

Manual of Petroleum Measurement Standards Chapter 8.1

Standard Practice for Manual Sampling of Petroleum and Petroleum Products

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Standard Practice for Manual Sampling of Petroleum and Petroleum Products¹

This standard is issued under the fixed designation D4057; 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.

This standard has been approved for use by agencies of the Department of Defense.

INTRODUCTION

The previous version of the manual sampling practice described the various sampling methods and apparatus, with much focus on crude oils and semi-solids and solids. Also, previous versions did not significantly address closed or restricted sampling, which continue to become more prevalent.

This version will provide guidance on manual sampling terminology, concepts, equipment, containers, procedures, and will provide some specific guidance related to particular products and tests. The type and size of the sample obtained, and the handling method, will depend on the purpose for which it was taken. Refer to the test method for any specific sampling and handling requirements up to the point of testing. It remains the responsibility of the subcommittee for the relevant test method to provide guidance, or warnings, regarding sample container selection; preparation; cleanliness; heat, pressure, or light; sample size requirements for testing and retention; and any other special handling requirements necessary to ensure a representative sample is tested.

In addition to the individual test method, for guidance on container, size, mixing and special handling, further guidance may be provided in Practice [D5854](#) (API MPMS Chapter 8.3), Practice [D5842](#) (API MPMS Chapter 8.4), and Practice [D4306](#). While this practice will provide some general guidance regarding sample chain of custody, Guide [D4840](#) should also be consulted.

This document has been developed jointly between the American Petroleum Institute (API) and ASTM International.

1. Scope

1.1 This practice covers procedures and equipment for manually obtaining samples of liquid petroleum and petroleum products, crude oils, and intermediate products from the sample point into the primary container are described. Procedures are also included for the sampling of free water and other heavy components associated with petroleum and petroleum products.

1.2 This practice also addresses the sampling of semi-liquid or solid-state petroleum products.

1.3 This practice provides additional specific information about sample container selection, preparation, and sample handling.

1.4 This practice does not cover sampling of electrical insulating oils and hydraulic fluids. If sampling is for the precise determination of volatility, use Practice [D5842](#) (API MPMS Chapter 8.4) in conjunction with this practice. For sample mixing and handling, refer to Practice [D5854](#) (API MPMS Chapter 8.3).

1.5 The procedures described in this practice may also be applicable in sampling most non-corrosive liquid industrial chemicals provided that all safety precautions specific to these chemicals are followed. Also, refer to Practice [E300](#). The procedures described in this practice are also applicable to sampling liquefied petroleum gases and chemicals. Also refer to Practices [D1265](#) and [D3700](#). The procedure for sampling bituminous materials is described in Practice [D140](#). Practice [D4306](#) provides guidance on sample containers and preparation for sampling aviation fuel.

¹ This practice is under the jurisdiction of ASTM Committee [D02](#) on Petroleum Products and Lubricants and the API Committee on Petroleum Measurement and is the direct responsibility of Subcommittee [D02.02](#) /COMQ 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 1981.

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1.6 *Units*—The values stated in SI units are to be regarded as the standard. USC units are reflected in parentheses.

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:²

- D86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure
- D97 Test Method for Pour Point of Petroleum Products
- D140 Practice for Sampling Bituminous Materials
- D217 Test Methods for Cone Penetration of Lubricating Grease
- D244 Test Methods and Practices for Emulsified Asphalts
- D268 Guide for Sampling and Testing Volatile Solvents and Chemical Intermediates for Use in Paint and Related Coatings and Material
- D287 Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)
- D323 Test Method for Vapor Pressure of Petroleum Products (Reid Method)
- D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis
- D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
- D473 Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method (API MPMS Chapter 10.1)
- D664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration
- D977 Specification for Emulsified Asphalt
- D1265 Practice for Sampling Liquefied Petroleum (LP) Gases, Manual Method
- D1267 Test Method for Gage Vapor Pressure of Liquefied Petroleum (LP) Gases (LP-Gas Method)
- D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method (API MPMS Chapter 9.1)
- D1657 Test Method for Density or Relative Density of Light Hydrocarbons by Pressure Hydrometer (API MPMS Chapter 9.2)
- D1838 Test Method for Copper Strip Corrosion by Liquefied Petroleum (LP) Gases
- D1856 Test Method for Recovery of Asphalt From Solution by Abson Method
- D2172 Test Methods for Quantitative Extraction of Bitumen From Bituminous Paving Mixtures
- D2622 Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry
- D3230 Test Method for Salts in Crude Oil (Electrometric Method)

- D3700 Practice for Obtaining LPG Samples Using a Floating Piston Cylinder
- D4006 Test Method for Water in Crude Oil by Distillation (API MPMS Chapter 10.2)
- D4007 Test Method for Water and Sediment in Crude Oil by the Centrifuge Method (Laboratory Procedure) (API MPMS Chapter 10.3)
- D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products (API MPMS Chapter 8.2)
- D4294 Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry
- D4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination
- D4377 Test Method for Water in Crude Oils by Potentiometric Karl Fischer Titration (API MPMS Chapter 10.7)
- D4530 Test Method for Determination of Carbon Residue (Micro Method)
- D4629 Test Method for Trace Nitrogen in Liquid Petroleum Hydrocarbons by Syringe/Inlet Oxidative Combustion and Chemiluminescence Detection
- D4807 Test Method for Sediment in Crude Oil by Membrane Filtration (API MPMS Chapter 10.8)
- D4840 Guide for Sample Chain-of-Custody Procedures
- D4928 Test Method for Water in Crude Oils by Coulometric Karl Fischer Titration (API MPMS Chapter 10.9)
- D4929 Test Methods for Determination of Organic Chloride Content in Crude Oil
- D5002 Test Method for Density and Relative Density of Crude Oils by Digital Density Analyzer
- D5191 Test Method for Vapor Pressure of Petroleum Products (Mini Method)
- D5762 Test Method for Nitrogen in Petroleum and Petroleum Products by Boat-Inlet Chemiluminescence
- D5842 Practice for Sampling and Handling of Fuels for Volatility Measurement (API MPMS Chapter 8.4)
- D5853 Test Method for Pour Point of Crude Oils
- D5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products (API MPMS Chapter 8.3)
- D5863 Test Methods for Determination of Nickel, Vanadium, Iron, and Sodium in Crude Oils and Residual Fuels by Flame Atomic Absorption Spectrometry
- D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance
- D6377 Test Method for Determination of Vapor Pressure of Crude Oil: VPCR_x (Expansion Method)
- D6470 Test Method for Salt in Crude Oils (Potentiometric Method)
- D6560 Test Method for Determination of Asphaltenes (Heptane Insolubles) in Crude Petroleum and Petroleum Products
- D6822 Test Method for Density, Relative Density, and API Gravity of Crude Petroleum and Liquid Petroleum Products by Thermohydrometer Method (API MPMS Chapter 9.3)

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.

D6849 Practice for Storage and Use of Liquefied Petroleum Gases (LPG) in Sample Cylinders for LPG Test Methods
 D7169 Test Method for Boiling Point Distribution of Samples with Residues Such as Crude Oils and Atmospheric and Vacuum Residues by High Temperature Gas Chromatography
 E300 Practice for Sampling Industrial Chemicals
 E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

2.2 *API Manual of Petroleum Measurement Standards*:³
 MPMS Chapter 8.2 Automatic Sampling of Petroleum and Petroleum Products (ASTM Practice D4177)
 MPMS Chapter 8.3 Standard Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products (ASTM Practice D5854)
 MPMS Chapter 8.4 Standard Practice for the Sampling and Handling of Fuels for Volatility Measurements (ASTM Practice D5842)
 MPMS Chapter 9.1 Standard Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum (ASTM Test Method D1298)
 MPMS Chapter 9.2 Standard Test Method for Density or Relative Density of Light Hydrocarbons by Pressure Hydrometer (ASTM Test Method D1657)
 MPMS Chapter 9.3 Standard Test Method for Density, Relative Density, and API Gravity of Crude Petroleum and Liquid Petroleum Products by Thermohydrometer Method (ASTM Test Method D6822)
 MPMS Chapter 10.1 Standard Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method (ASTM Test Method D473)
 MPMS Chapter 10.2 Standard Test Method for Water in Crude Oil by Distillation (ASTM Test Method D4006)
 MPMS Chapter 10.3 Standard Test Method for Water and Sediment in Crude Oil by the Centrifuge Method (Laboratory Procedure) (ASTM Test Method D4007)
 MPMS Chapter 10.4 Standard Test Method for Water and Sediment in Crude Oil by the Centrifuge Method (Laboratory Procedure)
 MPMS Chapter 10.7 Standard Test Method for Water in Crude Oils by Potentiometric Karl Fischer Titration (ASTM Test Method D4377)
 MPMS Chapter 10.8 Standard Test Method for Water in Crude Oils by Potentiometric Karl Fischer Titration (ASTM Test Method D4807)
 MPMS Chapter 10.9 Standard Test Method for Water in Crude Oils by Coulometric Karl Fischer Titration (ASTM Test Method D4928)
 MPMS Chapter 14.6 Pressure Pycnometer
 MPMS Chapter 17.1 Guidelines for Marine Cargo Inspection
 MPMS Chapter 17.2 Measurement of Cargoes Aboard Marine Tank Vessels
 MPMS Chapter 18.1 Measurement Procedures for Crude Oil Gathered from Small Tanks by Truck

2.3 *Gas Processors Association (GPA) Standards*:⁴
 GPA S 2174 Obtaining Liquid Hydrocarbon Samples for Analysis by Gas Chromatograph

2.4 *Other Publications*:

UOP163 Hydrogen Sulfide and Mercaptan Sulfur in Liquid Hydrocarbons by Potentiometric Titration⁵
 49 CFR 173 Shippers—General Requirements for Shipments and Packagings⁶

3. Terminology

3.1 Definitions:

3.1.1 *assay, n*—the procedure to determine the presence, absence, or quantity of one or more components.

3.1.2 *automatic sampler, n*—a device used to extract a representative sample from the liquid flowing in a pipe; the automatic sampler generally consists of a probe, a sample extractor, an associated controller, a flow measuring device, and a sample receiver.

3.1.3 *bubble point, n*—the pressure at which the first bubble of vapor forms is the bubble point when the pressure is lowered on a liquid held at a constant temperature.

3.1.3.1 *Discussion*—Bubble point pressures are higher at high temperatures.

3.1.4 *density, n*—for a quantity of a homogeneous substance, the ratio of its mass to its volume. The density varies as the temperature changes and is, therefore, generally expressed as the mass per unit of volume at a specified temperature.

3.1.5 *dissolved water, n*—water in solution in petroleum and petroleum products.

3.1.6 *emulsion, n*—a suspension of fine particles or globules, or both, of one or more liquids in another liquid.

3.1.7 *entrained water, n*—water suspended in the petroleum and petroleum products. Entrained water includes emulsions but does not include dissolved water.

3.1.8 *free water, n*—water that exists as a separate phase.

3.1.9 *flash point, n*—in petroleum products, the lowest temperature corrected to a barometric pressure of 101.3 kPa (760 mm Hg), at which application of an ignition source causes the vapors of a specimen of the sample to ignite under specified conditions of test.

3.1.10 *floating piston (variable volume) cylinder (FPC), n*—a high pressure sample container, with a free floating internal piston that effectively divides the container into two separate compartments.

3.1.11 *high pressure cylinder, n*—a receptacle used for storage and transportation of a sample obtained at pressures above atmospheric pressure.

⁴ Available from Gas Processors Association (GPA), 6526 E. 60th St., Tulsa, OK 74145, <http://www.gasprocessors.com>.

⁵ Available from ASTM International. Visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org.

⁶ Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, <http://www.access.gpo.gov>.

³ Available from American Petroleum Institute (API), 1220 L. St., NW, Washington, DC 20005-4070, <http://www.api.org>.

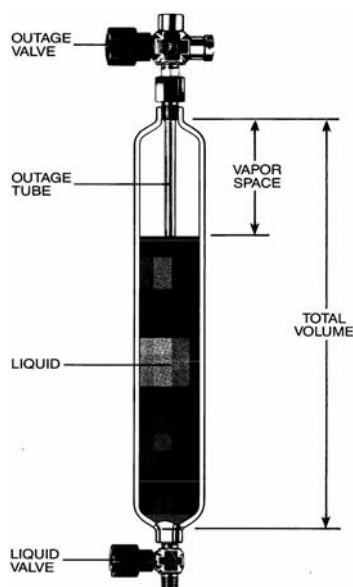


FIG. 1 Example of a Fixed Volume Cylinder with an Outage Tube

3.1.12 *inert gas, n*—a gas that does not react with its surroundings.

3.1.13 *inerting, v*—a procedure used to reduce the oxygen content of the vapor spaces by introducing an inert gas such as nitrogen or carbon dioxide or a mixture of gases such as processed flue gas.

3.1.14 *intermediate sample container, n*—a container into which all or part of the sample from a primary container (receiver) is transferred for transport, storage, or ease of handling.

3.1.15 *LPG (liquefied petroleum gas), n*—narrow boiling range hydrocarbon mixtures consisting mainly of propane or propylene, or both, and butanes or butylenes, or both, plus limited amounts of other hydrocarbons and naturally-occurring non-hydrocarbons.

3.1.16 *maximum fill density (reduced fill density), n*—the volume of a container occupied by the sample, usually expressed as a percentage of the total capacity. Transportation legislation such as U.S. CFR 49, Canadian Transportation of Dangerous Goods Regulations, and IATA regulations limit the percent fill of containers used for shipping LPG and may quote this requirement as a reduced fill density or maximum fill density (normally 80 % maximum liquid fill at 15°C). Lower percent fill (lower fill density) may be required if sampling at lower temperatures.

3.1.17 *on-board quantity (OBQ), n*—the material present in a vessel's cargo tanks, void spaces, and pipelines before the vessel is loaded. On-board quantity may include any combination of water, oil, slops, oil residue, oil/water emulsion, and sediment.

3.1.18 *outage tube (internal), n*—a “cut to length” tube placed inside of the cylinder used as a way to remove excess sample from the cylinder via manual evacuation after the sample cylinder assembly is removed from the sample point. Refer to Fig. 1 and Fig. 2.

3.1.19 *portable manual sampling unit, PSU, n*—an intrinsically safe device used in conjunction with a vapor control valve to obtain required cargo samples under closed or restricted system conditions. Refer to Fig. 3 and Fig. 4.

3.1.20 *primary sample container, n*—a container in which a sample is initially collected.

3.1.20.1 *Discussion*—Examples of primary sample containers include glass and plastic bottles, cans, core-type thief, and fixed and portable sample containers (receivers).

3.1.21 *remaining on board, ROB, n*—the material remaining in a vessel's cargo tanks, void spaces, and pipelines after the cargo is discharged. Remaining on board quantity may include any combination of water, oil, slops, oil residue, oil/water emulsions, and sediment.

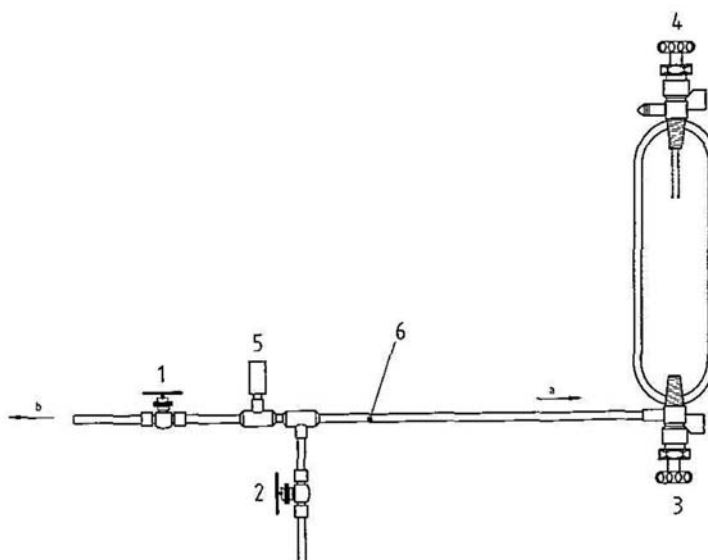
3.1.22 *sample, n*—a portion extracted from a total volume that may or may not contain the constituents in the same proportions that are present in that total volume.

3.1.23 *sample loop (fast loop or slip stream), n*—a low volume bypass diverted from the main pipeline.

3.1.24 *sampling, v*—all the steps required to obtain a sample that is representative of the contents of any pipe, tank, or other vessel and to place that sample in a container from which a representative test specimen can be taken for analysis.

3.1.25 *slip tube, n*—a graduated hollow rod fitted into a gas-tight housing, the lower end of which is open to the cargo's contents and the upper end is fitted with a valve.

3.1.26 *standpipes, n*—the vertical sections of pipe or tubing used for gauging extending from the gauging platform to near the bottom of tanks that are equipped with external or internal floating roofs. Standpipes may also be found on marine vessels. Standpipes are also known as “stilling wells” or “gauge wells.” Standpipes without slots do not allow the free flow of product through the standpipe, and are known as solid or unslotted standpipes.

**Key**

1 sample point control valve

2 vent / purge valve

3 liquid valve cylinder inlet

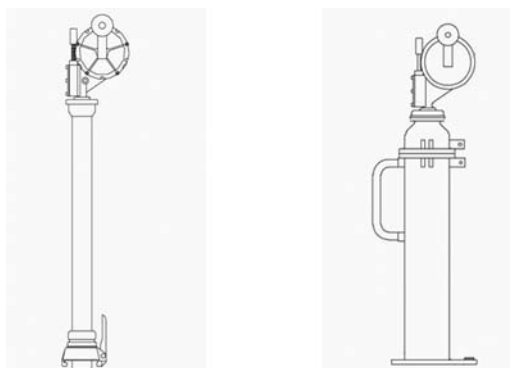
4 outage valve

5 pressure relief

6 tubing

a To liquid valve cylinder inlet

b Product source

FIG. 2 Example of a Fixed Volume Cylinder and Transfer Line**FIG. 3 Examples of a Small Volume (5 cm (2 in.)) and a Large Volume PSU (10 cm (4 in.))**

3.1.27 *ullage (outage)*, *n*—the volume of available space in a container unoccupied by contents.

3.1.28 *vapor control valve, VCV*, *n*—a valve fitted on a standpipe, expansion trunk, or the deck that permits use of the portable handheld gauging/sampling instruments while restricting the release of vapors into the atmosphere.

3.1.29 *vapor pressure*, *n*—the pressure exerted by the vapor of a liquid when in equilibrium with the liquid.

3.1.29.1 *Reid vapor pressure, RVP*, *n*—resultant total pressure reading, corrected for measuring error, of a specific

empirical test method (Test Method **D323**) for measuring the vapor pressure of gasoline and other volatile products.

3.1.29.2 *true vapor pressure, TVP*, *n*—the pressure at which the fluid is in equilibrium between its liquid and gas state.

Sample Types

3.1.30 *all-levels sample*, *n*—a sample obtained by lowering the closed sampling device to the bottom of the outlet suction level, but always above free water, then opening the sampler and raising it at a uniform rate such that it is between 70 and



FIG. 4 Examples of Closed/Restricted Sampling Equipment

85 % full when withdrawn from the product. Alternatively, all-levels samples may be taken with samplers designed for filling as they pass downward through the product.

3.1.30.1 *Discussion*—If required by the test method, the sampler may be greater than 85 % full when withdrawn but in no case shall it be completely full. In these cases, take special handling precautions to consider the hazards associated with product thermal expansion.

3.1.31 *boring sample, n*—a sample of the material contained in a barrel, case, bag, or cake that is obtained from the chips created by boring holes into the material with a ship auger.

3.1.32 *bottom sample, n*—a spot sample collected from the material at the bottom of the tank, container, or line at its lowest point. In practice, the term bottom sample has a variety of meanings. As a result, it is recommended that the exact sampling location (for example 15 cm (6 in.) from the bottom) should be specified when using this term. See Fig. 5.

3.1.33 *bottom water sample, n*—a spot sample of free water taken from beneath the petroleum contained in a ship or barge compartment or a storage tank.

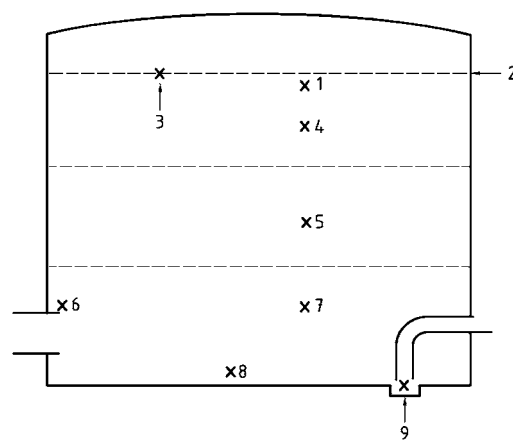
3.1.34 *clearance sample, n*—a spot sample taken with the inlet opening of the sampling device 10 cm (4 in.) (some regulatory agencies require 15 cm (6 in.)) below the bottom of the tank outlet. This term is normally associated with small (159 m³ (1000 barrels) or less) tanks, commonly referred to as lease tanks.

3.1.35 *composite sample, n*—a sample prepared by combining a number of samples and treated as a single sample. Also refer to “tank composite sample,” “volumetric composite sample,” “deck composite sample,” and “multiple tank composite sample” definitions.

3.1.36 *core sample, n*—a sample of uniform cross-sectional area taken at a given height in a tank.

3.1.37 *dead bottom sample, n*—a sample obtained from the lowest accessible point in a tank. This is typically directly from the floor (or datum plate) of the shore tank or the bottom of the vessel compartment.

3.1.38 *deck composite sample, n*—a sample typically made by compositing a portion of each sample obtained from all vessel compartments containing a particular product grade.



Key

- | | |
|----------------------|-----------------------------------|
| 1 Top sample | 6 Suction level or outlet samples |
| 2 Surface of product | 7 Lower sample |
| 3 Skim sample | 8 Bottom sample |
| 4 Upper sample | 9 Sump sample |
| 5 Middle sample | |

FIG. 5 Illustration of Common Spot Sample Positions

3.1.39 *dipper sample, n*—a sample obtained by placing a dipper or other collecting vessel in the path of a free-flowing stream to collect a definite volume from the full cross section of the stream at regular time intervals for a constant time rate of flow or at time intervals varied in proportion to the flow rate.

3.1.40 *drain sample, n*—a sample obtained from the water draw-off valve on a storage tank vessel or container. Occasionally, a drain sample may be the same as a bottom sample (for example, in the case of a tank car).

3.1.41 *floating roof sample, n*—a spot sample taken just below the surface to determine the density (API gravity) of the liquid on which the roof is floating.

3.1.42 *grab sample, n*—(a) *solid*—a sample obtained by collecting equal quantities from parts or packages of a shipment of loose solids so that the sample is representative of the entire shipment. (b) *liquid*—a sample collected at a specific location in a tank or from a flowing stream in a pipe at a specific time.

TABLE 1 Sampling from Horizontal Cylindrical Tanks

Liquid Depth (% of Diameter)	Sampling Level (% of Diameter above Bottom)			Composite Sample (Proportional Parts)		
	Upper	Middle	Lower	Upper	Middle	Lower
100	80	50	20	3	4	3
90	75	50	20	3	4	3
80	70	50	20	2	5	3
70	...	50	20	...	6	4
60	...	50	20	...	5	5
50	...	40	20	...	4	6
40	20	10
30	15	10
20	10	10
10	5	10

3.1.43 *grease sample, n*—obtained by scooping or dipping a quantity of soft or semi-liquid material contained from a package in a representative manner.

