEN valve (SV) completely to allow full flow.

10.3.1.5 Slowly OPEN the instrument outlet control valve and purge the sample through the instrument until only single-phase (minimal gas bubbles or sputtering) sample appears at the outlet. CLOSE the instrument outlet valve.

10.3.1.6 The instrument is now full of single-phase sample and ready for analysis.

10.3.1.7 Proceed with analysis as per test method and/or manufacturer instructions.

10.3.2 *Transfer to an Atmospheric Test Vessel*—Cooling the manual piston cylinder prior to transfer to an atmospheric test vessel will reduce the vapor pressure and minimize the loss of light end components during the transfer. Refer to the test method being performed for details on sample temperature requirements.

NOTE 14—Samples that contain significant quantities of methane, ethane, propane and/or fixed gases and are well beyond their critical points and do not respond to cooling for light ends containment. Loss of these components during sample transfer can bias testing results. In order for cooling to be effective in preventing vapor loss, the true vapor pressure sample of the sample, when cooled, should be below atmospheric pressure.

NOTE 15—Cooling crudes with high wax (paraffin) contents can result in wax precipitation within the MPC, which can affect results.

10.3.2.1 CONNECT small diameter low dead volume tubing to the sample cylinder Valve (SV) and insert to approximately 2 mm to 4 mm from the bottom of the test vessel.

10.3.2.2 While compressing the piston to maintain the sample in a single-phase state, slowly OPEN Valve (SV) to begin transferring sample to the test vessel. The sample transfer rate should be controlled to a slow rate to prevent any significant pressure drop within the MPC and transfer tubing, which can result in sample vaporization and formation of a vapor phase within the MPC. The tubing should remain below the liquid level during filling to minimize sample vaporization.

10.3.2.3 CLOSE Valve (SV) when the desired sample volume is achieved.

10.3.2.4 Proceed with analysis as per test method and/or manufacturer instructions.

11. Keywords

11.1 condensate sampling; crude oil sampling; high vapor pressure; live crude oil; manual piston cylinder; sampling

APPENDIX

(Nonmandatory Information)

X1. ADDITIONAL SAMPLE TRANSFER PROCEDURES

X1.1 *Transfer from an MPC to an Intermediate FPC Cylinder*—Some analytical methods require sample pressurization for injection beyond the maximum working pressure of the MPC. In this instance, it is necessary to transfer the sample from the MPC into an FPC. When transferring from one pressurized vessel (MPC) to another (FPC), caution should be exercised to ensure atmospheric air is not introduced into the sample, which can affect the analytical results. Transfer tubing volume shall be kept as low as is practical. This procedure may not apply to all types of cylinder valve configurations. Fig. X1.1 illustrates the configuration and transfer procedure.

X1.1.1 CONNECT following manufacturer's instructions, FILL the pre-charge side of the FPC with inert gas or water through valve (Z) and OPEN the FPC product valve (X) to allow expulsion of air as the piston is forced to the end of the cylinder. The inert gas or water source pressure shall be 10 % greater that MPC sample pressure.

X1.1.2 CLOSE FPC purge valve (Y).

X1.1.3 CONFIGURE transfer lines as per Fig. X1.1 using low dead volume fittings, valve, and tubing.

X1.1.4 CONNECT valve (V) to a vacuum pump and turn on the pump.

X1.1.5 OPEN vacuum valve (V) and FPC valve (X). Allow 5 min under vacuum pressure (<13 kPa) to remove residual air from the transfer line and cylinder dead volume. See Fig. X1.1-A.

X1.1.6 CLOSE vacuum valve (V) and FPC valve (X).

X1.1.7 Disconnect vacuum pump from vacuum valve (V).

X1.1.8 While compressing the MPC piston, SLOWLY OPEN the MPC sample valve (SV) to allow sample to fill the evacuated transfer line dead volume.

X1.1.9 While compressing the MPC piston, SLOWLY OPEN the FPC product valve (X) to allow sample to fill the evacuated FPC dead volume.

X1.1.10 While compressing the MPC piston, SLOWLY OPEN the FPC purge valve (Y) to purge a small volume of sample (1 mL to 2 mL) to remove any gas bubbles that may have formed when exposed to vacuum conditions. CLOSE the FPC purge valve (Y). See Fig. X1.1-B.

X1.1.11 Set the backpressure regulator (BPR) to 10 % above the MPC sample pressure.