3.1.44 *loading zone sample, n*—a sample taken from a tank prior to commencement of a transfer, intended to represent only the product expected to be transferred.

3.1.45 *lower sample, n*—a spot sample of liquid from the middle of the lower one-third of the tank's content (a distance of five-sixths of the depth liquid below the liquid's surface). See Fig. 5.

3.1.46 *middle sample, n*—a spot sample taken from the middle of a tank's contents (a distance of one half of the depth of liquid below the liquid's surface). See Fig. 5.

3.1.47 *multiple tank composite sample, n*—a mixture of individual samples or composites of samples that have been obtained from several tanks or ship/barge compartments containing the same grade of material. The mixture is blended typically in proportion to the volume of material contained in the respective tanks or compartments.

3.1.48 *representative sample, n*—a portion extracted from the total volume that contains the constituents in the same proportions that are present in that total volume.

3.1.49 *running sample, n*—a sample obtained by lowering an open sampling device to the bottom of the outlet suction level, but always above free water, and returning it to the top of the product at a uniform rate such that the sampling device is between 70 and 85 % full when withdrawn from the product.

3.1.49.1 *Discussion*—If required by the test method, the sampler may be greater than 85 % full when withdrawn but in no case shall it be completely full. In these cases, take special handling precautions to consider the hazards associated with product thermal expansion.

3.1.50 *spot sample, n*—a sample taken at a specific location in a tank or from a flowing stream in a pipe at a specific time.

3.1.51 *suction sample (outlet), n*—a spot sample taken at the lowest level from which product is expected to be pumped from the tank; see Fig. 5.

3.1.52 *sump sample, n*—spot sample taken from within the tank or vessel compartment sump; see Fig. 5.

3.1.53 *surface sample (skim sample), n*—a spot sample skimmed from the surface of a liquid in a tank. See Fig. 5.

3.1.54 *tank composite sample, n*—a blend created from a single tank, as an example combining the upper, middle, and

lower samples. For a tank of uniform cross section, such as an upright cylindrical tank, the blend consists of equal parts of the three samples. A combination of other samples may also be used, such as running, all-levels or additional spot samples. For a horizontal cylindrical tank, the blend consists of samples in the proportions shown in Table 1.

3.1.55 *tank tap sample, n*—a spot sample taken from a sample tap on the side of a tank. It may also be referred to as a tank-side sample.

3.1.56 *test specimen, n*—a representative sub-sample taken from the primary or intermediate sample container for analysis.

3.1.57 *top sample, n*—a spot sample obtained 15 cm (6 in.) below the top surface of the liquid. See Fig. 5.

3.1.58 *tube sample (thief sample), n*—a sample obtained with a sampling tube or special thief, either as a core sample or spot sample, from a specific point in the tank or container.

3.1.59 *upper sample, n*—a spot sample taken from the middle of the upper one third of the tank's contents (a distance of one-sixth of the depth of the liquid below the liquid's surface). See Fig. 5.

3.1.60 *volumetric composite sample, n*—a sample consisting of measured proportional parts from each zone if it is for a single tank. If the volumetric composite is for multiple tanks, or vessel compartments, it consists of measured proportional parts from each tank or compartment sampled.

3.1.61 *zone sample, n*—a sample taken as that part of the liquid column that is trapped within the whole height of a sampling device when it is sealed at a single spot location within a tank after having been fully flushed as it was lowered to that position.

4. Significance and Use

4.1 Samples of petroleum and petroleum products are obtained for many reasons, including the determination of chemical and physical properties. These properties may be used for: calculating standard volumes; establishing product value; and often safety and regulatory reporting.

4.2 There are inherent limitations when performing any type of sampling, any one of which may affect the representative nature of the sample. As examples, a spot sample provides a sample from only one particular point in the tank, vessel compartment, or pipeline. In the case of running or all-level



FIG. 6 Typical Sample Carrier

samples, the sample only represents the column of material from which it was taken.

4.3 Based on the product, and testing to be performed, this practice provides guidance on sampling equipment, container preparation, and manual sampling procedures for petroleum and petroleum products of a liquid, semi-liquid, or solid state, from the storage tanks, flowlines, pipelines, marine vessels, process vessels, drums, cans, tubes, bags, kettles, and open discharge streams into the primary sample container.

5. Health and Safety Precautions

5.1 *General*—This practice does not purport to cover all safety and health aspects associated with sampling. Personnel involved with sampling of petroleum and petroleum-related products should be familiar with their physical and chemical characteristics, including: potential for fire, explosion, and reactivity; toxicity and health hazards; and appropriate emergency procedures. Additionally personnel should comply with individual company safe operating practices and local, state, and national regulations, including the use of personal protective equipment (PPE). Upon completion of any sampling activity, ensure the sample point is left in a safe, secure, and clean condition with the handling of any waste in accordance with local requirements. All marine vessel sampling should be performed in the presence of a designated vessel representative.

5.2 *Sample Handling*—For safety and protection of the integrity of the samples, sample carriers are suggested in most instances. Refer to Fig. 6. Because of potential liquid thermal expansion, sample containers that are completely, or nearly full, are not to be transported or stored, unless special precautionary measures are taken. A safe fill of between 70 and 85 % is recommended. Refer to definition for maximum fill density and 9.30 for safe fill of pressurized cylinders. Take care to avoid heating samples in containers with gas-tight caps, lids, and stoppers. Handle any sample containing hazardous materials or the residue of hazardous materials offered for shipment/transportation by air, public roadway, rail, or water in such a

manner as to ensure compliance with requirements such as training, documentation, labeling, container, packaging, communications, and so forth, set forth in applicable regulations, such as those issued by the International Air Transport Association (IATA) and the U.S. Department of Transportation (DOT).

5.3 Sample Point Safety:

5.3.1 Provide sample points that enable samples to be taken in a safe manner, considering ventilation during sampling, clear access/egress, and lighting. Any potential hazards associated with sampling, or located near the sample point, should be clearly marked. It is recommended that a pressure gauge and a method of closed loop flushing with safe drainage, be provided at pipeline sample points. Sample points and related equipment should be maintained and inspected regularly.

5.3.2 Floating-roof tanks should be sampled from the top platform, thereby avoiding descent onto the floating roof. Descending onto a floating roof is normally considered entering a designated confined space, requiring all facility and regulatory requirements to be strictly followed, including obtaining a confined space permit, and rescue provisions arranged. Toxic and flammable vapors may accumulate on the roof.

5.4 Static Electricity Hazards:

5.4.1 A number of fires and explosions have occurred as a result of hydrocarbon vapors being ignited by static electricity. If electrical charges are not earthed or grounded, they are unable to dissipate and become “static.” This static electric charge can accumulate and freely migrate to a single point on the sample container by a difference in electrical potential, then jump off as a high-energy spark discharge to a nearby less charged surface, often hot and prolonged enough to ignite nearby hydrocarbon vapors above the lower explosive limit (LEL). This potential shall be managed by safely dissipating static charges, and through proper grounding, when sampling flammable products.

5.4.2 Footwear or clothing, capable of causing sparks, should not to be worn during sampling activities in which flammable vapors are likely to be present. Sampling should not be carried out during periods of atmospheric electric disturbance or hail storms. To ground any static charge on their person, the individual performing the sampling should touch part of the tank structure at least 1 m (3 ft) from the sample point immediately before sampling.

5.4.3 Precautions are to be observed before sampling to reduce the likelihood of a static charge being present. During tank filling or mixing operations, and for 30 min after the completion, sampling equipment shall not be introduced into, or remain in, the tank. With full observance of applicable regulatory requirements, and only under very specific and documented conditions, some exceptions to the 30-min relaxation period may apply. Some tanks and vessel compartments have inert gas blankets in the vapor space above the liquid. Unless the effectiveness of the inert blanket can be verified, all static charge precautions and recommendations should be observed.

5.4.4 Exercise caution when using equipment made of aluminum, magnesium, or titanium, which may generate incendiary sparks when struck against rusted steel. Some countries restrict the use of sampling equipment made from such materials or from alloys containing more than 15 % (m/m) in total of these metals or 6 % (m/m) of magnesium.

5.5 *Pipeline/Line Sampling*—When sampling from a flowing pipeline, maintain electrical continuity between the pipeline and the sample container via the connecting pipework. Do not use plastic containers since they are non-conductive and will not dissipate static electricity. Use a static grounding clamp or other arrangement that ensures adequate electrical continuity is maintained if sampling with a metal container. An effective ground should be verified.

NOTE 1—The API safety publication *Protection Against Ignitions Arising Out of Static, Lightning, and Stray Currents* states that electrical resistances of higher than 10 in metal circuits are indicative of a break in the continuity of the circuit, resulting in the undesirable accumulation of static electricity.⁷

5.6 *General Health Hazards and Precautions:*

5.6.1 Petroleum vapor dilutes oxygen in the air and may also be toxic. Hydrogen sulfide vapors are particularly hazardous. Harmful vapors or oxygen deficiency cannot always be detected by smell, visual inspection, or judgment. The use of oxygen and toxic gas monitors, PPE, and emergency rescue precautions should be considered for all sampling operations. Self-contained breathing apparatus (SCBA) may be necessary. Personnel should position themselves upwind of the sample point to minimize exposure to any harmful vapor which may be released.

5.6.2 This discussion on safety and health is not exhaustive. The appropriate Material Safety Data Sheet (MSDS), API, or ASTM International publication should be consulted, together with applicable regulatory requirements, and the International Safety Guide for Oil Tankers and Terminals (ISGOTT),⁸ Safety of Life at Sea (SOLAS),⁹ and Oil Companies International Marine Forum (OCIMF),¹⁰ while always observing company and local health and safety requirements.

6. Apparatus

6.1 *General Sample Container Design Considerations:*

6.1.1 Sample containers come in a variety of shapes, sizes, and materials. Select the proper container based on the product to be sampled to ensure that there will be no interaction between the product sampled and the container that would affect the integrity of either. The following are general design considerations for sample containers:

6.1.1.1 No internal pockets or dead spots;

6.1.1.2 Internal surfaces designed to minimize corrosion, encrustation, and water/sediment clingage;

⁷ *Protection Against Ignitions Arising Out of Static, Lightning, and Stray Currents*, Edition 7, American Petroleum Institute, Washington, DC, 2008.

⁸ *International Safety Guide for Oil Tankers and Terminals (ISGOTT)*, Hyperion Books, 2006.

⁹ *International Convention for the Safety of Life at Sea (SOLAS)*, International Maritime Organization, London, UK, 1974.

¹⁰ Available from Oil Companies International Marine Forum (OCIMF), 29 Queen Anne's Gate, London SW1H 9BU, U.K., <http://www.ocimf.com>.



FIG. 7 Amber and Clear Boston Round Bottles

6.1.1.3 An inspection cover/closure of sufficient size to facilitate filling, inspection, and cleaning;

6.1.1.4 Designed to allow the preparation, and transfer to the analytical apparatus, of a homogeneous mixture of the sample while minimizing the loss of any constituents that affect the representativeness of the sample and the accuracy of the analytical tests.

6.1.2 Additional considerations in the selection of sample containers are the type of mixing required before transferring from the primary container, and the analysis to be performed. To facilitate the discussion on proper handling and mixing of samples, sample containers are referred to as either primary or intermediate containers. Regardless of the type of sample container used, the sample container should be large enough to contain the required sample volume and sufficient ullage space for thermal expansion and mixing of the sample.

6.1.3 While this practice is meant to provide some guidance related to particular products and tests, it remains the responsibility of the subcommittee for the relevant test method to provide specific guidance regarding sample container selection, preparation, cleanliness, and sample size requirements for testing and retention. Also refer to Practice D5854 (API MPMS Chapter 8.3), Practice D5842 (API MPMS Chapter 8.4), and Practice D4306.

6.2 *Glass Bottles*—See Fig. 7. Glass containers are suitable for many sample test and storage requirements. Clear glass bottles can be easily examined visually for cleanliness, and

allow for visual inspection of the sample for haze (cloudiness), discoloration, free water, and solid impurities. The brown glass bottle affords some protection to the samples when light may affect the test results. Refer to Practice [D5854](#) (API MPMS Chapter 8.3).

6.3 Cans—When using cans, any seams shall have been soldered on the exterior surfaces with a flux of rosin in a suitable solvent. Such a flux is easily removed with gasoline, whereas many others are very difficult to remove. Cans made of stainless steel with welded seams, and aluminum bottles, are suitable for many sampling operations, but cleanliness is still required. Minute traces of flux may contaminate the sample so that results obtained on tests such as dielectric strength, oxidation resistance, and sludge formation may be erroneous. For aviation fuel sampling, refer to Practice [D4306](#).

6.4 Plastic Bottles—In general, plastic bottles made of suitable material may be used for the handling and storage of diesel oil, fuel oil, and lubricating oil. Bottles of this type should not be used for gasoline, aviation jet fuel, kerosene, crude oil, white spirit, medicinal white oil, or other petroleum products unless testing indicates there is no problem with solubility, contamination, or loss of light components. In no circumstances shall nonlinear (conventional) polyethylene containers be used to store samples of liquid hydrocarbons. This is to avoid sample contamination or sample bottle failure. Used engine oil samples that may have been subjected to fuel dilution should not be stored in plastic containers. Plastic bottles do have an advantage in that they will not shatter like glass or corrode like metal containers.

6.5 Container Closures:

6.5.1 Screw caps made of a material that will not deteriorate or contaminate the sample are to be used for glass bottles. Screw caps should provide a vapor-tight seal. Use care when using cork stoppers. Situations in which corks should not be used include: liquids in which loss of light ends may affect any test results; and liquids that are hygroscopic or have a low-water content specification. Rubber stoppers are not to be used.

6.5.2 Cans and plastic bottles should be closed with screw caps made of the same material as the container. Protect can screw caps with a disk faced with a material that will not deteriorate or contaminate the sample when used to store or transport samples. Consideration of closure type is important for samples in which vapor loss will affect the test results. Screw caps of a quality that provide a vapor-tight closure should be used for plastic bottles and cans. Use screw caps for containers used to take samples that will be tested for density or API gravity.

6.6 Container Cleanliness—Sample containers shall be clean and free from all substances that might contaminate the product being sampled (such as water, dirt, lint, washing compounds, naphtha and other solvents, soldering fluxes, acids, rust, and oil). Reusable containers shall be cleaned by a method that has been determined as acceptable for the intended use, for example by rinsing with a suitable solvent. Dry the container, for example either by passing a current of clean warm air through the container or placing it in a hot, dust-free

cabinet at 40°C (104°F) or higher. When dry, stopper or cap the container. Normally, it is not necessary to wash new containers.

6.7 Container Compatibility for Sample Mixing—The sample container should be compatible with the mixing system for remixing samples to ensure that a homogenous sample is transferred to an intermediate container or the analytical apparatus. This is particularly critical with crude oil, some black products, and condensates for sediment and water (S & W) analysis. Cylindrical containers are generally better suited for samples that are to be tested for S & W. Refer to Practice [D5854](#) (API MPMS Chapter 8.3) for sample mixing and handling requirements.

6.8 Low Pressure Variable Volume Containers—Low pressure variable volume sample container designs include collapsible plastics containers, bladders, and vessels fitted with a flexible internal diaphragm. Before use, variable volume sample containers are normally collapsed, evacuated or reduced to the nominal zero volume. The sample container size is dependent on the quantity required for analysis (and/or retention). Prior to use, it may be appropriate to rinse the sample containers with the product being sampled, in order to avoid contamination from previous sample residue and/or solvents used to clean the low pressure variable volume containers. The sample should be transported to the laboratory in the container in which it was originally obtained (the primary sample container). Plastics containers are not recommended for long-term sample storage, unless it has been demonstrated that the plastic is suitable (that is, compatible with the sample) so that the integrity of the sample is not compromised. (**Warning**—The use of containers made of non-linear polyethylene may lead to sample contamination and/or sample container failure.)

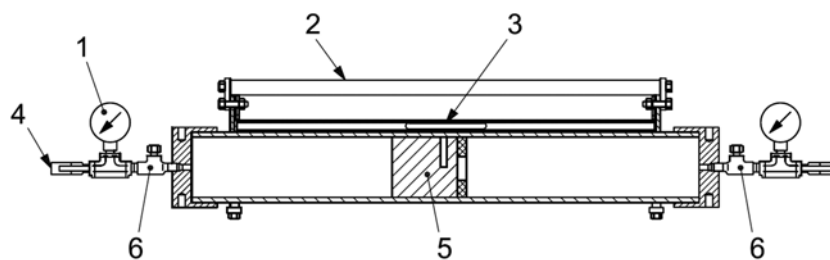
6.9 High Pressure Spot Sampling Systems and Cylinders:

6.9.1 Sample System Components—All equipment, such as transfer lines, valves and pressure gages, associated with sampling shall be corrosion resistant and designed consistent with the maximum anticipated pressure. Experience has shown that the transfer lines should have a minimum internal diameter of 3 mm (1/8 in.) nominal and be as short as practical to minimize line blockage or sample vaporization, or both. The use of filters, dryers, needle valves and related equipment are not recommended, unless provisions are made to prevent excessive flow restriction and pressure drop. It is recommended to use a “T” junction with a purge valve at the sample connection point to allow purging of the dead volume at the sampler connection. Flexible hose or tubing with adequate pressure rating may be used.

6.9.2 Cylinders—High pressure cylinders are used for the collection of light liquid hydrocarbons and gas samples and subsequent transportation and storage. Typical light liquid hydrocarbon sample containers are fixed volume cylinders or floating-piston cylinders (FPCs). Refer to Practices [D1265](#), [D3700](#), and [D6849](#).

Fixed Volume Cylinder

6.9.2.1 Fixed volume sample cylinders are also known as single cavity sample cylinders or spun cylinders. Refer to [Fig. 1](#) and [Fig. 2](#).

**Key**

- 1 pressure gauge
- 2 carrying handle
- 3 magnetic indicator
- 4 valve
- 5 floating piston
- 6 pressure relief device

FIG. 8 Example of Single Floating Piston (Variable Volume) Cylinder (FPC)

6.9.2.2 Valves—Fixed volume sample cylinders are typically available with either one or two valves serving as cylinder inlet and outlet valves. Recommend using two-valve fixed volume sample cylinders due to their ease of cleaning and purging prior to sampling. For repeated use, it is recommended that one-valve fixed volume sample cylinders be used for only one product, due to difficulty in cleaning.

6.9.2.3 Internal Outage (Ullage) Tubes—It is recommend that fixed volume cylinders be equipped with an internal outage (ullage) tube, designed to provide a vapor space of typically 20 % of the cylinder capacity, allowing for liquid thermal expansion. The end of the cylinder fitted with the outage (ullage) tube shall be clearly marked. If the cylinder does not have an internal outage (ullage) tube, use alternative purging and venting procedures to obtain a minimum 20 % ullage in the cylinder. An internal outage tube within a fixed volume sample cylinder may also be known as an ullage tube or dip tube.

Sample Cylinder Usage Considerations

6.9.2.4 The type of sample cylinder used and its materials of construction as well as hoses and fittings can affect the validity of the sample, as well as the accuracy of the analysis.

6.9.2.5 When the observed vapor pressure of the liquid being sampled is close to the line pressure, the reduction in sample pressure associated with the creation of the ullage space may result in phase separation that can make subsequent representative sub-sampling difficult. In these circumstances, an FPC should be used to ensure that the sample is maintained at sufficient pressure to prevent phase separation.

6.9.2.6 Where small concentrations of contaminants need to be quantified or where concentrations of volatile compounds other than the predominant component need to be quantified, an FPC is recommended.

NOTE 2—Practice D3700 describes a recommended practice for obtaining a representative sample of a light hydrocarbon fluid and the subsequent preparation of that sample for laboratory analysis when dissolved gases are present. Use of Practice D1265 using a fixed volume cylinder, will result in a small but predictable low bias for dissolved gases due to the liquid venting procedure to establish the 20 % minimum ullage.

6.9.2.7 Cylinder Construction—Cylinder construction is primarily dependent on the pressure and temperature of the

product to be sampled, and the pressure vessel approval and certification requirements in the jurisdictions in which it is to be used and transported. All sample cylinder material, and equipment used for obtaining the sample, shall meet appropriate standards for construction, cleanliness and suitability for use, including product compatibility. Use corrosion resistant metal sample cylinders certified by the authority having jurisdiction for pressure vessels with adequate pressure rating for the product being sampled. Common materials used are non-magnetic 300-series stainless steel, Monel (trademarked),¹¹ Viton elastomeric components, and possibly other materials. The size of the cylinder depends upon the amount of sample required to perform the anticipated laboratory tests, and to be retained. Blanking cover caps may be provided to seal the valve connection points of high pressure sample cylinders prior to their transportation between the sampling location and the laboratory.

Floating Piston Cylinders (FPC)

6.9.2.8 FPC, also known as a variable-volume cylinder or pressure-balanced piston cylinder, can be used to sample pure and multi-component liquids and when properly operated, maintains the sample as a single-phase liquid. This device uses one, or two pistons, inside of the sample cylinder to maintain separation between the liquid sampled and the backpressure inert gas. The two-piston cylinder is known as the double-piston cylinder (DPC). The inert gas is vented slowly to allow the sample to enter the cylinder while maintaining a constant pressure on the sample. FPCs are typically constructed from a honed metal tube equipped with end caps, valves, piston, a relief device to protect against over-pressure, and a method of displaying the piston position. See Fig. 8, Fig. 9, and Fig. 10.

6.9.2.9 Refrigerated Products—The FPC shall be safely cooled to the temperature of the product being sampled when sampling refrigerated liquids that are near or below atmospheric pressure. Low cargo temperature (more than approximately 15°C below ambient) and low cargo pressure (near atmospheric pressure) may affect the ability of the user to

¹¹ Monel is a trademark of Special Metals Corporation.

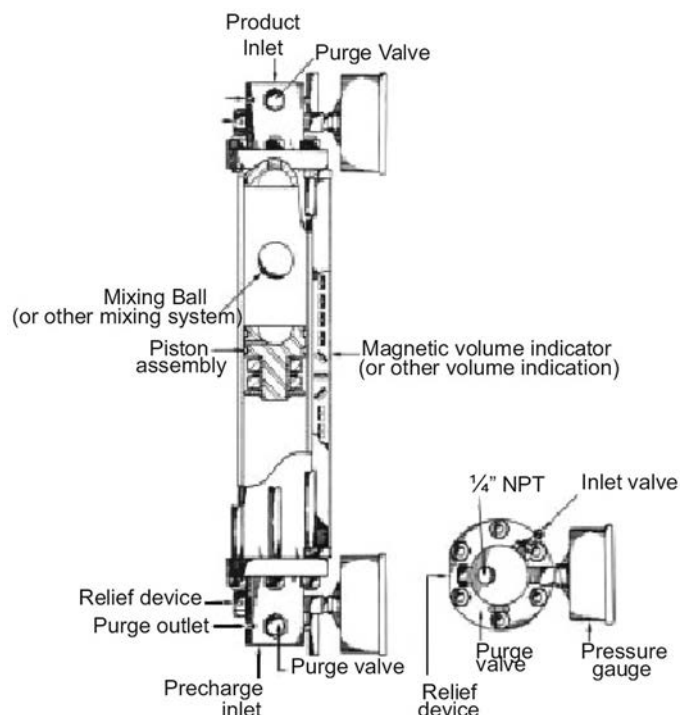
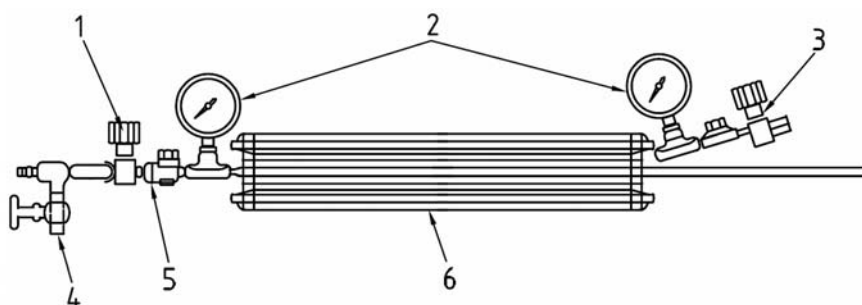


FIG. 9 Example of Single Floating Piston (Variable Volume) Cylinder (FPC)

**Key**

- 1 product inlet (fill) valve
- 2 pressure gauges
- 3 inert gas pre-charge inlet valve
- 4 purge (exhaust) valve
- 5 pressure relief device
- 6 single-piston variable volume sample cylinder

FIG. 10 Example of Single Floating Piston (Variable Volume) Cylinder (FPC)

successfully capture a representative liquid sample using an FPC if additional care is not taken; and the use of a vacuum pump may be required. Refer to Practice D3700 and GPA S 2174.

6.9.2.10 Piston Position Indicator—The FPC shall be equipped with a piston position indicator such as a magnetic follower, piston rod, or equivalent that is used to indicate the sample volume to comply with the maximum percent fill (maximum fill density) allowed for storage and transportation. Do not use FPCs that are not equipped with a piston position indicator without a procedure to allow the operator to verify fill

density immediately after sampling prior to transport. Consult the authority having jurisdiction for acceptable procedures. See Fig. 8 and Fig. 9.

6.9.2.11 Lubricants—Lubricants used to lubricate or seal the floating piston, O ring seals, and other components shall be inert to the product being sampled.

6.9.2.12 Cylinder Coatings—Some cylinders may be internally coated or lined to reduce the chances of bare metal surfaces reacting with trace reactive components, potentially altering the quality and integrity of the sample. For example, be absorbed into the structure of 316 stainless steel, so that testing

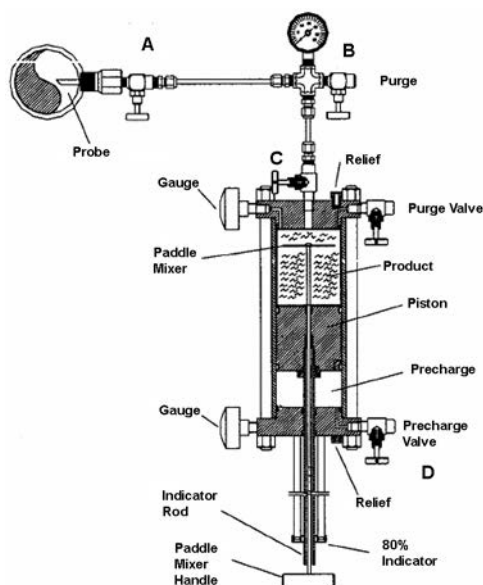


FIG. 11 Example of Typical High-Pressure Sampling System

for H_2S may require the cylinder to be coated. Protective internal coatings or surface treatments are acceptable for FPCs provided that they do not adversely affect the free movement of the piston, or effectiveness of the seals.

Cylinder Pressure Relief

6.9.2.13 Fit a ruptured disk or a self-resetting pressure relief valve to the cylinder to prevent overpressure as the result of liquid thermal expansion. It is recommended to use a spring relief valves if self-resetting is required. Typically, the maximum operating system pressure should be limited to 80 % of the nominal rating of the rupture disk for static operating pressure and ambient temperature. The relief maximum burst pressure shall not exceed the cylinder test pressure. Refer to applicable regulatory requirements for safe filling limits.

6.9.2.14 *Cylinder Pressure Relief Inspection*—The strength of rupture disks can deteriorate with time due to temperature, corrosion, and fatigue. In addition, pulsating pressure, vacuum/pressure cycling, heat, and corrosive fluids and atmospheres can reduce the disk's burst pressure. Relief valves and rupture disks should be inspected regularly. Do not alter valves or safety relief devices that are part of a cylinder permit or exemption.

NOTE 3—The USA has an exemption system, and Canada has a permitting procedure for non-American Society of Mechanical Engineers (ASME) or Department of Transportation (DOT) cylinders.

6.9.2.15 *Cylinder Pressure Relief Release*—The sudden release, typically accompanied by a loud noise and product released at high velocity, might create a hazard. If the sample cylinder pressure exceeds the relief device setting and part of the sample cylinder contents are vented, the composition of the remaining sample is likely different from the original contents. Another sample should be drawn and procedures revised to avoid the circumstances that led to the overpressure condition.

6.9.3 *Cylinder Approval and Rating*—If the cylinder is to be transported, it shall also conform to specifications published in transportation legislation such as U.S. CFR 49 or Canadian

Transportation of Dangerous Goods Regulations, and their supplements, reissues, or similar regulations in other jurisdictions.

6.9.4 *Cylinder Extreme Temperature Considerations*—For safe handling of 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 very cold (e.g. $-40^{\circ}C$ ($-40^{\circ}F$)) product is sampled, the cylinder should be expected to warm considerably before analysis is performed during transport and in the laboratory. During summer months, the temperature of the cylinder and product could reasonably be expected to rise to as high as $46^{\circ}C$ ($115^{\circ}F$) in hot environments. A cylinder initially filled to 80 % of its capacity will be over pressured and the relief device(s) will activate under these conditions. In such an extreme but not uncommon case the cylinder should not be filled more than approximately 60 % during the initial fill. The appropriate industry volume correction factor data and calculations should be consulted to determine the maximum fill for the product being sampled. It is recommended that users work with the manufacturer of these sample cylinders and sample collection systems any time ambient or product temperatures, or both, exceed the range of $-29^{\circ}C$ ($-20^{\circ}F$) to $60^{\circ}C$ ($140^{\circ}F$). Consider the effects of extreme temperature on metal, O-rings, valve seats, seals, gauges, relief devices, sample pump components and other devices and components in the system.

6.9.5 *Mixing Capability*—The cylinder may include a mechanism to mix the sample in the sample chamber in case of stratified mixtures or water haze that may settle after sampling. This mechanism may be a mechanical mixer/vortex plate on a movable rod, a freely moving rolling ball or slider, magnetically coupled stirrer, or similar device. Some designs of FPCs have two pistons, which enable the sample to be mixed within the cylinder (prior to sub-sampling) by repeatedly forcing it through a central mixing device. See Fig. 9 and Fig. 11.

6.9.6 *Sample Cylinder Cleanliness*—To ensure the sample cylinder and its components do not affect the integrity and

quality of the sample obtained, the user shall establish an acceptable cylinder cleaning and preparation process based on experience, and considering the following factors:

- (1) Type of cylinder and design,
- (2) Cylinder material and lining,
- (3) Cylinder components (valves, seals, etc.) and attached tubing/lines,
- (4) Recent history of the cylinder, including repair or last product,
- (5) Product being sampled,
- (6) Product test methods,
- (7) Purity of product being sampled, and concern with trace contaminants, and
- (8) Use of cleaning agents.

6.9.6.1 Sample Cylinder Cleaning and Purging Methods—Based on the above factors any one, or combination, of the following may be deemed as satisfactory cylinder cleaning methods:

- (1) Purging and flushing of the cylinder using a cleaning agent or solvent, for example acetone or methanol. It is recommended to purge and flush the sample cylinder at least three times.
- (2) Purging and flushing of the cylinder using an inert gas, such as helium and nitrogen. Testing of the inert gas present in the cylinder is recommended to confirm no trace of hydrocarbon gas or impurities. It is recommended to purge and flush the sample cylinder at least three times.
- (3) Purging and flushing of the cylinder using the product being sampled. It is recommended to purge and flush the sample cylinder at least three times.
- (4) Steam (not recommended for floating piston cylinders).
- (5) Warming and drying of cylinders.
- (6) Manufacturer's recommendations.

6.9.6.2 Cylinders used in continuous service, for example with specification LPG products or stabilized crude, may not require disassembly or cleaning before each use. For example, it may be acceptable to vent the previous product as a liquid, and remove remaining sample from dead volume by solvent rinsing, evacuation, gas purge, or equivalent procedure. (**Warning**—Disassembly of the piston cylinder for maintenance requires special precautions. If either end cap is removed while pressure is on the cylinder, the end caps and the piston can be ejected with such a force as to cause serious injury to personnel and damage to equipment. Consult the manufacturer's instructions, and regulatory guidelines, for the maintenance and safe disassembly instructions for all cylinders.) Refer to Practice **D6849**.

6.10 Special Sample Cylinders—Specially designed single purpose sample cylinders are often used for non-compositional tests, the testing of trace constituents using specific instruments, and physical property testing devices. Refer to Test Method **D1838**, Test Method **D1267**, API MPMS 14.6, and Test Method **D1657** (API MPMS Chapter 9.2) for details. In addition, spot samples may be tested for some components, including trace analysis using length-of-stain tubes, gas content using lower explosive limit or gas detectors, oxygen content using oxygen detectors and trace oxygen analyzers, and mois-

TABLE 2 Commonly Used Manual Sampling Devices for Liquid Products

Top Fill	Flow Through/Trap	Bottom Fill
Cage/weighted bottle	Zone/Core	Dead bottom sampler (Bacon Bomb)
Weighted beaker	Core/interface	Tube
Tap	Bottom water and ROB/OBQ High pressure cylinders	

ture content using dew point instruments. Refer to manufacturer's instructions and appropriate industry standards for use and applicability.

7. Sampling Devices/Equipment and Accessories

Introduction

7.1 The preferred manual sampling devices and processes are those that do not require the sample to be transferred from the primary sample container to an intermediate container. Sampling devices shall be designed, constructed, and maintained to ensure that they perform the purpose or function for which they are intended. They shall be of sufficient strength to withstand pressures likely to be generated and sufficiently robust to withstand normal handling. Liquid sampling devices shall be leak-tight to maintain the initial characteristics of the sample. Materials of construction for sampling devices and their accessories should be compatible with the product sampled and ensure that there will be no interaction between the product and the sampling device that would affect the integrity of either.

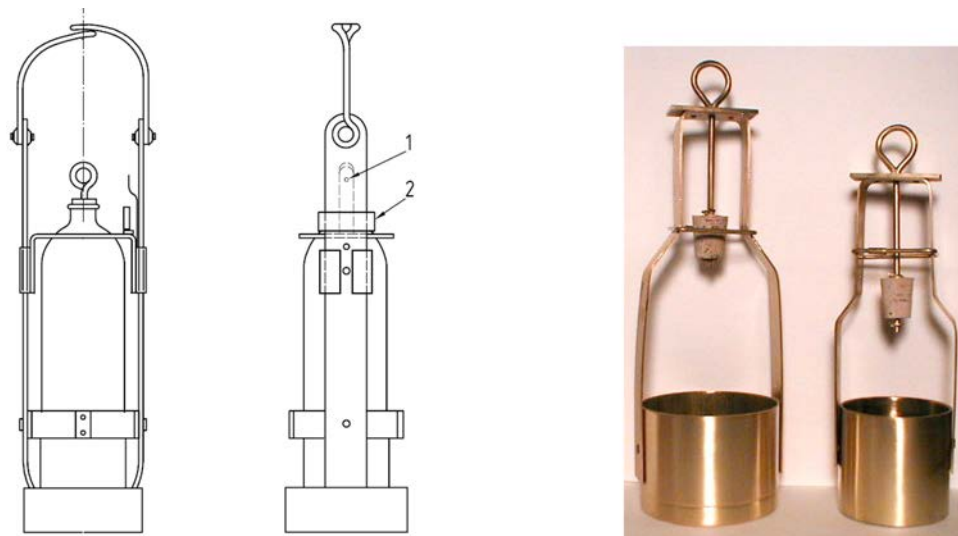
7.2 If there is any question regarding the applicability of the sampling device to a specific product, testing should be performed to verify compatibility. **Table 2** contains a listing of commonly used manual sampling devices for liquid products; categorized by the method by which the sampler is filled.

7.3 Cage Sampler:

7.3.1 A cage sampler shall be constructed of a spark-reducing metal, appropriately sized to hold the container, typically a 1 L (1 qt) bottle. See **Fig. 12**. The lowering and retrieval equipment shall be attached to the cage in such a manner that a stopper can be opened by means of a sharp jerk. A restricting device, such as a cap with a hole drilled in it, may be used to restrict the filling rate. The combined apparatus shall be weighted so as to sink readily in the product to be sampled. Cage samplers may be used to obtain spot, running and all-levels samples.

7.3.2 The use of a sampling cage, or any sampling process that avoids the need to transfer the sample into an intermediate sample container, is preferred for samples used for volatility testing.

7.4 Weighted Bottle—Configure the weighted bottle sampler by attaching a weighted line to the sample bottle as shown in **Fig. 13**. The stopper should be tied to the cord about 150 mm (6 in.) from the neck of the bottle. Attach the lowering device to the bottle in such a manner that the stopper may be opened by means of a sharp jerk of the lowering cord. A restricting



Key

- 1 Swivel point
- 2 Locking piece

FIG. 12 Cage Samplers

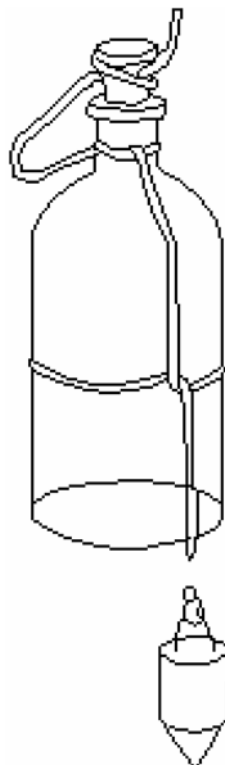
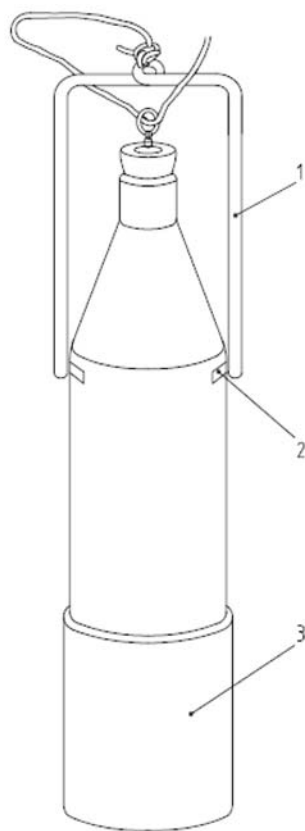


FIG. 13 Weighted Bottle

device, such as a cap with a hole drilled in it, may be used to restrict the filling rate. Weight the combined apparatus so as to sink readily in the product to be sampled. Design shall allow for filling the container at any desired level. A weighted bottle sampler may be used to obtain spot, running, and all-levels samples.

7.5 Weighted Beaker—The sampling beaker shall be constructed of a spark-reducing metal and weighted so as to sink readily in the liquid to be sampled. The lowering and retrieval equipment shall be attached to the sampler in such a manner that a stopper can be opened by means of a sharp jerk. To reduce the difficulty with cleaning the beaker, any weighting

**Key**

- 1 Wire handle
- 2 Wire lugs
- 3 Weighing material

**FIG. 14 Weighted Beakers**

material shall be fixed to the beaker in such a way that it does not come into contact with the sample. Weighted beaker sampler may be used to obtain spot, running, and all-levels samples. Caution shall be exercised to ensure that a residue from prior samplings does not contaminate subsequent samples obtained. Samples obtained using a weighted beaker requires transfer to an intermediate container, potentially affecting the integrity of the sample. See Fig. 14.

7.6 Bottom Water and Remaining On-Board/On-Board Quantity (ROB/OBQ) Sampler/Scraper—Typically constructed of copper or brass tubing, open at the top, designed with a clip on one side, and rounded bottom to allow the sampler to tip over on the tank or vessel compartment bottom. This device may have a removable bottom for cleaning. Sampler fills with product at first, hits the bottom, and falls over. Product rises to the top with free water displacing the product in the sampler. Use the sampler/scraper in a similar manner to sample very small volumes in a tank or OBQ/ROB product on marine vessels including, sludge and semi-liquid or high-viscosity material residue. See Fig. 15.

7.7 Tank Tap Sampling:

**FIG. 15 Example of Bottom Water and ROB/OBQ Sampler/Scraper**

7.7.1 Each tap should be a minimum of 1.25 cm (½ in.) in diameter. Taps with a 2.0 cm (¾ in.) diameter or more may be required for heavy, viscous liquids, for example, those with a relative density of 0.9465 or greater (18.0° API or less). On tanks that are not equipped with floating roofs, each sample tap should extend into the tank a minimum of 10 cm (4 in.). Some sample taps may be equipped with a delivery tube that permits the filling of the sample container from the bottom, as reflected in Fig. 16. Refer to Practice D5842 (API MPMS Chapter 8.4).

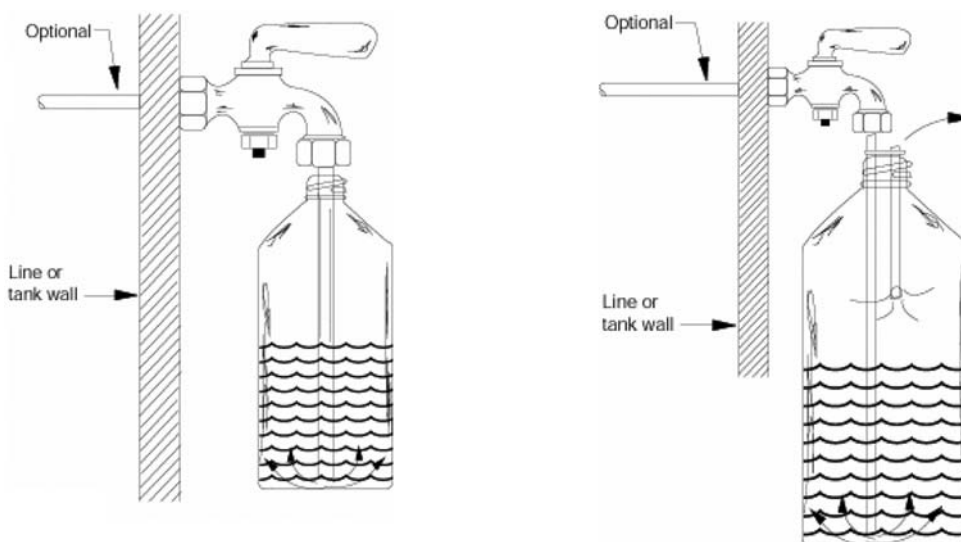


FIG. 16 Tank Sample Taps

7.7.2 Tap Sampling Requirements—At a minimum, a tank should be equipped with a sample tap that is at the elevation of the main tank outlet. For multiple level samples, the tank is to be equipped with at least three sampling taps placed equidistant throughout the tank height, based on normal liquid height operating levels of the tank. Additional sample taps, for example as many as a total of five or six, may be required based on the size of the tank, type of product service, anticipated varying liquid height operating levels, and history of product homogeneity. With reference to the circumferential location, sample taps should be located a minimum of 2.4 m (8 ft) from the tank inlet and 1.6 m (5 ft) from the tank outlet/drain. If a tube is used, take steps to ensure that the tube will not contaminate the product being sampled.

7.8 Pipeline Manual Tap Sample Probe:

7.8.1 A sample probe in a pipeline is used to direct sample from the flowing stream. Manual tap sample probes are often used for product identification, testing, and calibration of online instrumentation.

7.8.2 Probe designs that are commonly used are shown in Fig. 17 and described in the following:

7.8.2.1 Fig. 17A—A tube beveled at a 45° angle;

7.8.2.2 Fig. 17B—A short radius elbow or pipe bend. Chamfer the probe end on the inside diameter to give sharp entrance edge; and

7.8.2.3 Fig. 17C and Fig. 17D—A closed-end tube with a round orifice spaced near, but not at, the closed end.

7.8.3 Pipeline Manual Tap—Free Water/Haze Verification—If the sample system is designed such that the sample point on the pipeline is primarily intended to ascertain the presence of water possibly riding the bottom of the pipe at a particular point, or intended to detect possible particulate contamination, for example, such as pipeline origin and delivery points and pump stations, the sample point may be installed such that the sample is drawn directly from the bottom of the pipe using no internal probe.

7.9 Pipeline Sampler and Probe Location:

7.9.1 Since the product to be sampled may not always be homogeneous, the location, position, and size of the sample probe should minimize any separation of water and heavier particles that would make their concentration different in the gathered sample than in the main stream. It is recommended that the probe be in a horizontal plane to prevent drain back of any part of the sample to the main stream and located in an upward flowing vertical run of pipe. The probe may also be located in a horizontal run of pipe. Extend the sample probe into the center third of the pipe with the inlet facing up stream. The flowing product at the point of sampling should be adequately mixed. This can be accomplished from the normal flowing turbulent velocity, together with an in-line static mixer; typically either a series of baffles or a perforated plate. If the line is not equipped with a static mixer, it is recommended to locate the sample point for spot samples just downstream from common line equipment that may facilitate mixing, such as 45° elbows, control valves, manifolds, strainer baskets, meters, and provers. If using the sample point for custody transfer purposes, refer to requirements as listed in Practice D4177 (API MPMS Chapter 8.2).

7.9.2 Sample lines, used in conjunction with probes, should be as short as practical and purged before each sample is taken, to avoid sample contamination.

7.9.3 When sampling heated or high-viscosity liquids, it may be necessary to heat the sample line, valves, and sample container to a temperature sufficient to keep the product in a liquid state to ensure accurate sampling. To control the rate at which the sample is withdrawn, the probe should be fitted with valves or plug cocks. Extreme caution should be exercised at all times because of the higher probability of line plugs as a result of solidified product.

7.10 Zone Sampler (Core Sampler):

7.10.1 A zone sampler, also known as a core sampler or a core-type sampling thief, shall be designed and constructed such that, when lowered slowly, it is capable of trapping a vertical column of liquid at any selected level below the

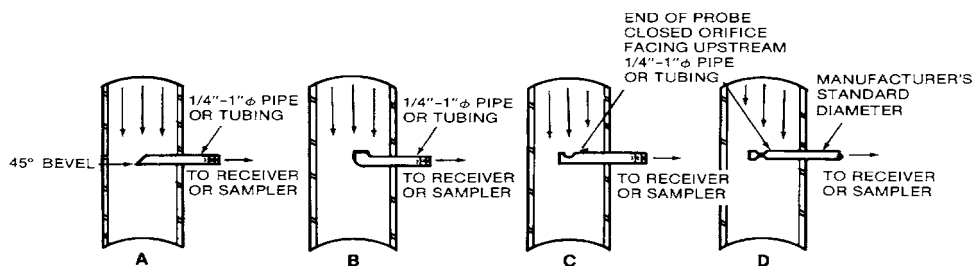


FIG. 17 Probe Designs

surface to within 2.0 to 2.5 cm ($\frac{3}{4}$ to 1 in.) of the bottom of the tank. A zone sampler shall consist of a tube made of spark-reducing metal, glass, or plastic material open at both ends to allow free flow of liquid through the sampler during lowering. See Fig. 18 and Fig. 19.

7.11 The closing of the lower end of the tube to trap the sample at the desired level may be achieved by various means including, but not limited to, the following:

7.11.1 A closure mechanism actuated by an extension rod or sharp jerk on the cord;

7.11.2 Simply raising the sampler causing the top and bottom flap valve to close;

7.11.3 A weight falling down a suspending cable to actuate the closure mechanism, and;

7.11.4 The zone sampler shall be capable of penetrating the product in the tank and being withdrawn without undue contamination of the contents.