X1.1.12 While compressing the MPC piston, SLOWLY OPEN the FPC pre-charge valve (Z) to VENT pre-charge inert gas or water through the BPR resulting in a differential pressure between the cylinders and allowing the sample to transfer to the FPC. See Fig. X1.1-C. CLOSE the FPC pre-charge valve when sufficient sample has transferred to perform the test required. The backpressure regulator will ensure the water pressure does not drop below the MPC sample pressure during the transfer.

X1.1.13 CLOSE the FPC product valve (X) and the MPC sample valve (SV).

X1.1.14 VENT any remaining pressure from the transfer line dead volume through vacuum valve (V).

X1.1.15 Follow instrument manufacturer instructions for sample injection and loading.

X1.2 *Transfer from an MPC to an Intermediate Constant Volume Cylinder (CVC) Using Water Displacement*—

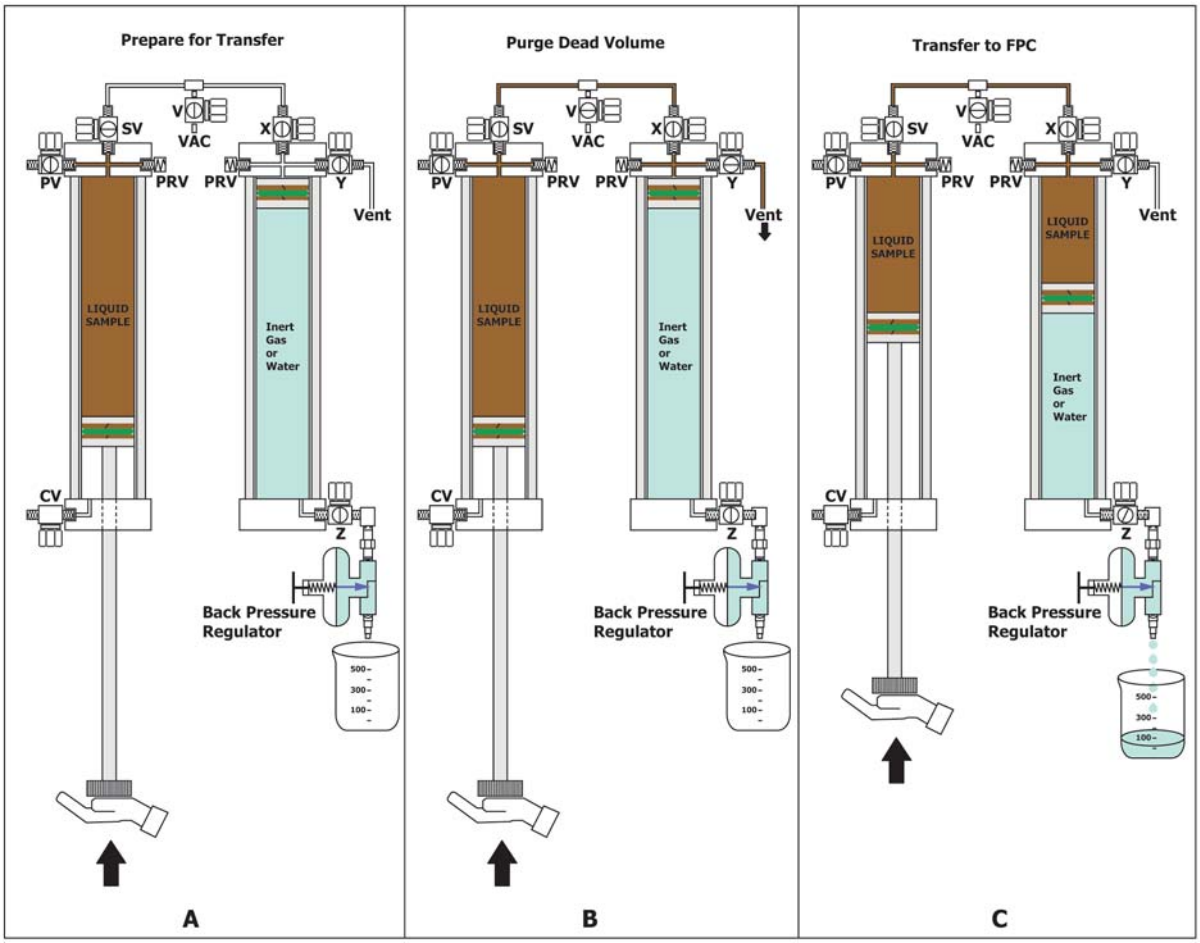

FIG. X1.1 Transfer from MPC to FPC

Some analytical methods require sample pressurization for injection beyond the maximum working pressure of the MPC. In this instance, it is necessary to transfer the sample from the MPC into a CVC. When transferring from one pressurized vessel (MPC) to another (CVC), caution should be exercised to ensure atmospheric air, which can affect the analytical results, is not introduced into the sample. Transfer tubing volume shall be kept as low as is practical. This procedure may not apply to all types of cylinder valve configurations. Figs. X1.2 and X1.3 illustrate the preparation and transfer procedure.