7.12 A zone sampler (see Fig. 18) may include the following features:

7.12.1 Extension rods for use in obtaining samples at levels corresponding with requirements for high-outlet connections or samples to determine high settled sediment and water levels;

7.12.2 Sample cocks for determining the height of sediment and water in the sampler core, as well as to place the sample directly into the testing apparatus. Refer to MPMS Chapter 18.1;

7.12.3 A clear cylinder that facilitates observing the gravity and temperature of the oil during a gravity test;

7.12.4 A cord marked so that the sample can be taken at any depth in the vertical cross section of the tank; and

7.12.5 A hook to hang the sampler in the hatch vertically.

7.13 *Dead Bottom Sampler*—Dead bottom samplers, such as bacon bombs, normally have a projecting stem that opens the inlet valve as the stem strikes the bottom of the tank. The sample enters the sampler through the bottom valve and air is released simultaneously through the top valve. The inlet valve automatically closes when the sampler is withdrawn. See Fig. 20. The dead bottom sampler can also be equipped with extension rods, typically 7.6 cm (3 in.), 15 cm (6 in.), and 30.5 cm (12 in.) to obtain samples from those points in a tank.

7.14 *Tube Sampler—Drum or Barrel*—Tube samplers are applicable for sampling liquids and semi-liquids in drums, barrels and cans. The tube is made of glass, plastic, or metal and is designed so that it will reach to within approximately 3 mm ($\frac{1}{8}$ in.) of the bottom of the container. The capacity of the tube can vary from 500 mL (1 pint) to 1 L (1 qt). A metal tube

suitable for sampling 189-L (50 gal) drums is shown in Fig. 21. Two rings soldered to opposite sides of the tube at the upper end are convenient for holding it by slipping two fingers through the rings, thus leaving the thumb free to close the opening.

7.15 *Dipper/Ladle Sampler*—The dipper will typically have a flared bowl and a handle of conventional length made of material such as tinned steel that will not affect the product being tested. The dipper shall have a capacity suitable for the amount to be collected and be protected from dust and dirt when not being used.

7.16 *Auger Sampler*—The auger sampler is applicable for sampling waxes and soft solids in drums, cases, bags, and cakes when they cannot be melted and sampled as liquids. The auger should be 2 cm ($\frac{3}{4}$ in.) in diameter (preferred), similar to that shown in Fig. 22, and of sufficient length to pass through the material to be sampled.

7.17 *Pail and Scoop Sampler*—Pail and scoop (grab) sampling is applicable for all lumpy solids in bins, bunkers, freight cars, drums, bags, boxes, and conveyors.

7.18 *Accessory Equipment*—Ropes, chains, and cables, which could be on a reel, are used to raise and lower sampling devices. This equipment shall be clean ensuring that no residue from previous use contaminates the sample being taken. Do not leave samplers suspended in a tank when not in use, as electrical continuity (bonding) may be lacking. While Section 5 provides some guidance regarding conductivity and safeguards, there may be specific and unique requirements with regard to conductivity, grounding, and bonding of some sampling equipment. Refer to the manufacturer's instructions and facility and regulatory requirements for such guidance.

7.18.1 *Rope*—Do not use ropes made of synthetic fibers. They shall be made of cotton or other non-static generating material and should include some means of determining the level of insertion into the tank or vessel. The rope may have a swivel clasp to link with the attached sample device designed to minimize twisting.

7.18.2 *Chain*—Chains shall be made of brass or other spark-reducing material. The chain should have a swivel clasp to link with the attached sample device designed to minimize twisting. Caution should be exercised using chains because electrical continuity cannot be guaranteed.

7.18.3 *Cable*—Cables shall be made of spark-reducing material. The cables shall have a swivel clasp to link with the attached sample device designed to minimize twisting.

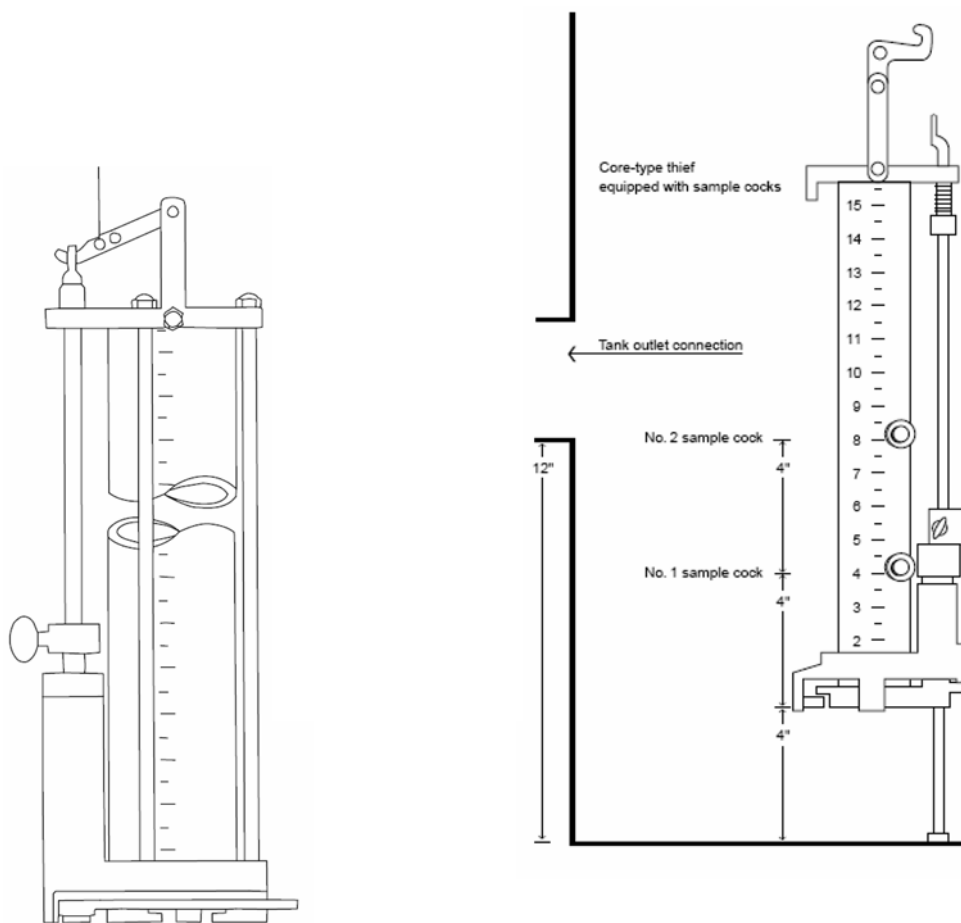


FIG. 18 Example of Core/Interface Samplers—Thief

7.18.4 Rod—Used primarily for shallow-depth sampling such as with truck and rail cars. It consists of a bottle cage attached to the end of a rigid pole or rod made from spark-reducing material.

7.18.5 Bonding Cable—Conductive cable used to provide electrical continuity between the sampling device, funnel, and/or primary container and a suitable ground point.

7.18.6 Retail Dispenser Nozzle Adapter—A nozzle extender, made from spark-reducing material, is designed to be fitted onto the end of a gasoline or diesel fuel nozzle. The adapter is equipped with a delivery tube that permits filling of the sample container from the bottom. See Fig. 23.

7.18.7 Other Equipment—A funnel may be used to transfer product from the sampling device to intermediate sample containers. Also, a graduated cylinder or other measuring device of suitable capacity is often required for determining sample quantity in many of the sampling procedures and for compositing samples. All accessory equipment used shall be clean, and not affect the integrity of the samples obtained.

8. Manual Sampling Concepts and Objective

8.1 Objective of Manual Sampling—The objective of manual sampling varies. In some instances, the intention is to obtain a small portion of product that is representative of the tank or container contents. In other instances, samples are specifically intended to represent product only at that one

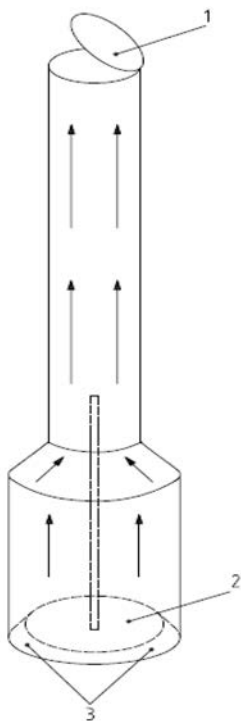
particular point in the tank, such as a top, dead bottom, or suction level sample. When a tank is determined to be homogenous, a series of spot samples may be combined to create a composite sample. Precautions should be taken to maintain the integrity of the sample by preventing it from being contaminated by the sample point, the sample apparatus and equipment, the cleanliness of the container, the weather, and sample transfer operations. Manual sampling may be applied under all conditions described in this practice. Alternative sampling procedures may be used as agreed upon by all interested parties.

8.2 Training—Personnel shall be trained for the sampling to be performed, including topics such as sample integrity, safety, and special handling related to specific tests.

8.3 Physical and Chemical Property Tests:

8.3.1 The sampling procedure, sample container, quantity required, and sample handling requirements will be based on the test(s) to be performed, sample instructions related to a custody transfer and sample retention requirements.

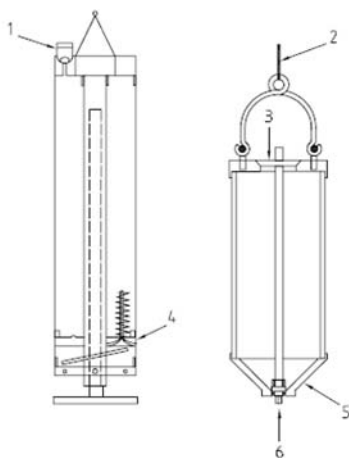
8.3.2 In many liquid manual sampling applications, the product to be sampled contains a heavy component (such as free water) that tends to separate from the main component. In these instances, it should be recognized that sampling will likely result in varying sample quality until such time as the heavy component completely settles out. This period could



Key

- 1 Upper flap valve
- 2 Bottom valve
- 3 Product flows through as sampler is lowered through the liquid

FIG. 19 Example of Core/Zone Samplers



Key

- 1 Ball valve/air outlet
- 2 Line for lowering
- 3 Air outlet
- 4 Spring-loaded inlet valve
- 5 Removable screw cap—facilitate cleaning
- 6 Weighted inlet valve (The optional extension rods trigger the opening and closing of the inlet valve based on height of rod.)

FIG. 20 Example of Dead Bottom Samplers

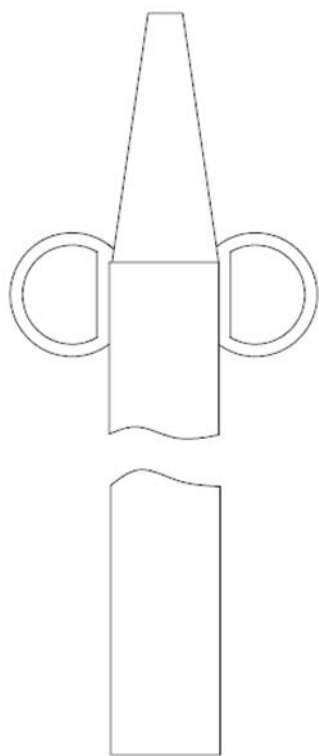


FIG. 21 Tube Sampler



FIG. 22 Auger Sampler



FIG. 23 Example of Nozzle Extender

range from only a few minutes to several weeks based on, but not limited to, the product, temperature, agitation and the use of chemical additives.

9. Sampling Requirements, Considerations, and Procedures

9.1 *General*—This section provides requirements, recommendations and procedures for sampling preparation, sampling, and sample handling. More than one sampling method may provide satisfactory samples. A summary of the manual sampling procedures and their applications is presented in [Table 3](#).

9.2 Sampling Requirements and Considerations:

9.2.1 *Sample Request*—The sampling process is often initiated with a sample request. The request should provide adequate details and information necessary to establish the sample procedure, device, container, sample quantity, and sample handling. To assess sampling requirements, the following information should be established:

- (1) Requestor contact information;
- (2) Date of request and required timeline;
- (3) Batch/job/vessel/voyage number;
- (4) Product description—Applicable safety data sheet (MSDS) and Department of Transportation (DOT) information should be provided or available;

TABLE 3 Typical Sampling Procedures and Applicability

Application	Type of Containment/Vessel/Tank	Procedure
Petroleum liquids	Storage tanks, tank cars, tank trucks	Bottle sampling Zone/Core sampling Tap sampling High pressure cylinder sampling
	Marine vessels	Bottle sampling Zone/Core sampling Automatic sampling High pressure cylinder sampling
	Pipelines	Automatic sampling Manual pipeline sampling High pressure cylinder sampling
Petroleum liquids—water/sediment—bottom sampling	Storage tanks, marine vessels, tank cars, and tank trucks	Core sampling Bottom water and ROB/OBQ sampler
Petroleum liquids—water/sediment—bottom sampling	Storage tanks with taps	Tap sampling
Petroleum liquids	Drums, barrels, cans	Tube sampling
Petroleum liquids/water	Free or open discharge streams; open tanks or kettles with open heads; tank cars, tank trucks, drums	Dipper sampling
Petroleum liquids/water	Free or open discharge streams	Dipper sampling
Asphaltic and bituminous materials	Storage tanks, marine vessels, tank cars, lines, packages	Core sampling Tap sampling ^A Throw-away container sampling
Waxes, solids, bitumens, other soft solids	Barrels, cases, bags, cakes	Boring sampling
Petroleum coke, lumpy solids	Freight cars, conveyors, bags, barrels, boxes	Grab sampling
Greases, soft waxes, asphalts	Kettles, drums, cans, tubes	Grease sampling

^A Refer to Practice D140.

(5) Product location, tank or vessel number(s), and volume(s);

(6) Test name(s) and method(s), and;

(7) Special handling requests or precautions, such as splitting of samples, additional retention requirements, or suspected product stratification. In some cases, a test method may require a special container, a minimum amount of sample, or a special sample handling procedure. At times, multiple test methods may have conflicting requirements, such as volatility testing and homogenizing for S & W. In these instances, it may be necessary to obtain separate samples for each test.

9.2.2 Equipment and Sample Container Preparation—Use only sampling equipment and containers that are resistant to solvent action by the product handled. Inspect all sampling equipment, including container, caps, lids, and stoppers to ensure that they are clean and dry. Any residual material left in a sampling device or sample container may contaminate the sample. It is recommended to rinse the sample container and equipment with the product to be sampled before drawing samples.

9.2.3 Sample Transfers and Handling Requirements:

9.2.3.1 The number of transfers from the primary container to intermediate sample containers and the testing apparatus should be minimized to maintain the product integrity and representation. Common areas of concern related to transfers include loss of light ends, water, and heavy component

disparities and possible residue contamination. Some of the tests affected might include, but are not limited to, flash point, vapor pressure (RVP), density, S & W, product clarity, ash, trace metals, and micro-separometer (MSEP).

9.2.3.2 Each time a sample is transferred, including to the test apparatus, the sample should be completely mixed to ensure it is homogenized, unless the specific test method allows or specifies otherwise. The transfer should be completed during the period that the sample remains homogenous. There may be specific practices that allow or specify the sample being placed directly into the test apparatus, such as from the thief via petcock into the centrifuge tube for small crude gathering tanks. Refer to API MPMS Chapter 18.1.

9.2.4 Requirements for Mixing and Compositing Individual Samples:

9.2.4.1 When compositing samples (e.g. a multiple compartment composite sample for a marine vessel or a tank composite sample for a shore tank), each primary sample should be thoroughly mixed before pouring off into the intermediate composite container. Based on the product, and as described in the applicable test methods, adequate mixing may be obtained by shaking (manual or mechanical) or through the use of a power mixer, also known as a homogenizer. Higher viscosity products, such as residual fuel oil, and IFO 380 bunker fuel, and most crude oils should be homogenized before pouring into the composite sample, and into the test apparatus.

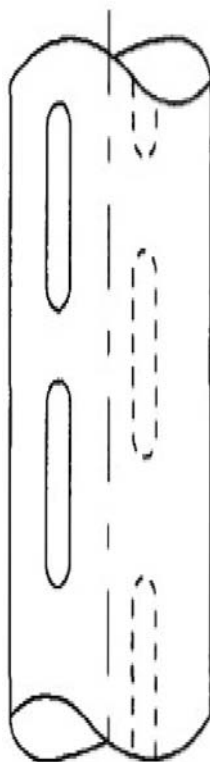


FIG. 24 Slotted Standpipe

Manual and mechanical shaking of higher viscosity products, such as residual fuel oil, IFO 380 bunker fuel and most crude oils, may not impart sufficient energy to create and maintain a homogeneous representative sample. The specific mixing or homogenizing method should be recorded. Refer to Practice **D5854** (API MPMS Chapter 8.3). There may be job-specific instructions to retain a portion of each individual sample.

9.2.4.2 With the intent that the final composite sample be representative of the combined parcels, compartments or components represented in the field, make the blend proportional to the quantities of each parcel, compartment, or component. The type of composite sample should be clearly marked on the sample tag and the analytical report, for example, “Vessel Volumetric Composite” or “Shore Tank Volumetric Composite.”

9.2.5 *Container Outage (Ullage)*—Because of potential liquid thermal expansion, do not fill sample containers more than 70 to 85 % of the containers capacity, allowing room for expansion based on the temperature of the liquid at the time of filling and the probable maximum temperature to which the container may be subjected. If required by the test method, the sample ullage may be greater than 85 %, but in no case shall it be completely full. In these cases, take special handling precautions to consider the hazards associated with product thermal expansion. For safe fill of high-pressure cylinders, refer to maximum fill density and 9.30.

9.2.6 *Standpipes:*

9.2.6.1 Shore tanks, particularly floating-roof tanks, and vessel compartments are often fitted with standpipes. Often these standpipes are unslotted and sometimes poorly or inadequately slotted preventing the standpipes from being flushed

out adequately during filling or tank circulation. Samples shall not be obtained from solid or unslotted standpipes, since it is unlikely that the samples will be representative of the tank or vessel compartment contents. Sampling through poorly or inadequately slotted standpipes may also result in unrepresentative samples. Additionally, in the case of both shore tanks and marine vessels, samples obtained from all standpipes may become contaminated from the presence of rust, foreign matter, moisture (condensation), and residue from prior products remaining on the standpipes. See Fig. 24.

9.2.6.2 In all cases, when possible, obtain samples directly from the tank or vessel compartment rather than through a standpipe. If sampling is performed through a slotted or perforated standpipe, it shall have either two rows of slots or two rows of holes (that is, perforations) located on the opposite sides of the pipe, perpendicular to the shell wall, that starts at the lower end of the pipe and continue to above the maximum liquid level. Typical sizes of the slots are 2.5 cm (1 in.) in width and 25 cm (10 in.) in length. The typical diameter of the perforation is 5 cm (2 in.). The maximum spacing between perforations or slots if not overlapping shall be 30 cm (12 in.).

Non-homogeneous (Stratified) Product

9.2.7 A tank, marine vessel compartment, or batch may not be homogenous for many reasons, some of which may include:

9.2.7.1 *Water in Petroleum*—The concentration of dispersed water in the oil is generally higher near the bottom of a tank or pipeline. A running or all-level sample, or a composite sample of the upper, middle, and lower samples, may not be representative of the concentration of the dispersed water present. The interface between oil and free water may be difficult to locate,

especially in the presence of emulsions, layers, or water-bearing sediments. The free water level may vary across the tank bottom surface. Additionally, the bottom may be covered by pools of free water or water/oil emulsion impounded by layers of sediments or wax.

9.2.7.2 Layers of varying density or viscosity, for example, residual fuels with blend components such as light cycle oil. This will often lead to non-homogenous test results of other properties such as ash, metals, pour point or S & W.

9.2.7.3 Layers of varying volatility and light components impact properties such as vapor pressure, distillation, or flash point. Examples include distillate mixed in gasoline, gasoline mixed in distillate, kerosene in diesel fuel oil, crude oil blended into residual fuel oil or varying components such as naphtha and reformat blended into gasoline.

9.2.7.4 Liquids coexisting in a tank with solid or semi-solid materials typically referred to as “bottoms,” for example, crude oil and heavy fuel oils.

9.2.8 Depending on the extent of stratification, it can be very difficult to obtain a representative manual sample. Spot samples, such as upper, middle and lower (UML), or top, middle and bottom (TMB) are recommended to establish the extent of stratification. Running or all-levels samples will contain all product layers in the vertical column from the sample point, although the rate of fill will be variable based on the depth of the product. Even with care, it may not be possible to exactly reproduce a manual sample from a non-homogeneous or stratified tank.

9.2.9 Spot samples may be taken to assess the level of stratification of a particular property within a shore tank or marine vessel compartment. The determination as to what constitutes stratification should be based on agreement of interested parties. In addition, the test method precision, or laboratory site precision may be considered as aids in determining if the analytical results differ significantly enough to indicate stratification. Refer to Practice D6299 and Guide E882. As agreed between interested parties, either individual spot sample test results can be averaged, a volumetric composite sample can be prepared and tested, or an alternate sample or sample location may be agreed upon.

9.2.10 If available, a validated automatic flow proportional pipeline sampler is preferred for non-homogenous products. An automatic sampler equipped with a mixing device immediately upstream of the sampler is particularly well suited to ensure mixing and water dispersion for heavy products and crude oils.

9.2.11 If an auto in-line sampler satisfying Practice D4177 (API MPMS Chapter 8.2) is not available, and manual samples are the only available option, the interested parties should consider agreeing upon the following:

9.2.11.1 *Additional Sampling and Testing*—To assess the level of stratification, draw spot samples, such as top, upper, middle, lower, and suction levels and test them individually for API gravity or density, viscosity, S & W, vapor pressure (RVP), Flash Point, or any other property of interest. Based on the differences of these properties relative to the test method precision, the extent of stratification can be determined. If the tank is considered to be non-homogeneous for the properties

tested, additional spot samples may be taken at equidistant levels, and all the individual test results mathematically averaged. If the tank is determined to be homogenous individual samples obtained, such as those referenced above, should be representative. See 9.2.9.

9.2.11.2 *Sample Downstream of Stratification*—If available for consideration and agreeable by interested parties, the product may be sampled following the transfer (for example, marine vessel load or discharge or tank transfer). The product in the receiving tanks or compartments may be mixed adequately as a result of the transfer. Sampling and testing of properties of interest should be performed on the product to ensure it has been thoroughly mixed and homogeneous.

9.3 Marine Cargoes:

9.3.1 Interested parties should agree upon sampling instructions and requirements prior to any transfer, including what sample(s) will be considered the custody transfer sample. Considering terminal, vessel and regulatory requirements, the following is an example of typical manual samples that might be obtained for certain marine movements:

9.3.1.1 Running or all-levels samples from the shore tank before a vessel load and before and after a vessel discharge. Additional samples, such as spot samples, obtained based on specific requests from interested parties.

9.3.1.2 Running or all-levels samples from each vessel compartment after a load and before a discharge. Additional samples, such as spot samples, obtained based on a specific request from interested parties. Marine vessel samples may be taken either through open hatches or via vapor control valves (VCVs).

9.3.1.3 Manual samples may be obtained from the shore pipeline, or vessel manifold, typically at the start of the transfer as specifically requested by interested parties.

9.3.2 *Deck Composites*—Periodically field “deck composite samples” are requested. A deck composite is typically made by compositing a portion of each sample obtained from all vessel compartments containing a particular product or crude oil grade. The samples are not normally homogenized before compositing and are typically not volumetrically composited based on the quantity in each vessel compartment. It is for these reasons that deck composite samples are typically not considered representative of the product sampled. The sample label shall clearly read “deck composite sample.” Deck composite samples are not recommended for volatility testing, such as Vapor Pressure, as described in the applicable test methods.

9.3.3 *Sampling High Pressure Products on Marine Vessels*—High pressure sampling procedures will likely be affected by the type and design of the marine vessel. Marine vessels transporting high pressure cargoes are generally classified into five groups: fully pressurized, semi-pressurized (semi-refrigerated), ethylene, fully refrigerated, and LNG. A special consideration is that barges are generally fully pressurized and are not fitted with cargo pumps, but are instead unloaded with vapor displacement from a shore compressor. It may be necessary to sample the gas phase in addition to the liquid phase, such as when the cargo tank had contained inert gas or a different cargo, or when the possibility of contamination exists. For some cargoes, the previous cargo vapors, the

inert gas or other contaminants in even small concentrations can adversely affect the new cargo. The cargo sample may be used for composition, density or specification limits. Refer to API MPMS 17.10.2.

9.4 Sample Labeling:

9.4.1 The label or sample tag shall satisfy applicable regulatory, company, and custody transfer requirements. Use waterproof and oil-proof ink or a pencil hard enough to dent the tag. Soft pencils and ordinary ink markers are subject to obliteration from moisture, oil smearing, and handling. Label or tag the container immediately after a sample is obtained. See Fig. 25.

9.4.2 Many sample containers require special shipping packaging before they can be transported from the point of collection. All employees handling and shipping samples shall be familiar with applicable requirements and regulations.

9.4.3 The following information is typical on a label:

- (1) Unique job identification (ID)
- (2) Date and time sample was taken
- (3) Product name/description
- (4) Facility
- (5) Tank/vessel/pipeline identifier or description
- (6) Type of sample
- (7) Before or after: load/discharge/transfer—sample only
- (8) Name of individual who obtained the sample
- (9) Applicable hazard communication information
- (10) Special conditions such as rain or snow while sampling

9.4.4 *Sampling Report and Chain of Custody*—In addition to the sample label/tag, a sample report and chain of custody report are often required describing the samples taken. At any point when samples are handed over to another responsible party, whether the laboratory technician or someone transporting the samples, it is recommended that the transfer of samples be documented, for example, on a chain-of-custody form. Refer to Guide D4840.

9.4.5 *Samples for Volatility Testing*—It is recommended that samples used for volatility testing not be transferred into an intermediate sample container. If the sample is transferred from the primary sample container, it should be transferred to the intermediate sample container without delay. Keep the container closed except when the container is being filled. After delivery to the laboratory, these samples should be cooled before the containers are opened. Refer to Practice D5842 (API MPMS Chapter 8.4).

9.4.6 *Sample Storage and Disposal*—Samples should be stored in such a manner as to enable quick retrieval and kept safe and secure to prevent theft, tampering, weathering, or degradation from light, heat, cold, and any other conditions. Sample containers should have adequate caps to prevent loss of light ends. Store and dispose of samples in such a way to ensure compliance with company policy and regulatory requirements.

Sampling Methods and Procedures

9.5 Running and All-Levels Sampling:

9.5.1 Obtain a running sample by lowering an open sampling device to the bottom of the outlet suction level, but

always above the free water, and returning it to the top of the product at a uniform rate such that the sampling device is between 70 and 85 % full when withdrawn from the product. If required by the test method, the sampler may be greater than 85 % full when withdrawn but in no case shall it be completely full. In these cases, take special handling precautions to consider the hazards associated with product thermal expansion.

9.5.2 Obtain an all-levels sample by lowering the closed sampling device to the bottom of the outlet suction level, but always above the free water, then opening the sampler and raising it at a uniform rate such that it is between 70 and 85 % full when withdrawn from the product. Alternatively, all levels samples may be taken with samplers designed for filling as they pass downward through the product. If required by the test method, the sampler may be greater than 85 % full when withdrawn but in no case shall it be completely full. In these cases, take special handling precautions to consider the hazards associated with product thermal expansion.

9.5.3 The running and all-levels methods have the advantage of sampling the full column of liquid as compared to spot samples that individually represent a single spot in the vertical column. However, running and all-levels samples may not necessarily be representative because the tank volume may not be proportional to the depth and the rate of filling is proportional to the square root of the depth of immersion.

9.5.4 *Running and All-levels Sampling Procedure*—The running and all-levels sample procedure is as follows:

(1) Inspect the sample container, typically a bottle or beaker, for cleanliness and use only clean, dry equipment.

(2) Place the container in a sampling cage or attach the weighted line to the bottle.

(3) Based on the height and viscosity of product, a restricted flow cap, a notched cork, or a restricted opening may be used to ensure the container emerges between 70 and 85 % filled. Refer to Table 4 for recommended opening sizes.

(4) For a running sample, lower the sampling device at a uniform rate to the bottom of the outlet suction level, while staying above the free water level, and without hesitation, raise it such that the sampling device is between 70 and 85 % full when withdrawn from the liquid.

(5) For an all-levels sample, lower the closed sampling device to bottom of the outlet suction, while staying above the free water level. Pull out the stopper with a sharp jerk of the sample line and raise it at a uniform rate such that it is between 70 and 85 % full when withdrawn from the liquid. Alternatively, all-levels samples may be taken with samplers designed for filling as they pass downward through the product.

(6) Verify that the volume of sample is 70 to 85 % full. If not, discard the sample and repeat the sampling procedure, adjusting the restriction and or the sampling rate. Also, if the sample device opening enters the free water zone of any tank or vessel compartment, discard the sample and repeat the running or all-levels sampling procedure staying above the free water.

Sample Identification No.	
Product Name / Grade	
Terminal, Station or Lease	
Sampling Date and Time	
Gauger	
Type of Sample:	
<input type="checkbox"/> All-Level	<input type="checkbox"/> Running
<input type="checkbox"/> Bottom	<input type="checkbox"/> RVP
<input type="checkbox"/> Clearance	<input type="checkbox"/> Top
<input type="checkbox"/> Composite	<input type="checkbox"/> UML
<input type="checkbox"/> Line	<input type="checkbox"/> 1-Foot
<input type="checkbox"/> Outlet	<input type="checkbox"/> Other: _____
Type of Sample:	
<input type="checkbox"/> Barge Name	
<input type="checkbox"/> Pipeline Batch No.	
<input type="checkbox"/> Railcar No.	
<input type="checkbox"/> Ship Name	
<input type="checkbox"/> Tank No.	
<input type="checkbox"/> Truck No.	
<input type="checkbox"/> Other	
Lab / Job Reference	
Date & Time in Lab	
Technician	

CUSTOMER

FRONT OF TAG

PRODUCT DESCRIPTION _____ UN #/CAS # _____

TANK: # _____ SHORE / VESSEL _____ DATE _____ TIME _____

GAUGE: _____ FT. _____ IN. _____ BEFORE/AFTER= / =LOAD/DIS/TRANSFER

VESSEL _____ LOCATION _____

INSPECTOR _____ TESTED/RETAINED _____

JOB REF # _____ LAB REF # _____

CLIENT(S) _____ OFFICE _____

SAMPLE TYPE: ALL LEVEL/RUNNING/LOAD ZONE/UPPER/MIDDLE/LOWER/TOP/SPIGOT/LINE

COMPOSITE SUBMITTED / SPECIFY OTHER: _____

BACK OF TAG

SAMPLE HAZARD WARNINGS	FOR CHEMICAL EMERGENCY	
	SPILL, LEAK, FIRE, EXPOSURE, OR ACCIDENT	
	CALL CHEMTREC • DAY OR NIGHT	
	*800-424-9300	
	<input type="checkbox"/>	FLAMMABLE
	<input type="checkbox"/>	TOXIC
	<input type="checkbox"/>	CORROSIVE
	<input type="checkbox"/>	OXIDIZER
	<input type="checkbox"/>	IRRITANT
	<input type="checkbox"/>	H ₂ S
DO NOT GET IN EYES, ON SKIN, BREATHE VAPORS & KEEP AWAY FROM FLAME		







FIG. 25 Examples of Sample Labels/Tags

(7) If sample will remain in the primary container, remove the sample from the cage, wipe the outside, cap tightly, and clearly label.

(8) If sample is to be transferred from the primary sample container, empty the contents into an intermediate sample container, using a clean funnel if necessary, cap tightly, and

TABLE 4 Recommended Weighted Sampling Bottle or Beaker Openings

Material	Centimetres
Light lubricating oils, kerosenes, gasolines, diesel fuels, distillates	2
Heavy lubricating oils, nontransparent gas oils (such as vacuum gas oil)	4
Light crude oils less than 43 cSt at 40°C	2
Heavy crude and fuel oils	4

clearly label. Take precautions to protect the integrity of the sample while transferring during inclement weather.

9.6 Spot Sampling:

9.6.1 A spot sample is one taken at a specific location in a tank or from a flowing stream in a pipe at a specific time. It is recommended to use spot samples, such as upper, middle, and lower (UML) or top, middle, and bottom (TMB) to determine the extent of stratification, or to determine the quality of product at a specific location, in the vertical column being sampled. Compositing of spot samples should be performed only when the tank is known to be well mixed and homogenous or it is established that the spot samples are representative of the extent and levels of stratification in the entire tank or vessel compartment. Caution should be exercised when compositing spot samples since they represent product from only those respective points in the tank or vessel compartment and may not represent levels of significant stratification at any other point in the tank or marine vessel compartment, for example, S & W in crude oil.

9.6.2 Spot Sampling Procedure:

- (1) Inspect the sampling device (typically a bottle or beaker) for cleanliness and use only clean, dry equipment.
- (2) Determine the liquid level in the tank.
- (3) Prepare the sampling device, for example place the container in a sample cage, or attach the weighted line to the bottle.
- (4) Insert the stopper in the sample device.
- (5) Lower the sampling assembly to the required location. When samples are required at more than one level, obtain the samples beginning at the top and working downwards to minimize disturbance to the product in the vertical column being sampled. See [Table 5](#).
- (6) At the required location, pull out the stopper with a sharp jerk of the sample line.
- (7) Allow sufficient time for the sampling device to fill completely at the specific location.
- (8) Withdraw the sampling device.
- (9) Verify the sampling device is completely full. If it is not full, empty the sampling device and repeat the sampling procedure.
- (10) If sample will remain in the primary sample container, remove it from the cage, discard approximately 20 % of the sample, wipe the outside, cap tightly, and clearly label.
- (11) If the sample is to be transferred from the primary sample container, the intermediate container should be filled between 70 and 85 %, using a clean funnel if necessary, cap

TABLE 5 Spot Sample Minimum Requirements

Liquid Level	Number of Samples		
	Upper	Middle	Lower
Liquid level ≤ 3 m (≤ 10 ft)		X	
Liquid level >3 and ≤ 6 m (>10 and ≤ 20 ft)	X		X
Liquid level >6 m (>20 ft)	X	X	X

tightly, and clearly label. Take precautions to protect the integrity of the sample while transferring during inclement weather.

9.6.3 Core (Thief) Sampling Procedure:

(1) Inspect the sampler (thief), graduated cylinder (if used), and sample container for cleanliness and use only clean, dry equipment.

(2) Determine the liquid level in the tank.

(3) Check the sampler (thief) for proper operation.

(4) Open the bottom closure and set the tripping mechanism, if applicable.

(5) Lower the sampler to the required location. See [Table 5](#).

(6) At the required location, close the bottom closure on the thief with a sharp jerk of the line. Alternatively, the type of core sampler may automatically close/seal when raised.

(7) Withdraw the sampler.

(8) If only a middle sample is required, pour the complete sample into the sample container. If samples are required at more than one location, measure out a specified amount of sample with the graduated cylinder and pour it in the sample container. The amount of sample measured will depend on the size of the thief and the tests to be performed but should be consistent for the samples taken at different levels.

(9) Discard the remainder of the sample from the sampler as required.

(10) Repeat sampling procedure to obtain a sample(s) at the other sample location(s) as required.

(11) Ensure that the sample is capped tightly and clearly labeled.

9.7 Bottom Sampling:

9.7.1 Bottom samples may be necessary for a variety of reasons including: determination of free water, S & W and non-merchantable material for custody transfer or tank cleaning, concerns with tank bottoms affecting the quality of the product if a tank will be stripped or emptied during a transfer, and sampling for possible microbial activity.

9.7.2 Bottom sampling essentially allows samples to be obtained anywhere from the bottom of the tank to a height of 300 mm (1 ft) in the shore tank, marine vessel compartment, or container. Bottom sampling can be performed with the open core (thief) sampler, dead bottom sampler, and the bottom water and ROB/OBQ sampler/scrapper. Since the term “bottom sample” has a variety of meanings, the exact sampling location (for example, dead bottom, and 75 mm (3 in.) from the bottom) should be recorded on both the sample tag/label and on any analytical report.

9.7.3 *Dead Bottom Sampling Procedure*—The optimal point for lowering the sampler and the level for obtaining a bottom sample are dependent on the specific purpose for the sample

TABLE 6 Tap Sampling Requirements

Tank Capacity/Liquid Level	Sampling Requirements
Tank capacity less than or equal to 1590 m ³ (10 000 bbls)	Total sample from the lower tap
Level below middle tap	Equal amounts from the middle and lower taps
Level above middle tap-level closer to middle tap	2/3 of total sample from the middle tap and 1/3 of total sample from the lower tap
Level above middle tap-level closer to upper tap	Equal amounts from the upper, middle, and lower taps
Level above upper tap	Obtain samples as listed in Table 5. If liquid level and taps available do not allow the proper number of samples to be obtained, additional manual samples are to be obtained, such as open hatch sampling from the top of the tank.
Tank capacity of greater than 1590 m ³ (10 000 bbls)	

and should be established before sampling commences. For shore tanks, the facility owner should provide information related to the tank floor configuration (for example, flat, cone down, crown up, sump), the location and type of datum plate, and the location and accessibility of the sump and water draw.

(1) Lower the clean dry dead bottom sampler slowly until it touches the bottom of the tank or vessel compartment floor.

(2) On impact with the tank floor, the sample will begin to fill from the bottom, simultaneously displacing air through the top valve. Allow time for the sampling device to fill. Extension rods can be placed on the nipple of the bottom valve allowing product to be sampled from varying bottom levels.

(3) For “open” core-type devices, gently raise the sample apparatus 5 to 10 cm and then lower it until it strikes the bottom trapping the sample in the sampling device.

(4) After obtaining the sample, remove from the tank or vessel, and transfer the contents to a suitable intermediate container.

(5) Ensure that the sample is capped tightly and clearly labeled.

9.7.4 Bottom Water and ROB/OBQ Sampler/Scraper Procedure:

(1) Lower the clean, dry sampler until it touches the bottom of the tank or vessel compartment floor.

(2) If sampling water under the product, the sampler fills with product at first, hits the bottom, and falls over. The design will allow the sampler to tip over on the tank or vessel compartment floor. Product rises to the top with free water displacing the product in the sampler. Allow time for the sampling device to fill with water.

(3) If sampling small volumes or residual material, lower the sampler until it hits the bottom and falls over. Allow time for the sampling device to fill. For semi-liquid or high-viscosity material residue, the sampler may need to be moved on the tank floor to accumulate product clinging to the sampler.

(4) After obtaining the sample, remove from the tank or vessel, and transfer the contents to a suitable intermediate container.

(5) Cap the sample tightly and clearly label.

9.7.5 Tank Tap Sampling—In the case of storage tanks, there may be one or more taps. A tap sample is considered a spot sample and it may be combined with other samples to form a composite.

9.7.6 Tank Tap Sampling Procedure:

(1) Inspect the sample container(s) and graduated cylinder (if used) for cleanliness.

(2) Determine the liquid level in the tank.

(3) Flush the sample tap and piping until they have been completely purged. (**Warning**—Open the taps with care when sampling under pressure. Make no attempt to clear a blocked connection by forcing a rod or any other tool through an opened valve.)

(4) Collect the samples as set forth in Table 6. If a delivery tube is used, ensure the end of the delivery tube is maintained below the liquid level during the withdrawal of the sample to minimize splashing and potential loss of light ends.

(5) If the sample was collected in a graduated cylinder, deposit the sample in the sample container.

(6) Disconnect the delivery tube and cooler, if used.

(7) Ensure that the sample is capped tightly and clearly labeled.

9.7.7 If the contents of a tank fail to reach the upper or middle sample connections on a tank equipped with three connections, take the samples for the tank as given in Table 6.

9.8 Tanks Other Than Upright Cylindrical:

9.8.1 Horizontal Tanks with Circular or Elliptical Cross Section—Except as noted, take samples as spot samples from the levels indicated in Table 1. By mutual agreement, a single spot sample at the location corresponding to 50 % of the contained volume may be considered sufficient. Alternatively, it may be acceptable to use one of the other methods.

9.8.2 Tanks with Other Geometrical Shapes—Take spot samples from spherical, spheroid, and irregular shape tanks. Determine the actual levels at which the samples are to be taken to allow for the volume distribution over the height of the tank.

Sampling Using Closed and Restricted Equipment

9.9 General—While recognizing open sampling as the preferred method to obtain the most representative samples, safety and environmental regulations may prohibit the opening of the tank or vessel compartment hatches allowing the release of inert gas or hydrocarbons vapors. The requirement to use restricted or closed equipment is typically based on the availability of an inert gas system, safety and environmental regulations, the hazardous nature of the product, and vessel owner or facility policy. As a result, sampling shall be performed through vapor control valves (VCVs) using either “closed” system sampling equipment with no vapor release, or “restricted” system sampling equipment with limited vapor release. Personnel sampling on board a marine vessel should be accompanied by a designated ship’s representative.



FIG. 26 Examples of Typical Vapor Control Valves

9.10 *Vapor Control Valves (VCVs)*—The installation of VCVs should be in accordance with the requirements of the ship's classification society and the appropriate regulatory authorities. The type and size of the VCVs should be determined beforehand so that proper equipment and fittings can be available. The type and size of the VCV available for sampling can vary depending on the vessel's trade and products handled and can often have a significant impact on the ability to obtain timely samples. A 100-mm (4 in.) VCV is the recommended standard size. This standard size will allow access for various types of samplers and related fittings. Smaller VCVs can severely limit the type of sampling equipment to be used, increase the amount of time to obtain samples, and possibly the quality of the samples. See Fig. 26.

9.11 *Restricted Sampling Equipment*—Portable sampling units (PSUs) designed to obtain samples under restricted conditions operate via the VCVs fitted to the vessel normally using a simple tape system. See Fig. 3 and Fig. 4. Samplers (sample containers) of various types can be placed inside the extension tube, lowered to the desired level in the tank, and then retrieved. The VCV can then be closed and the sampler removed with a limited loss of vapor.

9.12 *Closed Sampling Equipment*—Closed system equipment is designed to be completely gas tight during sampling to prevent release of vapor to the atmosphere. See Fig. 27. The tape housing is hermetically sealed and the design should allow sample transfer to intermediate (transportation) sample container with minimal vapor loss. Typically, the same VCV used for gauging is also used for sampling. Additional options include:

- (1) Use of multiple, sealed primary samplers to avoid sample transfer;
- (2) Vapors held up within the housing to be displaced back to the tank or to an absorbent canister; or
- (3) Purge the system with inert gas.

Sampler Types for Closed and Restricted Sampling

9.13 *General*—Closed and restricted sampling equipment can be used to perform several different types of sampling operations similar to those that are performed using open manual sampling, for example, running, all-levels, spot, bottom, and dead bottom samples. Samplers (sample containers) of various types can be placed inside the extension tube, lowered to the desired level in the tank, and then retrieved. This is achieved by using different designs of samplers with the PSU.

9.14 *Running and All-Levels Samplers*—Running samplers are equipped with a fixed or adjustable restricted flow opening at the top of the sampler designed to limit the flow into the sampler thereby allowing the sampler to be lowered to the tank bottom and raised to the surface without filling completely. Obtain the sample continuously, at a steady rate, as the sampler moves down through the product column and back up to the surface. All-levels samplers only collect the sample in one direction. For both running and all-levels samples, the sample container shall be between 70 and 85 % full when sampling is completed. If the sample container is full, it cannot be determined at which point the sampler completely filled in the tank or compartment, and the sample cannot be considered a representative running sample. Many closed and restricted sampling systems do not permit the visual verification of the primary sample container to confirm that running or all-level samples have been taken properly, containing between 70 and 85 %. See Fig. 4 and Fig. 28.

9.15 *Spot Sampler*—Use spot samplers to take samples at designated tank levels such as top, upper, middle, lower, or bottom. These samplers shall be manually opened to fill when

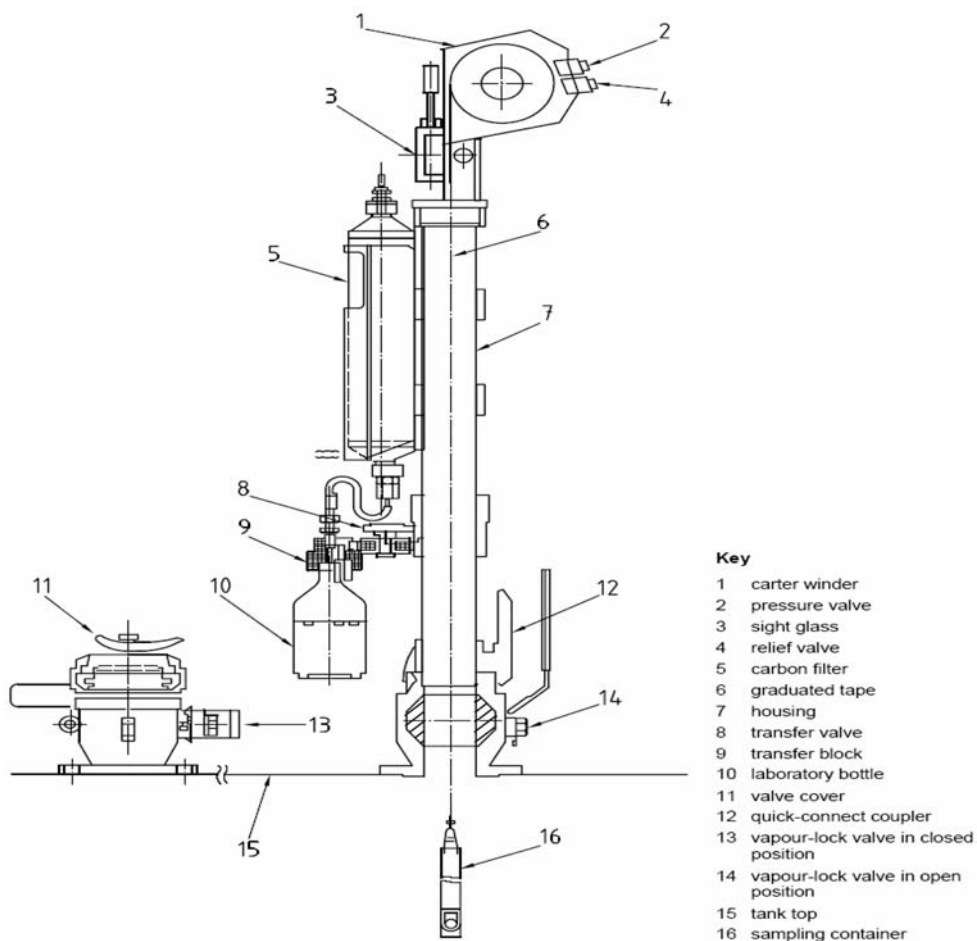


FIG. 27 Example of a Lock Valve and Closed System Sampler



FIG. 28 Example of Closed System Restricted Flow Sampler

they have been lowered to the required level and are primarily used to determine if a tank or vessel compartment is stratified.