NOTE X1.1—Water displacement is not suitable for use if water is also a component of interest or if water can affect the test results.

X1.2.1 *CVC Preparation*—Fig. X1.2 illustrates the preparation method used. The cylinder shall remain upright during preparation.

X1.2.1.1 Install ¼-in. hose barb fitting in valve (Y).

X1.2.1.2 Connect a length of low-pressure flexible tubing from the outlet of the water reservoir to valve (Y) on the cylinder.

X1.2.1.3 Fill reservoir with water with sufficient volume to fill the cylinder without emptying the reservoir entirely. (1000 mL recommended). Record the initial volume. See Fig. X1.2-A.

X1.2.1.4 OPEN the cylinder valve (Y).

X1.2.1.5 OPEN the reservoir outlet valve to begin filling the cylinder with water.

X1.2.1.6 OPEN cylinder valve (X), allowing air to vent, and continue filling until water exits valve (X), then immediately CLOSE valve (X). See Fig. X1.2-B.

X1.2.1.7 CLOSE valve (Y). The cylinder is now filled completely with water and prepared for sampling. Record the final volume. See Fig. X1.2-C.

NOTE X1.2—Do not fill the intermediate CVC with cold water and then allow it warm significantly. Cylinder rupture could occur due to thermal expansion of the liquid. Use both cylinder and water at room temperature.

X1.2.1.8 The fill volume will be the difference between the initial and final volumes. This volume will be used during the sample transfer.

X1.2.2 *Sample Transfer from an MPC to a CVC*—Fig. X1.3 illustrates the transfer from an MPC to CVC. CVC shall remain in an upright position during sample transfer.

X1.2.2.1 CONFIGURE transfer lines as per Fig. X1.3 using low dead volume fittings, valves, tubing.

X1.2.2.2 CONNECT vacuum valve (V) to a vacuum pump and turn on the pump.

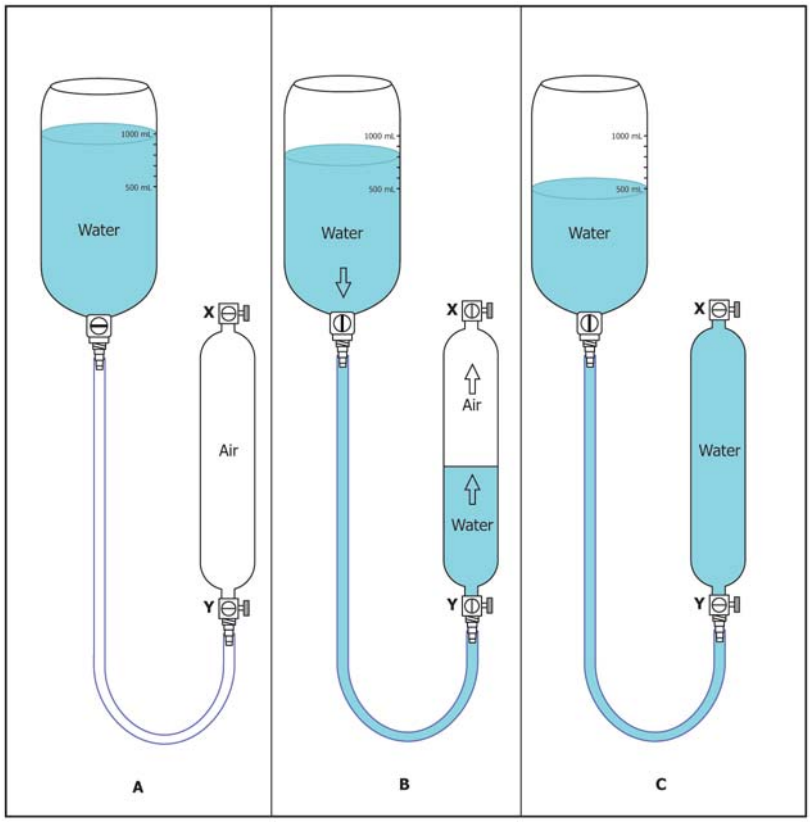

FIG. X1.2 CVC Preparation

X1.2.2.3 OPEN vacuum valve (V) and allow 5 min under vacuum pressure (<13 kPa) to remove residual air from the transfer line dead volume. See Fig. X1.3-A.