9.16 Dead Bottom Sampler—Use the dead bottom sampler to collect a sample directly from the tank or compartment floor, to assess free water and sediment. This sampler, which may be equipped with extension rods for varying heights, is tripped open when it reaches the tank bottom and fills from the bottom.

9.17 Zone Sampler—Most zone samplers, also known as core samplers, operate on the basis of an open-ended tube with a free-floating ball valve at the bottom. When the sampler is introduced into the tank, liquid flows through the tube while it descends through the liquid. When the sampler is stopped, the ball valve closes and the sampler retains the column of product equivalent to the height of the sampler, from that level in the tank or compartment. The quality of the sample is largely dependent on the integrity of ball floating during descent and the seal of the ball valve as the sampler is retrieved, both of which can be affected by the product being sampled.

Closed and Restricted Sample Integrity

9.18 Closed and Restricted Sampling Equipment Cleanliness—To avoid contamination, properly clean samplers, fittings, and related equipment before use and between grades. This may or may not require partial disassembly of the unit. The traditional method of rinsing the primary sampling container with the product to be sampled may not be permitted under certain closed or restricted sampling conditions. However, if this method is used, care should be given to the handling, containment, and safe disposal of such rinse liquids. Fixed sampling equipment, standpipes, Vapor Control Valves (VCVs), and other fittings may contain residue from prior products, grease, rust, or scale, and may have been used for introducing additives to the tank either for the current or previous cargo, all of which has the potential to contaminate samples.

9.19 Sample Transfer—When performing restricted or closed sampling, it is normally not possible to retain the sample in the primary sampling container. The sample is typically required to be transferred from the sampling device into an intermediate sample container. This transfer process may affect the integrity of the samples obtained in the following ways:

- (1) Light ends may be lost from the sample which may affect test results, including but not limited to, vapor pressure (RVP, TVP), flash point, density, assay, distillation, and hydrogen sulfide (H_2S);
- (2) Clingage may take place in the primary sampling container likely resulting in a non-representative transfer to the intermediate sample container; and
- (3) Potential contamination from the intermediate container and inclement weather.

9.19.1 Stratification and Nature of Product—Sampling stratified and high-pour or high viscosity products can be further complicated using closed or restricted sampling equipment. For example, on high-pour point or high-viscosity products the weight of the sampler may be insufficient to penetrate readily through the liquid or may even become “stuck” in the extension tube housing, particularly while

experiencing colder ambient temperatures. In these instances, the operator may think that the sampler has reached the desired depth when in fact the tape has merely stopped running with the sampler suspended at some undetermined level. Under these circumstances, and considering the limitations of closed and restricted samplers particularly with non-homogeneous product, alternate samples should be agreed upon between interested parties. For example, additional or alternate shore tank samples or spot line samples obtained during the transfer should be considered. Under these circumstances, all relevant samples and analytical results should be considered and agreed upon by all interested parties.

Manual Pipeline Sampling

9.20 Manual pipeline sampling is applicable to liquids in pipelines, filling lines, and transfer lines. All manual pipeline samples are considered spot samples, only representative of the product in the line at that particular point in the pipeline batch or parcel. The reasons manual pipeline samples are taken can vary significantly. One manual pipeline sample might be needed at the beginning of a product pipeline or marine vessel transfer to assure a clear and bright product of the right kind. Another sample might assist with the monitoring and measuring of a pipeline change of product (COP) between pipeline batches. Yet another might assist quality control and measurement with online instrumentation calibration. These instruments include density, flash point, vapor pressure (RVP), haze, dye, octane and sulfur analyzers as well as product interface detectors.

9.21 For custody transfer, continuous automatic sampling is generally preferred. Refer to Practice **D4177** (API MPMS Chapter 8.2). In the event of automatic sampler failure, or if samples cannot be obtained from a marine vessel or shore tanks, manual spot line samples may be necessary. As agreed by interested parties, take manual samples as necessary during the transfer to obtain samples as representative as possible. The degree of product homogeneity, total transfer time, and testing requirements are just some factors to be considered when establishing the number, size and frequency of samples to be taken.

9.22 Manual Pipeline Sampling Procedure—The contents of flowing pipelines are often under considerable pressure and, as such, have some inherent additional hazards associated. A pressure gauge near the sample point is recommended.

(1) Adjust the valve or plug cock from the sampling probe so that a steady stream is drawn from the probe.

(2) After thoroughly flushing the sample probe and line, divert the sample stream to the sample container continuously or intermittently to provide a quantity of sample that will be of sufficient size for analysis. For volatility testing, samples should be obtained by filling from near the bottom of the container to minimize any risk of the evaporative loss of light ends, and a sample cooler may be used if appropriate. Refer to Practice **D5842** (API MPMS Chapter 8.4).

(3) If samples are required over a complete batch for custody transfer, the size of the samples and the intervals between each sample should be based on flow rate, and on testing and sample retention requirements, as agreed by all

TABLE 7 Minimum Number of Packages to be Selected for Sampling

Packages in Lot	Packages to be Sampled	Packages in Lot	Packages to be Sampled
1 to 3	All	1332 to 1728	12
4 to 64	4	1729 to 2197	13
65 to 125	5	2198 to 2744	14
126 to 216	6	2745 to 3375	15
217 to 343	7	3376 to 4096	16
344 to 512	8	4097 to 4913	17
513 to 729	9	4914 to 5832	18
730 to 1000	10	5833 to 6859	19
1001 to 1331	11	6860 and greater	20

interested parties. The time, and the amount of product transferred, should be recorded to correspond with each sample taken.

(4) Upon completion, the samples may be composited in one container or maintained separately and tested as agreed by all interested parties. Individual test results may be arithmetically averaged adjusting for variations in flow rate during the agreed upon time period.

(5) Tightly cap and clearly label all samples.

9.23 Pipeline Sampling of High Vapor Pressure Liquids—High pressure fixed volume cylinders and high pressure FPCs shall be filled in accordance with the procedures detailed in **9.30**.

9.24 Rail Tank Cars—For tank cars, if open sampling is acceptable, use the procedures described for sampling horizontal cylindrical tanks. If restricted or closed system sampling is required, use one of the procedures described for sampling tanks fitted with vapor control valves.

9.25 Road Vehicle Tanks—For road vehicle tanks, if open sampling is acceptable, use the procedures described for sampling horizontal cylindrical. If restricted or closed system sampling is required, use one of the procedures described for sampling tanks fitted with vapor control valves. If the product is known to be homogenous, another common practice is to sample from a bleeder valve on the truck.

9.26 Dispenser (Nozzle) Sampling—This procedure is applicable for sampling light fuels from retail-type dispensers. Fit a nozzle extension to allow fuel to be dispensed to the bottom of the sample device without splashing. See **Fig. 23**. When the nozzle is fitted with a vapor recovery system, a spacer will be needed to hold back the nozzle sleeve. Fill the sample device slowly, through the nozzle extension, until between 70 and 85 % full. Remove the nozzle and extension and close or cap the container immediately. If the sample is to be analyzed for vapor pressure, refer to Practice **D5842** (API MPMS Chapter 8.4).

9.27 Dipper/Ladle Sampling—Dipper sampling is applicable for sampling liquids and semi-liquids where a free or open discharge stream exists, as in small filling and transfer pipelines used for filling barrels, packages, and cans. The dipper device should have a capacity suitable for the amount of sample to be collected and shall be protected from dust and dirt when not being used.

9.27.1 Dipper/Ladle Sampling Procedure:

(1) Ensure the dipper is clean and free of dust and dirt.

(2) Insert the dipper in the free-flowing stream so that a sample is collected from the full cross section of the stream.

(3) Take samples at time intervals chosen so that a complete sample proportional to the pumped quantity is collected.

(4) Transfer samples into the intermediate container as soon as they are collected.

(5) Keep the container closed except when transferring samples into it. As soon as portions of the sample have been collected, close the container and clearly label it.

(6) The gross amount collected should be approximately 0.1 %, but not more than 150 L of the total quantity being sampled.

Sampling Package Lots (Cans, Drums, Barrels, or Boxes)

9.28 Take samples from a sufficient number of the individual packages to prepare a composite sample that is intended to represent the entire lot or shipment. Alternatively, samples may be tested separately. Select at random the individual packages to be sampled. The number of random packages will depend upon several practical considerations, such as:

(1) Tightness of the product specifications;

(2) Sources and type of the material and whether or not more than one production batch may be represented in the load; and

(3) Previous experience with similar shipments, particularly with respect to the uniformity of quality from package to package.

9.28.1 In most cases, the number specified in **Table 7** will be satisfactory. If agreed between interested parties, a more statistically rigorous determination may be used.

9.28.2 Sampling Barrels, Drums, and Cans—Obtain samples from the number of containers per shipment as mutually agreed. In the case of expensive solvents, which are typically purchased in small quantities, it is recommended that each container be sampled. Withdraw a portion from each container to be sampled using the tube sampling procedure or bottle sampling procedure possibly using smaller bottles. Prepare a composite sample large enough to accommodate testing, retesting, splitting, and sample retention requirements. The composite should contain portions from each container sampled of the same batch and container size.

9.28.3 Tube Sampling:

(1) The tube sampling procedure is applicable for sampling liquids of 13.8 kPa (2 psig) vapor pressure (RVP) or less and semi-liquids in drums, barrels, and cans.

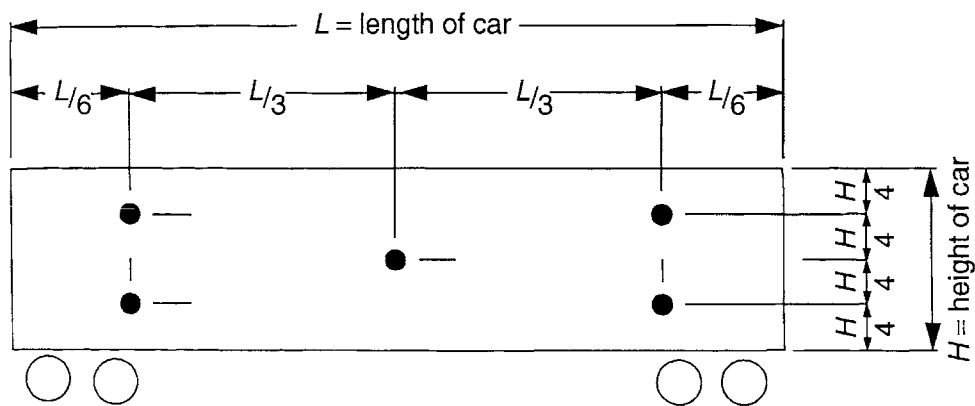


FIG. 29 Location of Sample Points at Different Levels for Rail Cars

(2) Use a glass or metal tube, designed so that it will reach to within about 3 mm ($\frac{1}{8}$ in.) of the bottom of the container. Capacity of the tube can vary from 500 mL (1 Pt) to 1 L (1 qt). A metal tube suitable for sampling 189 L (50 gal) drums is shown in Fig. 21. Two rings soldered to opposite sides of the tube at the upper end are convenient for holding it by slipping two fingers through the rings, thus leaving the thumb free to close the opening. Use clean, dry cans or glass bottles for sample containers.

(3) Typically, the sample is obtained from the top while stood upright. If the drum has a side bung, place the drum or barrel on its side with the bung up. If detection of water, rust, or other insoluble contaminants is desired, let the barrel or drum remain in this position long enough to permit the contaminants to settle. Remove the bung and place it beside the bung hole with the product side up. Close the upper end of the clean, dry sampling tube with the thumb and lower the tube into the product to a depth of about 30 cm (12 in.). Remove the thumb, allowing product to flow into the tube. Again, close the upper end with the thumb and withdraw the tube. Rinse the tube with the product by holding it nearly horizontal and turning it so that the product comes in contact with the part of the inside surface that will be immersed when the sample is taken. Avoid handling any part of the tube that will be immersed in the product during the sampling operation. Allow the tube to drain. Insert the tube into the product again, holding the thumb against the upper end. If an all-levels sample is desired, insert the tube with the upper end open at a rate that permits the liquid level in the tube to remain at the same level as the liquid in the drum. Place the thumb on the top of the tube, withdraw the tube quickly, and transfer the contents to the sample container. Do not allow the hands to come in contact with any part of the sample. Close the sample container; replace and tighten the bung in the drum or barrel. Clearly label the sample container and deliver it to the laboratory.

(4) Obtain samples from cans of 18.9 L (5 gal) capacity or larger in the same manner as for drums and barrels using a tube of proportionately smaller dimensions. For cans of less than an 18.9 L (5 gal) capacity, use the entire contents as the sample, selecting cans at random as indicated in Table 7 or in accordance with the agreement between the purchaser and the seller.

Solid and Semi-Solid Sampling

9.29 Boring Sampling—The boring sample procedure is applicable for sampling waxes and soft solids in barrels, cases, bags, and cakes when they cannot be melted and sampled as liquids. Layering of product may require special sampling as agreed upon by interested parties. The auger should be of sufficient length to pass through the material to be sampled. Refer to Section 6 for a description of the auger.

9.29.1 Boring Sampling Procedure—Remove the heads or covers of barrels or cases. Open bags and wrappings of cakes. Remove any dirt, sticks, string, or other foreign substances from the surface of the material. Bore three test holes through the body of the material, one at the center and the other two halfway between the center and the edge of the package on the right and left sides. If any foreign matter is removed from the interior of the material during the boring operation, include it as part of the borings. Put the three sets of borings in individual sample containers and clearly label.

9.29.2 Grab Sampling:

(1) The grab sampling procedure is applicable for sampling all lumpy or granulated solids in bins, bunkers, freight cars, barrels, bags, boxes, and conveyors. It is particularly applicable for the collection of green petroleum coke samples from railroad cars and the preparation of such samples for laboratory analysis. Refer to Practice D346 when other methods of shipping or handling are used. Petroleum coke may be sampled while being loaded into railroad cars from piles or after being loaded into railroad cars from coking drums.

(2) Use a polyethylene pail of approximately 9.5-L (2.5-gal) capacity as the sample container. Use a stainless steel or aluminum No. 2 size scoop to fill the container.

(3) **Grab Sampling Procedure**—Lumpy and granulated solids are usually heterogeneous and difficult to sample accurately. It is preferable to take samples during the unloading of cars or during transit; obtain a number of portions at frequent and regular intervals and combine them.

(4) When sampling from railroad cars, use one of these procedures (5 or 6) below:

(5) **Being loaded from a pile**—Take a full scoop of sample at each of the five sampling points shown in Fig. 29 and deposit

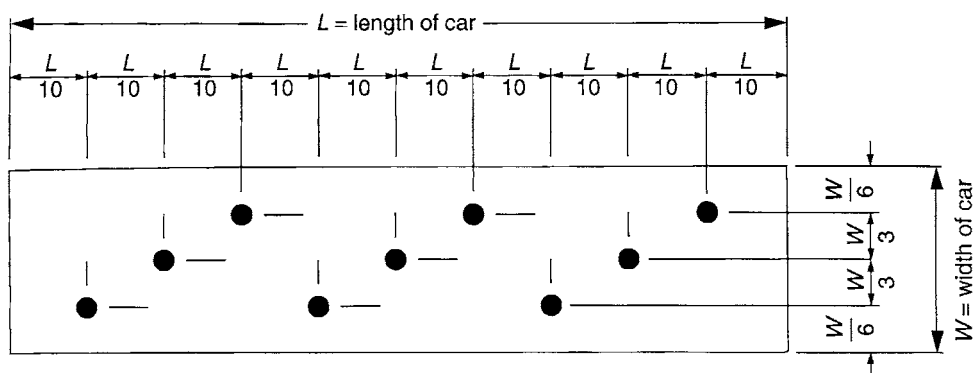


FIG. 30 Location of Sample Points from Exposed Surface for Rail Cars

in a polyethylene pail. Each sample should be equidistant from the sides of the railroad car. Cover the sample and deliver to the laboratory.

(6) *After direct loading from coking drums*—At any five of the sample points shown in Fig. 30, take a full scoop of coke from about 30 cm (12 in.) below the surface and deposit it in a polyethylene pail. Cover the sample, clearly label, and deliver to the laboratory.

(7) When sampling from conveyors, take one scoop for each 7 to 9 tonnes (7 to 9 long tons) of coke transported. These samples may be handled separately or composited after all samples representing the lot have been taken.

(8) When sampling from bags, barrels, or boxes, obtain portions from a number of packages selected at random as shown in Table 7, or in accordance with the agreement between interested parties. In most cases, the number specified in Table 7 will be satisfactory. If agreed between interested parties, a more statistically rigorous determination may be used.

(9) Carefully mix the grab sample and reduce it in size to a convenient laboratory sample by the quartering procedure described in Practice D346. Perform the quartering operation on a hard, clean surface free from cracks and protected from rain, snow, wind, and sun. Avoid contamination with cinders, sand, chips from the floor, or any other material. Protect the sample from loss or gain of moisture or dust. Mix and spread the sample in a circular layer and divide it into quadrants. Combine two opposite quadrants to form a reduced sample. If this sample is still too large for laboratory purposes, repeat the quartering operation. In this manner, the sample will finally be reduced to what is intended to be a representative, suitable size for laboratory purposes. Clearly label and deliver the sample to the laboratory in a suitable container.

9.30 Spot Sampling of Liquids Using Cylinders:

9.30.1 *Sampling Practices and Procedures*—The following are provided as practices and procedures for obtaining liquid spot samples from pipelines, shore storage tanks, marine vessel tanks, rail cars and trucks using fixed volume and FPCs. Refer to Fig. 11 for a typical high pressure sampling system. It is recognized that other practices and procedures or use of alternate equipment, piping, valves, and tubing arrangements, may produce acceptable samples. Regardless of the process and procedures employed, the following shall be observed:

(1) The contents of all lines and components, from the sample probe to the inlet valve of the sample cylinder, are purged and representative of the product being sampled, prior to filling the sample cylinder;

(2) The sample shall remain in a liquid state at all times;

(3) All components that come in contact with the sample shall be compatible with the product being sampled;

(4) Strictly observe safe fill and safety precautions;

(5) Extreme care, good judgment, and sampling experience are necessary to obtain representative samples and to maintain sample integrity for the tests being performed; and

(6) Because of the potential hazards associated with high pressure cylinder sampling, sampling should be performed by, or under the supervision of, persons familiar with the necessary safety precautions. Refer to 6.9. Also refer to Practice D1265 and GPA S 2174 for obtaining samples in fixed volume cylinders.

9.31 *Sampling Related Purging and Venting*—Vapors vented during purging and sampling shall be controlled to ensure compliance with applicable safety and environmental regulations. The cylinder may be partially filled and then emptied prior to collection of the sample as an alternative to venting hydrocarbon to flush lines.

9.32 *Sample Homogeneity*—When it has been established that the product being sampled is homogeneous, either as determined by the nature of the product or through mixing or circulation, a liquid sample may be taken from any part of the system.

9.32.1 *Slip Tubes and Fixed Sample Lines*—Marine vessel cargo tanks and shore tanks, for example spheres and bullets, may be equipped with one or more slip tubes, or one or more sample lines having a fixed depth into the tank. The slip tube or sample line shall be equipped with a probe outlet valve and connections, impervious to the product being sampled, which permit the safe flushing of the probe and line contents, and the accumulation of sample in the cylinder. By design, the depth of the slip tube can be adjusted to ensure a liquid sample is obtained. Samples taken through fixed sample lines will provide either vapor or liquid at that level. In addition, the depth of the fixed sample lines should be recorded since it may be significant if the product being sampled is not homogenous.

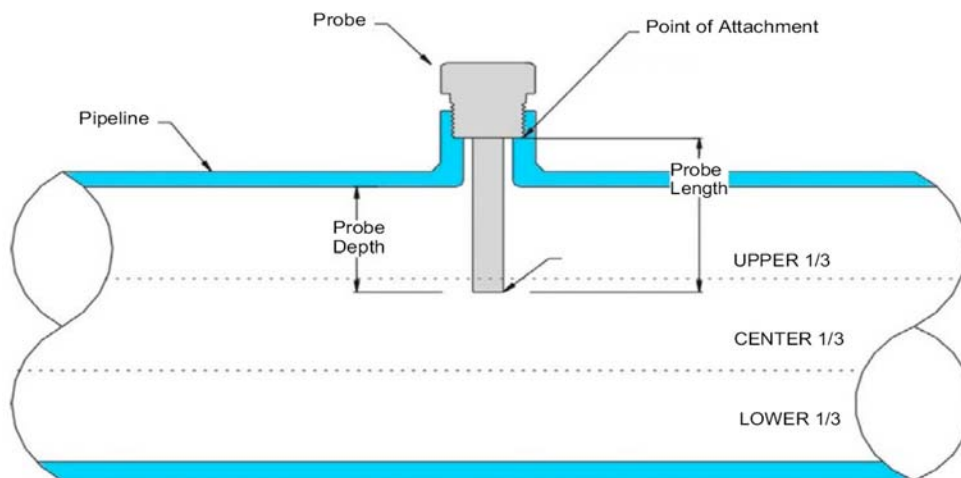


FIG. 31 Example of Probe in High-Pressure Sampling System Pipeline

If the product is not homogenous, fixed sample lines should not be used for obtaining representative samples. An alternate sampling method, as agreed upon by interested parties, should be established, such as additional spot samples, a flow-proportional composite sampler, or an alternate sample point.

9.33 Line Sampling—Thorough purging of sample lines, pumps, and connections is necessary to avoid contamination of the sample. Sample loops should preferably be installed around pumps, valves, or other sources of pressure drop to minimize atmospheric emissions from purging of sample lines. 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 both the lines or the cylinder dead volume, or both, if any, prior to sample collection. See Fig. 31.

9.33.1 Sample Probe—The use of a sample probe in a flowing line is recommended. The sample probe should be located on the side or top of a line, extending into the center third of the flowing stream. Sample points should not be installed on the bottom of a line, unless provisions are made to flush any accumulated debris from the sample point immediately prior to sampling.

9.33.2 Circulating for Line Sampling—Due to the nature of the product and sample points available on shore or marine vessel tanks, it might be necessary or desirable to sample the product as it is being circulated through the pipeline or vessel cargo line. In these instances, it is recommended that the product be circulated for a minimum of 15 min before obtaining samples. The sample point shall be located in the flowing stream, or sample loop, and not located in a dead leg, or otherwise out of the main flow of the product.

9.34 Sample Storage and Disposal—Samples should be stored in such a manner as to enable quick retrieval and to prevent theft, tampering, weathering, or degradation from light, heat, cold, and any other conditions. In addition to the cylinder valves, caps or threaded plugs may be considered as an additional assurance to prevent loss of samples. Store and dispose samples in such a way to ensure compliance with company policy and regulatory requirements.

9.35 Other Liquid Sample Cylinders—Refer to the listed practices, methods, or manufacturer's instructions, or a combination thereof, for the following tests:

- (1) Copper Strip Corrosion Cylinder—Test Method **D1838**;
- (2) Vapor Pressure Cylinder—Test Method **D1267**;
- (3) Pressure Hydrometer—Test Method **D1657**;
- (4) Pressure Hydrometer Test Method for Density or Relative Density—API MPMS Chapter 9.2;
- (5) Pressure Pycnometer—API MPMS 14.6.

Sampling into Fixed Volume Sample Cylinders

9.36 General—The liquid is sampled at the observed product pressure, and maintained at or above this observed pressure during transportation and subsequent sub-sampling. It is essential to create an ullage volume, typically of approximately 20 %, within the fixed volume sample cylinder immediately after taking the sample to prevent any unsafe increase in sample pressure due to thermal expansion.

9.37 Selection and Leak Testing of the Fixed Volume Sample Cylinder—The working (sample) volume of a fixed volume sample cylinder is typically 20 % less than the nominal volume to allow for the necessary ullage space. Select a fixed volume sample cylinder with the required capacity and a rated working pressure that exceeds the pipeline pressure. Verify that the pressure test certification for the fixed volume sample cylinder, and if so equipped verify the bursting (rupture) disk, is current. Ensure the fixed volume sample cylinder is clean and dry. Pressurize the fixed volume sample cylinder with inert gas to at least 100 kPa (14.5 psig) more than the anticipated pipeline pressure, and test for leaks. If a valve or fitting is found to leak, replace it and re-test, or use another fixed volume sample cylinder.

9.37.1 Purging the Sample Line:

- (1) Confirm that all valves are initially closed.
- (2) If the fixed volume sample cylinder has an internal outage tube fitted, determine which fixed volume sample cylinder valve is connected to the tube and connect this valve

to the sample point control valve. If the fixed volume sample cylinder has a second valve, connect that valve to a closed drain or other safe disposal route. (**Warning**—Ensure the fixed volume sample cylinder is properly grounded before any sampling commences.)

(3) Purge the sample line by displacing at least 150 % of the line's volume (from the sample probe in the pipeline to the sample point) to the vent immediately before the sample point valve.

(4) Close the vent valve.

9.37.2 *Purging a One-Valve Fixed Volume Sample Cylinder:*

(1) Open the one valve fixed volume sample cylinder inlet valve to partly fill the fixed volume sample cylinder.

(2) Close the sample point control valve, and open the line vent valve to purge the fixed volume sample cylinder.

(3) Close the vent valve and repeat the partial filling and venting process at least twice more to purge the fixed volume sample cylinder.

9.37.3 *Purging a Two-Valve Fixed Volume Sample Cylinder:*

(1) For a two-valve fixed volume sample cylinder, open the fixed volume sample cylinder inlet valve to fill the fixed volume sample cylinder partly.

(2) Slowly open the fixed volume sample cylinder outlet valve to its vent.

(3) Close the sample point control valve, and allow part of the fixed volume sample cylinder contents to escape to vent through the fixed volume sample cylinder outlet valve.

(4) Close the fixed volume sample cylinder vent valve and open the sample line vent valve to allow further venting.

(5) Close the sample line vent valve and repeat the partial filling and venting process at least twice more to purge the fixed volume sample cylinder.

9.37.4 *Filling the Fixed Volume Sample Cylinder:*

(1) On completion of the purging operation, open the sample point control valve to fill the fixed volume sample cylinder;

(2) Close the fixed volume sample cylinder inlet valve;

(3) Close the sample point control valve;

(4) Open the sample line vent valve to safely depressurize the sample line;

(5) Disconnect the fixed volume sample cylinder.

9.37.5 *Providing a Safe Ullage Space within a Fixed Volume Sample Cylinder Using an Internal Outage Tube:*

(1) Position the fixed volume sample cylinder upright with the sample inlet valve (and internal outage tube) at the top. (**Warning**—Electrostatic safety considerations may mean that it is advisable to fit the fixed volume sample cylinder with an earth (grounding) connection before proceeding to the next partial venting step.)

(2) Slowly open the fixed volume sample cylinder inlet valve slightly until liquid is observed to escape. Allow the excess liquid to escape, but, as soon as it is observed that the escaping liquid material changes to vapor quickly, close the valve. If no liquid escapes initially then the fixed volume sample cylinder was not filled sufficiently, and the sample should be discarded and the sampling procedure repeated.

9.37.6 *Providing a Safe Ullage Space within a Fixed Volume Sample Cylinder Using a Weighing Procedure:*

(1) Weigh the filled fixed volume sample cylinder and deduct the tare weight to determine the total weight of sample that has been taken. Calculate the weight of sample that represents a 20 % ullage, and vent this amount by slowly opening the fixed volume sample cylinder inlet valve slightly. (**Warning**—Electrostatic safety considerations may mean that it is advisable to fit the fixed volume sample cylinder with an earth (grounding) connection before conducting the venting operation.)

(2) Close the fixed volume sample cylinder inlet valve and re-weigh the fixed volume sample cylinder to verify that a safe ullage space has been created. Repeat the partial venting operation if the gross weight still exceeds the tare weight plus 80 % of the original sample weight. (**Warning**—If the fixed volume sample cylinder weighing cannot be performed at the sampling location, it is important to ensure that a small quantity of the liquid phase sample is vented immediately to prevent excessive pressure build-up as a result of sample expansion due to any subsequent increase in temperature. The full weighing and ullaging procedure should then take place as soon as possible after transportation to a suitable location where the facilities are available.)

9.37.7 *Fixed Volume Sample Cylinder Sample Handling:*

(1) Upon completion of sampling, the fixed volume sample cylinder should be checked for leaks, for example with a proprietary leak detecting fluid, soapy water, or by immersing in water. If any leaks are detected, discard the sample and repair or replace the fixed volume sample cylinder then repeat the sampling procedure.

(2) Clearly label or tag the fixed volume sample cylinder and prepare it for transportation by packing in a suitable container, as required by the appropriate transportation regulations.

(3) Transport the fixed volume sample cylinder to the laboratory/test location without delay. Where intermediate storage is necessary, the sample should be protected from temperature extremes.

Sampling into Single Floating Piston Cylinders (FPCs)

9.38 *General*—The liquid is sampled at the observed product pressure, and during transportation and subsequent subsampling the liquid is maintained at a pressure close to the observed pressure (or higher) at which the sample was obtained. The sample is accumulated against one side of the floating piston by carefully reducing the pressure of an inert gas buffer on the opposite side of the piston. If the liquid is refrigerated, the effect of low temperatures on the cylinder piston seals, and extraordinary product volumetric expansion within the FPC should be considered. Refer to 6.9.4. See Fig. 8, Fig. 9, and Fig. 10.

9.39 *Selection and Leak Testing of the Single Floating Piston Cylinder (FPC)*—Select an FPC with the required capacity and a rated working pressure which exceeds the pipeline pressure and meets all applicable transportation requirements, rules, and regulations. Refer to 6.9. Confirm that all components, including the piston seal elastomer are compatible with the liquid, and capable of operating effectively at the anticipated temperature. Ensure the FPC is clean and dry.

Refer to 6.9.6. Pressurize both sides of the single floating-piston variable volume sample cylinder with inert gas to at least 100 kPa (14.5 psig) more than the anticipated pressure, and test for leaks. Test the FPC piston seals for leakage by pressurizing each side in turn to the same pressure while the other side is open to atmosphere. If a valve, fitting or seal is found to leak, replace it and re-test, or use another FPC.

9.40 Pre-Charging the Single Floating Piston Cylinder (FPC):

- (1) Open the sample inlet valve.
- (2) Connect the inert gas pre-charge valve to a supply of the appropriate inert gas. The preferred pre-charge gas is one that is not normally present in the sample (such as, helium, nitrogen, or argon) or one that will not be detected should it leak into the sample. Refer to Practice D3700 for guidance on inert gas usage, and refer to the relevant test methods.
- (3) Open the inert gas pre-charge valve slowly, and pressurize to at least 100 kPa (14.5 psig) above the pipeline pressure, or that recommended by the single floating-piston variable volume sample cylinder manufacturer, so that the piston is fully displaced against the sample inlet end plate.
- (4) Close all valves.
- (5) Depressurize the line safely.
- (6) Disconnect the inert gas supply.
- (7) Transport the pre-charged FPC to the sample point within its transportation case.

9.41 Purging the Sample Lines and Single Floating Piston Cylinder (FPC):

- (1) Connect the pre-charged FPC sample inlet valve to the sample point, either directly or using minimal tubing.
- (2) With the FPC inlet valve closed, open the sample point control valve.
- (3) Open the FPC purge (exhaust) valve to flush out sample line upstream of the FPC inlet valve. The flow should be fast enough to flush any debris from the line, but short enough duration to minimize venting. If the FPC does not have a purge (exhaust) valve, the line from the sample line control valve to the sample point should be similarly flushed out before connecting the FPC to be filled. In either case, the purge/vent line should be piped to a flare line or elsewhere in the process to minimize emissions to the atmosphere.
- (4) Close the FPC purge valve.

9.42 Filling the Single Floating Piston Cylinder (FPC):

- (1) Verify that the pressure in the pre-charge chamber of the FPC is higher than the pressure at the sample point.
- (2) Open the FPC inlet valve. Provided the pre-charge chamber is at a higher pressure than the line, the piston should not move before the inert gas is vented. If the piston does move before the gas is vented, reject, and use another FPC, increasing the pressure as appropriate. (**Warning**—Do not attempt to fill a single FPC when there is little or no pressure on the pre-charge side of the cylinder, because the piston and attached indicator rod can move with extreme speed and force.)
- (3) Slowly vent the inert gas from the balance chamber through the FPC inert gas pre-charge valve. When the pressure at the balance side approaches the pressure at the sample point, sample will begin to flow into the FPC, pushing the floating piston back. (**Warning**—Vent the inert gas slowly when

approaching the sample pressure. Rapid venting can result in excessive vaporization, or cause the piston to move abruptly, or both.)

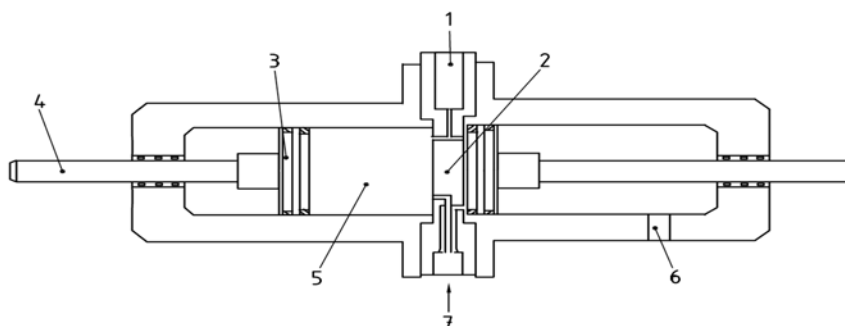
(4) Monitor the piston position indicator, stopping at the desired fill level (or fill density), not to exceed 80 % at 15°C (59°F). The pressure difference between the sample and the inert pre-charge sides of the piston should not exceed 100 kPa (14.5 psig) at any time during sampling to ensure that the sample does not drop below its bubble point and allow gas breakout to occur. (**Warning**—When filling a single FPC below about -5°C, the maximum fill density shall be reduced below 80 % to account for the additional thermal expansion (based on the product being sampled), and satisfy regulatory requirements for increased outage or reduced fill density.)

- (5) Close the FPC inert gas pre-charge valve.
- (6) Close the FPC inlet valve.
- (7) Close the sample line control valve.
- (8) Before disconnecting the FPC, depressurize the sample line preferably to a flare line or elsewhere in the process to minimize emissions to the atmosphere.
- (9) Disconnect the FPC. At the end of sampling, the sample in the FPC should be at the same pressure as the sample point, which may not be appropriate for subsequent use in the laboratory. It is good laboratory practice to adjust the pressure in the FPC to a suitable pressure prior to analysis. Refer to Practice D3700 for guidance, and refer to the relevant test methods.
- (10) Clearly label or tag the FPC and prepare it for transportation by packing in a suitable container, as required by the relevant transportation regulations.
- (11) Transport to the laboratory/test location without delay. Where interim storage is necessary, the sample should be protected from extremes of temperature.

9.43 Sampling into Double-Piston (Variable Volume) Cylinders:

9.43.1 *General*—The liquid is sampled at the observed product pressure, and during transportation and subsequent sub-sampling the liquid is maintained at a pressure close to the observed pressure (or higher) at which the sample was obtained. Sample is accumulated against only one of the floating pistons by carefully reducing the pressure of the inert gas buffer on the opposite side of that piston. The inert gas pressure on the second piston is kept significantly greater than the pipeline pressure throughout the sample accumulation stage to ensure that the DPC is not overfilled. Refer to Fig. 32 for an example of a DPC.

9.43.2 *Selection and Leak Testing of the Double Piston Cylinder (DPC)*—The working sample volume of a DPC is only about 50 % of the total volume. Select a DPC with the required capacity and a rated working pressure which exceeds the pipeline pressure and meets all applicable transportation requirements, rules, and regulations. Refer to 6.9.2 and 6.9.3. Confirm that all components, including the piston seal elastomer are compatible with the liquid, and capable of operating effectively at the anticipated temperature. Ensure the DPC is clean and dry. See 6.9.6.1. Leak test all components, and if a valve, fitting or seal is found to leak, replace it and re-test, or use another DPC.

**Key**

- 1 sub-sampling port
- 2 mixing baffle
- 3 piston
- 4 piston indicator rod
- 5 sample chamber
- 6 inert gas port
- 7 sample inlet

FIG. 32 Example of Double Piston (Variable Volume) Cylinder (DPC)**9.44 Pre-Charging the Double Piston Cylinder (DPC):**

- (1) Open the sample inlet valve.
- (2) Connect both inert gas pre-charge inlet valves to a supply of the appropriate inert gas. The preferred pre-charge gas is one that is not normally present in the sample (such as, helium, nitrogen, or argon) or one that will not be detected should it leak into the sample. Refer to Practice D3700 for guidance on inert gas usage, and refer to the relevant test methods.
- (3) Open the inert gas pre-charge inlet valves slowly, and pressurize to at least 500 kPa (72.5 psig) above the pipeline pressure, or that recommended by the floating-piston variable volume sample cylinder manufacturer, so that both pistons are fully displaced against the central sample inlet/baffle plate.
- (4) Close all valves.
- (5) Depressurize the line safely.
- (6) Disconnect the inert gas supply.
- (7) Transport the pre-charged DPC to the sample point within its transportation case.

9.45 Purging the Sample Lines and the Double Piston Cylinder (DPC):

- (1) Connect the pre-charged DPC inlet valve to the sample point, either directly or using minimal tubing.
- (2) Connect the sample outlet port/septum adapter valve to a closed drain or other safe disposal route.
- (3) Open the DPC outlet port valve fully and partially open the DPC inlet valve (typically one-quarter turn).
- (4) Carefully open the pipeline sample point control valve to thoroughly flush the sample line and DPC dead volume.
- (5) Connect the pre-charged DPC inlet valve to the sampling point. Connect the sample outlet port/septum adapter valve to a closed drain or other safe disposal route.
- (6) Control the rate of liquid flow through the DPC by carefully opening the inlet valve and the sample point valve further until the dead volume has been thoroughly flushed.

9.46 Filling the Double Piston Cylinder (DPC):

- (1) Close the DPC outlet port valve fully on completion of the flushing operation. Neither piston should move at this stage due to the buffer gas pressure being higher than the pipeline pressure.
- (2) Slowly open one (but not both) of the inert gas end valves to gradually reduce the inert gas pressure on one side of the DPC. The reduction in the inert gas pressure will allow sample to accumulate into the working side of the DPC, as a result of the pressure differential between the pipeline and the inert gas buffer.
- (3) The inert gas valve is to be carefully adjusted to ensure that the corresponding sample inlet flow rate is properly controlled. The pressure difference between the pipeline and the working inert gas buffer should not exceed 100 kPa (14.5 psig) at any time during the sample accumulation period.
- (4) Close the valves when the piston position indicator shows that the double DPC has been filled to approximately 90 % of the filled side of the DPC (that is, about 45 % of the nominal total capacity).
- (5) Before disconnecting the DPC, depressurize the sample line preferably to a flare line or elsewhere in the process to minimize emissions to the atmosphere.
- (6) Finally, and as necessary, connect the working inert gas inlet valve to an inert gas supply and increase the inert gas pressure to at least 100 kPa (14.5 psig) above the pipeline pressure. Refer to Practice D3700 for guidance on inert gas usage, and refer to the relevant test methods. It is not necessary to equalize the pressure between both inert gas buffers, but this may be done provided that the resulting final inert gas pressure is maintained above the pipeline pressure.
- (7) Disconnect the DPC.
- (8) Clearly label or tag the DPC and prepare it for transportation by packing in a suitable container, as required by the appropriate transportation regulations.

(9) Transport to the laboratory/test location without delay. Where intermediate storage is necessary, the sample should be protected from extremes of temperature.

9.47 Sub-Sampling and Mixing—Before sub-sampling the primary sample for analysis, it may be homogenized by forcing the total sample quantity repeatedly through fine-bore transverse holes in the central baffle plate. This is achieved by alternately reducing the pressure of one inert gas buffer relative to the other (while maintaining the sample pressure above its bubble point). Alternate methods of mixing may also be acceptable based on product, equipment and experience. The efficiency of this multi-phase sample homogenization may be verified by controlled injection/recovery tests. Consequently, these DPCs are recommended for taking time-synchronized spot samples that are used for the calibration and verification of automatic pipeline water content analyzers operating on high pressure pipelines, including unstabilized crude oil and condensate production, and being used in fiscal or allocation accounting applications.

10. Special Instructions for Specific Products

10.1 This section provides specific additional equipment, container, sampling and sample handling guidance for the referenced products. It is meant to supplement, and be used in conjunction with, the guidance provided in the other sections. In addition, reference the test methods to determine if any special sampling and sample handling is required. Refer to **Appendix X1** listing sample handling guidance for common crude oil tests.

Crude Oil

10.2 Properties—By its very nature, crude oil is typically non-homogenous, containing some percentage of sediment and water. It also typically contains volatile light ends, and finally crude oil will often exhibit high pour point and high viscosity properties, due to its asphaltenes and paraffin wax content. Each of these properties presents unique challenges with regard to obtaining representative samples from the field, and maintaining the integrity of the sample representation to the analytical apparatus.

10.2.1 Crude Oil Sediment and Water Component - Stratification—The water and sediment component of the crude oil will tend to naturally separate and stratify in shore tanks, marine vessel compartments and in flowing pipelines. The concentration of dispersed water and sediment in the oil is normally higher near the bottom of a shore tank, marine vessel compartment or pipeline. A running or all-level sample, or a composite of spot samples, for example upper, middle and lower samples, may not provide a sample representative of the concentration of the dispersed water and sediment present. Because of this stratification tendency, the representativeness of a manual sample may vary greatly based on many factors such as, but not limited to: the timing of obtaining manual samples following a transfer or tank mixing; velocity of the transfer rate, or capability of mixers; piping and inlet configurations; mixing elements in the transfer line; density of the crude oil relative to water; temperature and viscosity of the crude oil; use of additives; etc. In addition, the oil, water and

sediment interfaces are often difficult to identify and quantify, especially in the presence of emulsions. Finally, the free water, sediment and emulsion levels can vary across the bottom of the shore tank, marine vessel compartment or pipeline.

10.2.2 Volatile Light Ends—Volatile light ends may be lost in manual sampling operations, particularly if the product is transferred from the primary sample container, potentially affecting vapor pressure, distillation, density, and possibly other volatility related properties. Transferring crude oil samples from the primary sample container to an intermediate container is a common and required practice using certain closed and restricted sampling systems.

10.2.3 High Pour Point and High Viscosity Crude Oil—If not maintained at a sufficiently high temperature, wax deposition on the walls of the shore tank, the marine vessel compartment, and the pipeline may occur likely leading to unrepresentative samples. Exercise great care, particularly with regard to heating and homogenizing, to ensure the sample remains representative during all handling stages from the primary sample container to the test apparatus. Refer to Practice **D5854** (API MPMS Chapter 8.3).

Obtaining Crude Oil Samples

10.2.4 Plastic Containers—Plastic containers are generally not recommended for sampling crude oil primarily due to, but not limited to, the inability to retain volatile light-end components, and inability to heat high pour or high viscosity crude oils without deformation.

10.2.5 Shore Tank and Marine Vessel Compartment Manual Sampling:

10.2.5.1 Common Methods—The following methods are common for sampling crude oil shore tanks and marine vessel compartments:

- (1) Running or all-levels sampling,
- (2) Spot sampling—typically upper, middle, and lower (UML) or top, middle, and bottom (TMB),
- (3) Dead bottom sampling, with or without extension rods.

10.2.5.2 Crude Oil Stratification—Spot samples may be taken to assess the level of stratification of a particular property within a shore tank or marine vessel compartment. The use of composite samples based on spot samples, such as upper, middle and lower (UML), is not recommended for S & W determination as the heaviest S & W is often located below the lower sample; therefore, the use of running or all level samples is recommended.

10.2.5.3 Closed System Crude Oil Sampling Issues—Closed and restricted manual sampling provides additional challenges in obtaining representative samples of crude oil containing water and sediment, including: (1) product residue on the primary sampler; (2) incompatible fittings; (3) inert gas pressure while sampling; (4) difficult and cumbersome using closed system equipment through vapor control valves; (5) size restrictions of primary sampler; (6) inability to see product being sampled; and (7) limited visibility of the sample volume obtained. During cold weather, high pour crude oils are very difficult to sample due to the wax build-up on deck equipment. Volatile light-end components may be lost in manual closed or restricted sampling operations, particularly with equipment that requires product to be transferred from the primary sample

container. It is for many of these reasons that open tank and marine vessel compartment sampling is the preferred method to obtain the most representative manual samples.

10.2.5.4 Sampling Small Crude Oil Lease Tanks—Small crude oil lease tanks of less than 159 m³ (1000 barrels) used for custody transfer related to truck or pipeline gathering systems often require an assessment of crude oil quality be made onsite prior to beginning the custody transfer. The API MPMS Chapter 18.1 provides custody transfer guidelines for sampling, gauging, obtaining temperatures, and determining crude quality from small tanks to a tank truck.

10.2.6 Obtaining Large Sample Volumes of Crude Oil—If large-volume samples are required which may not, because of volatility or other considerations, be obtained by the bulking of smaller quantities, mix the tank contents thoroughly by the means available (for example, circulation, tank side mixer). Confirm homogeneity by tests on samples taken at different levels as agreed by interested parties. Fill the sample container using a sample inlet designed to fill from near the bottom of the container, with the sample taken from tank-side taps, or from a sample point on the tank circulation system.

10.2.7 Obtaining Spot Line Samples of High Pour Point Crude Oils from Shore Tanks or Pipelines—If the crude oil being sampled has a high pour point, it may be necessary to insulate the sample line thermally or to provide means of heating the sampling connections in order to prevent solidification.

10.2.8 Obtaining Spot Line Samples of Crude Oil for Volatility Measurement from Shore Tanks or Pipelines—If testing for volatility properties, fill the sample container using a sample inlet designed to fill from near the bottom of the container to minimize the loss of light ends. Cooling of the sample container and the use of a cooler for sample transportation may also be considered. Also refer to Practice D5842 (API MPMS Chapter 8.4).

10.2.9 Obtaining Spot Line Samples of Low Pressure Crude Oil from Shore Tanks or Pipelines—Fill fixed volume sample container after thoroughly flushing the sample probe and line. Empty low pressure variable volume sample container (for example, collapsible flexible containers, bladders or diaphragm containers) prior to use. Where appropriate, the container may be evacuated. The container shall be filled directly after thoroughly flushing the sample probe and sample line.

10.2.10 Obtaining Spot Pipeline Samples of High Vapor Pressure Crude Oil—Refer to 9.30. (**Warning**—Use of low pressure variable volume containers (for example, collapsible flexible containers, bladders or diaphragm containers), or low pressure fixed volume containers (for example, bottles or cans) for sampling high vapor pressure pipeline liquids are not recommended. If such containers are used, phase separation may occur as the pressure is reduced and light ends may be lost. Manufacturers' guidelines and limitations are to be strictly followed regarding the pressure limitations, and handling, of the variable volume container.)