X1.2.2.4 CLOSE vacuum valve (V).

X1.2.2.5 Disconnect vacuum pump from vacuum valve (V).

X1.2.2.6 While compressing the MPC piston, SLOWLY OPEN the MPC sample valve (SV) to allow sample to fill the evacuated transfer line dead volume.

X1.2.2.7 While compressing the MPC piston, SLOWLY OPEN the CVC valve (X) to allow sample to contact with the displacement water within the CVC (see Fig. X1.3-B).

X1.2.2.8 SET the backpressure regulator (BPR) to 10 % above the MPC sample pressure.

X1.2.2.9 While compressing the MPC piston, SLOWLY OPEN the CVC valve (Y) to begin displacing water through the BPR and allowing the sample to transfer to the CVC. See Fig. X1.3-C. The backpressure regulator will ensure the water pressure does not drop below the MPC sample pressure during the transfer.

X1.2.2.10 CLOSE the CVC valve (Y) when sufficient sample volume has been transferred to perform the test required, as observed by the water displaced from the CVC.

X1.2.2.11 CLOSE the CVC product valve (X) and the MPC sample valve (SV).

X1.2.2.12 VENT any remaining pressure from the transfer line dead volume through vacuum valve (V).

X1.2.3 *Transfer from a CVC to a Pressurized Instrument*—An additional CVC is used to act as a separation cylinder between pressurized inert gas source and displacement water, which is then used to displace sample from the sample cylinder into the instrument. When transferring from a pressurized vessel (MPC) to an instrument, minimize contact with atmospheric air by using transfer-tubing volumes as low as is practical. Fig. X1.4 illustrates the configuration. The sample cylinder and separation cylinder shall remain upright throughout sample transfer to instrument.

X1.2.3.1 PREPARE a second separation cylinder as per X1.2.1 with the exception to only fill to 80 % of the total cylinder volume by monitoring the volume of water used to fill.

X1.2.3.2 CONNECT the separation cylinder valve (G) to an inert gas source such as argon or nitrogen.

X1.2.3.3 CONNECT the separation cylinder valve (W) to the sample cylinder as configuration in Fig. X1.4.

X1.2.3.4 ADJUST the inert gas pressure to 10 % higher than the sample cylinder pressure.

X1.2.3.5 OPEN the separation cylinder valve (G) to pressurize the separation cylinder.

X1.2.3.6 OPEN the separation cylinder valve (W) and the sample cylinder valve (Y) to equalize the pressure between the two cylinders.

X1.2.3.7 CONNECT the sample cylinder valve (X) to the instrument.

X1.2.3.8 CONNECT a flow control valve, such as a needle or ball valve, to the instrument outlet to allow flow control when purging the sample.

X1.2.3.9 CLOSE the control valve.

X1.2.3.10 CONNECT sample cylinder valve (X) to the inlet of the instrument.

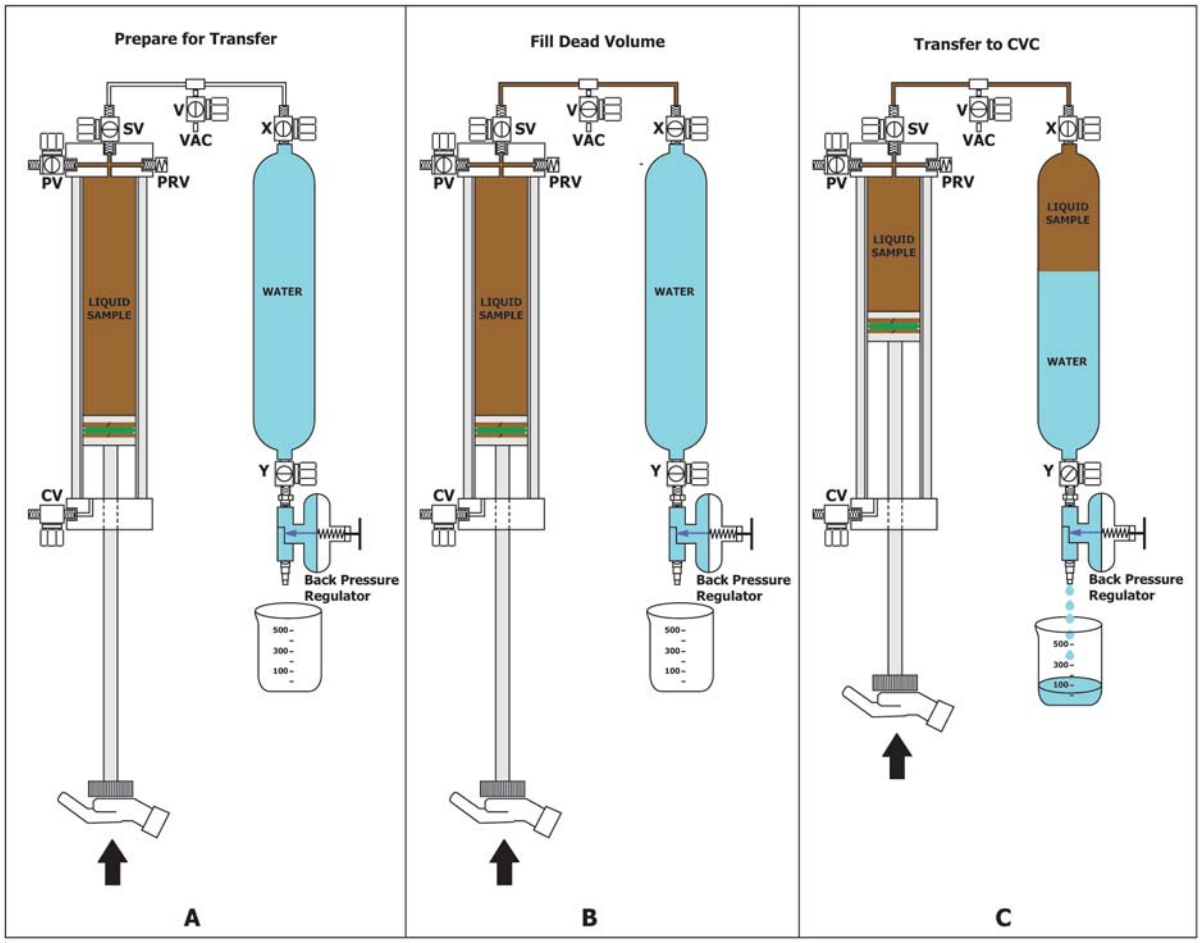

FIG. X1.3 Transfer from MPC to CVC

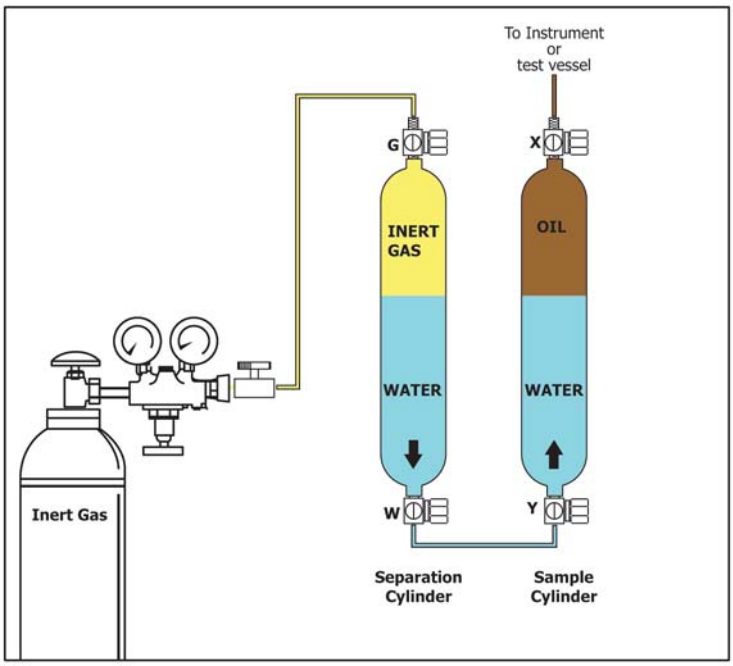

FIG. X1.4 Separation Cylinder Configuration for CVC Displacement

X1.2.3.11 OPEN sample cylinder Valve (X) to begin filling the transfer line and instrument. When filling is complete OPEN valve (X) completely to allow full flow.

X1.2.3.12 Slowly OPEN the instrument outlet control valve and purge the sample through the instrument until only single-phase (no gas bubbles or sputtering) sample appears at the outlet. CLOSE the instrument outlet control valve.

X1.2.3.13 The instrument is now full of single-phase sample and ready for analysis.

X1.2.3.14 Proceed with analysis as per manufacturer instructions.