10.2.11 Obtaining Spot Samples of Crude Oil Production Flowing Pipelines:

10.2.11.1 The following procedure should be used for atmospheric crude oil sampling from crude oil production and process systems for performance monitoring and quality analysis.

(1) Prior to sampling, prepare all sampling equipment in compliance with applicable safety procedures and facility specific requirements.

(2) Ground the sample can to a suitable and designated bonding point. In addition, in order to earth any static charge on their person, the individual obtaining the sample shall touch some part of the structure, at least 1 m (3 ft) from any sample point immediately before carrying out the sampling operation. (**Warning**—Plastic containers are not to be used for sampling and subsequent homogenization due to the risk of static build-up. When connecting earth leads to the sample cans, ensure metal to metal contact is not compromised by painted surfaces or coatings. To ensure metal to metal contact clean a small area of paint away from the rim of the can and manipulate the earth lead clip back and forth on connecting surface to make sure direct contact is achieved. Alternatively use a purpose designed grounding lead and static earthing clamp. Some devices are fitted with tungsten carbide teeth to cut through paint and provide visual indication that a resistance of 10 ohms or less is present and do not have the potential to accumulate sufficient energy to create a spark.)

(3) Flush the sample point and associated upstream piping into a grounded metal container until the sample line has been thoroughly purged of crude oil, and any residual water or deposition products.

(4) Fill the metal sample can such that it is between 70 and 85 % full in order to provide ullage space for subsequent homogenization, and to allow for possible crude oil thermal expansion.

(5) Cap and immediately label the sample container in preparation for transportation or delivery for testing or retention.

Handling of Crude Oil Samples

10.3 General—The handling of samples shall be based on individual and composite testing requirements, anticipated retesting, retain and sharing of samples. This practice is written with the understanding that any unique or special sample handling instructions will be provided in the relevant test method.

10.3.1 Sequence of Test Methods—When performing several tests on a sample of crude oil it is very important to ensure that the sequence of testing is evaluated to minimize altering the properties of the remaining sample to be tested or retained. If crude oil samples are to be tested for vapor pressure, H₂S, or any other test in which retention of light ends is critical, sub samples for these test methods should be taken first before any other sample handling procedures are performed, as provided in the specific test methods. For some tests, such as vapor pressure and some H₂S test methods, the primary sample container is typically used to provide the sample directly into the analytical instrument. If the test methods have conflicting requirements, separate samples may be necessary for testing

directly from each individual primary sample container, as described or required in the test method.

10.3.2 Crude Oil Sample Transportation—The sample is to be transported to the test laboratory in the original sample container, without transfer or compositing (bulking), in order to maintain the integrity of the sample. If the sample needs to be transferred, it shall be performed following the recommendations as provided in Practice **D5854** (API MPMS Chapter 8.3). Many test methods specify time constraints and limitations on how soon a sample is to be tested following the time the samples were obtained. In these instances, the samples shall be transported to the testing location in a safe and timely manner to maintain the integrity of the sample, and sample container, or stored in an appropriate cool, dark, dry location. If the sample is to be tested for volatility measurement, refer to Practice **D5842** (API MPMS Chapter 8.4) and the related test methods.

10.3.3 Heating of Crude Oil Samples—Crude oils stored at temperatures below their cloud point will show wax deposition on the walls of the sample containers. The wax coming out of the solution will be, preferentially, the wax with the high melting point. It is this type of wax that has the most pronounced influence on the crude oil pour point and, at the same time, is the most difficult to redissolve or disperse in the crude oil. In order to achieve complete solubility of the wax, heat crude oil samples to a temperature above the cloud point. Since this value is seldom known, heating the sample to a temperature of 20°C above the expected pour point will usually satisfy the cloud point requirement, although exceptions do occur. The vast majority of crude oils show a significant vapor pressure even at ambient temperatures. Crude production is usually stabilized at a vapor pressure of 50 kPa (Test Method **D323**, RVP at 37.8°C) or below. However, occasionally, high vapor pressure crudes (80 kPa) are produced and marketed. Before testing, never subject a crude oil sample to a temperature higher than 60°C or to a temperature above the bubble point (vapor pressure 100 kPa). As a rule of thumb, the vapor pressure doubles for every 20°C increase in temperature.

Crude Oil Mixing and Homogeneity

10.4 General—The proper means and effectiveness of mixing in order to achieve homogeneity depend on the physical properties (for example, viscosity, temperature, volatility, density, water and sediment) of the crude oil and the capacity, shape, and type of container in which the crude oil arrives at the laboratory. It is virtually impossible to anticipate every possibility and achieve optimum results under all circumstances. Refer to Practice **D5854** (API MPMS Chapter 8.3) and the test method for specific guidance.

10.4.1 Cans, 1 to 15 L—(1 qt to 4 gal) Store the container at a temperature 20°C (36°F) above the expected pour point preferably in a water bath kept at the appropriate temperature. Alternatively, store the container in an explosion-proof oven, bearing in mind that local surface temperatures might be much higher than the oven temperature reading indicates. The time required to dissolve the wax will depend on the type of wax and the size of the container. For a 1-L (1-qt) tin, 2 h has been found to be adequate. For larger tins, longer times will be

required. Although it is strongly recommended that the containers are closed when heated, after approximately 30 min, it is advised to slowly release the excess pressure before continuing the heating. Mixing can be accomplished by a mechanical shaker or by vigorous manual shaking. Although the use of (high speed) mixers or similar devices might be effective, it will require that the container be open for some time, during which the escape of light ends can be excessive, and hence, this procedure is not recommended. (**Warning**—(1) Exercise care when opening the container as significant vapor pressure will have built up. Opening the container may induce foaming with resultant spillage of sample and possible injury to personnel. (2) During this operation significant amounts of highly flammable vapors might escape. Vent in a safe area. Samples used for custody transfer shall be homogenized and composited following the guidance as provided in Practice **D5854** (API MPMS Chapter 8.3).)

10.4.2 Crude Sample Bottles—Based on the pour point and viscosity, and testing requirements, the sample may require heating, homogenizing and compositing or other necessary steps to prepare the sample, or a portion of the sample. If heating is required, place all samples that will be used to make a composite into the appropriate mechanism to heat the samples. Heat the samples only enough to liquefy the sample, for example maybe 20 to 30°C (36 to 54°F) above the pour point. (**Warning**—Depending on the sample to be tested and test method to be performed, glass sample containers containing crude oil shall be vented slightly during the warming process. Samples shall remain in the heating apparatus until temperature equalizes with environment.) (**Warning**—Exercise special care when heating bottles that are closed with a cork. The pressure increase due to the heating may blow out the cork. Take proper measures to safeguard against such an event. Samples used for custody transfer shall be homogenized and composited following the guidance as provided in Practice **D5854** (API MPMS Chapter 8.3).)

10.4.3 Crude Oil Drums, 15 to 200 L—The most effective way of achieving homogenization is mixing the contents of the drum on a roller bank in a hot room kept at a temperature between 40 and 60°C for 48 h. Alternatively, keep the drum at a temperature of 20°C above the expected pour point for 48 h and roll the drum for at least 15 min before taking a sample. If heating of the drum is not feasible, the only alternative is extensive rolling for at least 30 min to disperse the wax and wax particles as effectively as possible. In order to avoid the cumbersome procedure of (re)mixing the contents of large sample containers, it is recommended to draw an adequate number of sub-samples in smaller containers while the crude oil is considered to be homogenous. (**Warning**—(1) Before embarking on any heating or mixing procedure, or both, ensure that the drum and plugs can withstand the expected pressure build-up and can be handled safely without leakage. (2) Exercise care when opening the container as significant vapor pressure will have built up. Opening the container may induce foaming with resultant spillage of sample and possible injury to personnel.)

Refined Products

10.5 Gasoline and Distillate Products:

10.5.1 Gasoline and light distillate products are usually homogeneous, but they are often shipped from tanks that have clearly separated water on the bottom. Tank sampling of gasoline and distillate products, in accordance with the procedures outlined in Section 9 is acceptable.

10.5.2 *Light-Sensitive Samples*—It is important that samples sensitive to light, such as gasoline, be kept in the dark if the testing is to include the determination of such properties as color, octane, sludge-forming characteristics, stability tests, or neutralization value. Brown glass bottles may be used. Wrap or cover clear glass bottles immediately after filling.

10.5.3 *Sample Handling of Refined Materials*—Protect highly-refined products from moisture and dust by placing paper, plastic, or metal foil over the stopper and the top of the container.

10.5.4 *Distillation of Petroleum Products*—When obtaining samples of volatile liquids that are to be tested using Test Method **D86**, a sample maintained in the primary sample container is recommended because the sample is taken directly in the bottle. Before obtaining the sample, pre-cool the bottle by immersing it in the product, allowing it to fill, and discarding the first filling. If the bottle procedure cannot be used, obtain the sample by the tap sampling procedure. Do not agitate the bottle while drawing the sample. After obtaining the tap sample, close the bottle immediately with a tight-fitting stopper and store it in an ice bath or refrigerator at a temperature of 0 to 4.5°C (32 to 40°F). Refer to Test Method **D86**.

10.5.5 *Vapor Pressure*—When sampling petroleum and petroleum products that are to be tested for vapor pressure, refer to Practice **D5842** (API MPMS Chapter 8.4).

10.6 *Oxidation Stability Precautions*—Very small amounts (as low as 0.001 %) of some materials, such as inhibitors, have a considerable effect on oxidation stability tests. Avoid contamination and exposure to light while taking and handling samples. To prevent undue agitation with air, that promotes oxidation, do not pour, shake, or stir samples to any greater extent than necessary. Never expose them to temperatures above those necessitated by atmospheric conditions.

10.7 *Sample Containers*—Use only brown glass or wrapped clear glass bottles as containers, since it is difficult to make certain that cans are free of contaminants, such as rust and soldering flux. Adequately prepare and clean bottles as described in 6.6. Rinse thoroughly with distilled water, dry, and protect the bottles from dust and dirt.

10.8 *Sampling*—A sample maintained in the primary sample container is recommended because the sample is taken directly in the bottle. This reduces the possibility of air absorption, loss of vapors, and contamination. Just before sampling, rinse the bottle with the product to be sampled.

10.9 *Aviation Fuel*—When sampling aviation fuel, in addition to the individual test method, Practice **D4306** should be consulted for recommended cleaning procedures for containers related to specific test methods.

10.9.1 Sampling equipment fabricated from copper or its alloys shall not be used for sampling aviation fuels.

10.9.2 Before sampling, the sampler and the container shall be flushed and rinsed thoroughly at least three times with the product to be sampled and allowed to drain before use.

10.10 *Residual Fuel Oils/Bunker Fuel*—Residual fuel oils usually are non-homogeneous. Tank samples of residual oils may not be representative for the following reasons:

(1) The concentration of entrained water is generally higher near the bottom. The running sample or the composite of the upper, middle, and lower sample may not represent the concentration of entrained water.

(2) The interface between oil and free water is difficult to measure, especially in the presence of emulsion layers, or sludge.

(3) The determination of the volume of free water is difficult because the free water level may vary across the tank bottom surface. The bottom is often covered by pools of free water or water emulsion impounded by layers of sludge or wax.

10.11 *Asphaltic Materials*—When sampling asphaltic materials that are to be tested using Test Methods **D1856** or **D2172**, obtain samples by the boring procedure or the grab procedure. A sample of sufficient size to yield at least 100 g (3.5 oz) of recovered bitumen is required. About 1 kg (2 lb) of sheet asphalt mixtures usually will be sufficient. If the largest lumps in the sample are 2.5 cm (1 in.), 1.8 kg (4 lb) will usually be required and still larger samples if the mixtures contain larger aggregates.

10.11.1 *Emulsified Asphalts*—It is frequently necessary to test samples in accordance with the requirements of Specification **D977** and Test Methods **D244**. Obtain samples from tanks, tank cars, and tank trucks by either the core thief or bottle sampling procedure using a bottle that has a 4-cm (1.5-in.) diameter or larger mouth. Also, refer to Practice **D140**. Use the dipper procedure to obtain samples for fill or discharge lines. **Table 7** should be consulted for sampling packages. If the material is solid or semisolid, use the boring sampling procedure. Obtain at least 4 L (1 gal) or 4.5 kg (9.9 lb) from each lot or shipment. Store the samples in clean, airtight containers at a temperature of not less than 4°C (39°F) until tested. Use a glass or black iron container for emulsified asphalts of the RS-1 (Rapid Setting) type, as referenced in Specification **D977**.

10.12 *Industrial Aromatic Hydrocarbons*—*Industrial Aromatic Hydrocarbons*—For samples of industrial aromatic hydrocarbons (benzene, toluene, xylene, and solvent naphthas), proceed in accordance with Sections 6-9, with particular emphasis on the procedures pertaining to precautions for care and cleanliness.

10.13 *Lacquer Solvents and Diluents*—When sampling bulk shipments of lacquer solvents and diluents that are to be tested using Guide **D268** observe the precautions and instructions described in 6-9, with particular emphasis on the procedures pertaining to precautions for care and cleanliness.

10.14 *Grease Sampling*—This procedure covers practices for obtaining samples from production lots, shipments of lubricating greases, soft waxes, or soft bitumens similar to

TABLE 8 Size of Grease Samples

Container	Lot or Shipment	Minimum Sample
Tubes or packages, less than 0.45 kg (1 lb)	All	Enough units for a 4.4 kg (10 lb) sample
0.45 kg (1 lb) cans	All	Three cans
2.3 or 4.6 kg (5 or 10 lb) cans	All	One can
Larger than 4.6 kg (10 lb)	Less than 4536 kg (10 000 lb)	1 to 1.4 kg (2 to 3 lb) from one or more containers
Larger than 4.6 kg (10 lb)	4536 to 22 680 kg (10 000 to 50 000 lb)	1 to 2.3 kg (2 to 5 lb) from two or more containers
Larger than 4.6 kg (10 lb)	More than 22 680 kg (50 000 lb)	1 to 2.3 kg (2 to 5 lb) from three or more containers

grease in consistency. This procedure is quite general because a wide variety of conditions are often encountered, and the procedure may have to be modified to meet individual specifications.

10.14.1 Inspection:

(1) If the material is lubricating grease and inspection is made at the manufacturing plant, take samples from finished shipping containers of each production batch or lot. Never take grease samples directly from grease kettles, cooling pans, tanks, or processing equipment. Do not sample the grease until it has cooled to a temperature not more than 9°C (16°F) above that of the air surrounding the containers and it has been in the finished containers for at least 12 h. When the containers for a production batch of grease are of different size, treat the grease in each container size as a separate lot. When inspection is made at the place of delivery, obtain a sample from each shipment. If a shipment consists of containers from more than one production batch (lot numbers), sample each batch separately.

(2) If the material being inspected is of grease-like consistency, but is not actually lubricating grease but some mixture of heavy hydrocarbons, such as microcrystalline waxes or soft bitumens, it is permissible to take samples from pans, tanks, or other processing equipment, as well as from containers of the finished product. The grease sampling method shall be applicable to such stocks only if for some reason it is not possible to apply heat and convert the material into a true liquid.

10.14.2 *Sample Size*—Select containers at random from each lot or shipment to give the required quantity specified in Table 8.

10.14.3 Grease Sampling Procedure:

(1) Examine the opened containers to determine whether the grease is homogeneous, comparing the grease nearest the outer surfaces of the container with that in the center, at least 15 cm below the top surface, for texture and consistency. When more than one container of a lot or shipment is opened, compare the grease in all open containers.

(2) If no marked difference in the grease is found, take one portion from the approximate center and at least 7.5 cm (3 in.) below the surface of each opened container in sufficient quantity to provide a composite sample of the desired quantity (see Table 8). Withdraw portions with a clean scoop, large spoon, or spatula and place them in a clean container. Very soft,

semi-fluid greases may be sampled by dipping with a 0.45-kg (1 lb) can or suitable dipper. If any marked difference in the grease from the various locations of an opened container is found, take two separate samples of about 0.45 kg (1 lb) each, one from the top surface adjacent to the wall and the other from the center of the container, at least 15 cm (6 in.) below the top surface. If any marked variations are noted between different containers of a lot or shipment, take separate samples of about 0.45 kg (1 lb) from each container. When taking more than one sample of a batch or shipment, because of lack of uniformity, send them to the laboratory as separate samples.

(3) If more than one portion is required to represent a lot or shipment of grease softer than 175 penetrations (refer to Test Methods D217), prepare a composite sample from the containers of the same size and batch by mixing equal portions thoroughly. Use a large spoon or spatula and a clean container. Avoid vigorous mixing or working of air into the grease. As grease samples become partially “worked” in being removed from containers, the procedure is not suitable for obtaining samples of greases softer than 175 penetration on which unworked penetration is to be determined. For greases having a penetration of less than 175, cut samples from each container with a knife in the form of blocks about 15 by 15 by 5 cm (6 by 6 by 2 in.). If required, make unworked penetration tests on blocks as procured and other inspection tests on grease cut from the blocks.

11. Keywords

11.1 automatic sampler; all-levels sample; boring sample; boring sampling; bottle/beaker sampling; bottom sample; bottom water sample; clearance sample; composite sample; core sample; core thief spot sampling; dead bottom sample; deck composite sample; dipper sample; dipper sampling; dissolved water; drain sample; emulsion; entrained water; extended tube sampling; floating piston cylinder; floating roof sample; free water; grab sample; grab sampling; grease sample; grease sampling; high pressure sample cylinder; intermediate sample container; loading zone sample; lower sample; marine custody transfer; maximum fill density (reduced fill density); middle sample; multiple tank composite sample; outage tube (internal); portable manual sampling unit; PSU; primary sample container; representative sample; running sample; sample; sample containers; sample handling; sample labeling; sample mixing; sample shipment; sampling; sampling cage; spot

sample; standpipes; static sampling; suction sample (outlet);
 sump sample; surface (skim) sample; tank composite sample;
 tap sample; tap sampling; test specimen; top sample; tube or

thief sample; tube sampling; upper sample; volumetric com-
 posite sample; zone sample

APPENDIX

(Nonmandatory Information)

X1. CRUDE OIL SAMPLE HANDLING—TEST MATRIX

X1.1 See [Table X1.1](#).

TABLE X1.1 Crude Oil Sample Handling—Test Matrix

Test Name	Test Method ASTM	Test Method Specific Guidance on Sample Container Use or Material for Immediate Testing ^{A,B}	Typical Sample Volume Required for Test (mL)	Special Field/Lab Handling of Sample Container ^{C,D}
Acid Number of Petroleum Products by Potentiometric Titration	D664	Heat the sample to 60°C in the original container and agitate until all of the sediment is homogeneously suspended. If the original container is a can or if it is glass and more than three-fourths full, transfer the entire sample to a clear-glass bottle having a capacity at least one third greater than the volume of the sample. Transfer all traces of sediment from the original container to the bottle by vigorous agitation of portions of the sample in the original container.	50	Strict observance of the sampling procedure is necessary since the sediment itself is acidic or basic or has absorbed acidic or basic material from the sample. Failure to obtain a representative sample causes serious errors. As oil can change appreciably in storage, test samples as soon as possible and note the date of sampling and testing.
API Gravity by Hydrometer Density, Relative Density (Specific Gravity), or API Gravity by Hydrometer Density, Relative Density, and API Gravity by Thermohydrometer	D287 D1298/API 9.1 D6822/API 9.3	Whenever possible, mix the sample in its original closed container in order to minimize the loss of light components, especially with waxy and volatile crude oils. Refer to test method for details. Refer to Table 2 in Test Method D287 regarding original closed container.	600	Extreme care shall be taken to minimize losses of light components, including the transfer of the sample to a chilled container immediately after sampling. Mixing volatile samples in open containers can lead to loss of light components and possibly affect the value of the density obtained, so mixing in closed, pressurized containers or at sub-ambient temperatures is recommended. If the crude oil has a pour point above 10°C, or a cloud point or WAT above 15°C, warm the sample to 9°C above the pour point, or 3°C above the cloud point or WAT, prior to mixing. For crude petroleum, bring the sample close to the reference temperature or, if wax is present, to 9°C above its pour point or 3°C above its cloud point or WAT, whichever is higher. Bring the hydrometer cylinder and thermometer to within approximately 5°C of the test temperature.
Density AND Relative Density for Crude Oils by Digital Density Analyzer	D5002	The test specimen can be taken from either a properly mixed laboratory sample, using an appropriate syringe or directly from the mixing container if the proper density analyzer attachments and connecting tubes are used.	2	
Asphaltenes (Heptane Insolubles) in Crude Petroleum and Petroleum Products	D6560		100	Test portions from the laboratory samples shall be drawn after thorough mixing and subdivision.
Carbon Residue (Micro Method -MCRT)	D4530		15	Stir the sample to be tested, first warming if necessary to reduce its viscosity. Samples that are homogeneous liquids can be transferred directly to vials using a rod, syringe, or eyedropper.
Organic Chloride Content in Crude Oil	D4929		500	To preserve volatile components, which are in some samples, do not uncover samples any longer than necessary. Samples should be analyzed as soon as possible, after taking from bulk supplies, to prevent loss of organic chloride or contamination due to exposure or contact with sample container. When heating is required, care should be taken so that no organic chloride containing hydrocarbons are lost.
Boiling Point Distribution of Crude Oil by High Temperature Gas Chromatography (High Temperature Simulated Distillation (HTSD))	D7169		10	Ensure that the sample is a representative sample. Care should be taken that the solution is prepared a short time prior to running the analysis. Samples can be stored in the auto sampler vials. Store crude oil samples at 4°C or below until ready for analysis. If the sample is submitted for other analyses, remove a small aliquot (~10 mL) early in the testing sequence in order to avoid loss of volatile components.
Mercaptan Sulfur	UOP163		200	Minimize sample exposure to air.

TABLE X1.1 Continued

Test Name	Test Method ASTM	Test Method Specific Guidance on Sample Container Use or Material for Immediate Testing ^{A,B}	Typical Sample Volume Required for Test (mL)	Special Field/Lab Handling of Sample Container ^{C,D}
Metals by AA (Ni, V, Fe, Na)	D5863, Method B		25	Prior to weighing, stir the sample and then shake the sample in its container. Employ adequate mixing and sampling procedures for crude and heavy oils. Use paint mixers for mixing of crude oils. If the sample does not readily flow at room temperature, heat the sample to a sufficiently high and safe temperature to ensure adequate fluidity.
Trace Nitrogen in Liquid Petroleum Hydrocarbons by Syringe/Inlet Oxidative Combustion and Chemiluminescence Detection	D4629		3	Test samples should be analyzed as soon as possible after taking from bulk supplies to prevent loss of nitrogen or contamination due to exposure or contact with the sample container. If the test sample is not used immediately, then thoroughly mix it in its container prior to taking a test specimen. Some test samples require heating in order to thoroughly homogenize.
Nitrogen by Boat-Inlet Chemiluminescence	D5762			
Pour Point of Petroleum Products	D97		50	The pour point of crude oils is very sensitive to trace amounts of high melting waxes. Exercise meticulous care to ensure such waxes, if present, are either completely melted or, if volatility constraints prevent heating to complete melting, homogeneously suspended in the sample. Inspect the walls of the original container to ensure that no high melting point material is left sticking to the wall. If necessary, reheat the test sample to a temperature at least 20°C above the expected pour point but not higher than a temperature of 60°C. It is not possible to define universal mandatory rules for the preparation of crude oil test samples.
Pour Point for Crude Oils	D5853, Method A			
S & W by Centrifuge—Field Procedure	API 10.4		100	Refer to API 10.4 and API 18.1
S & W by Centrifuge—Laboratory Procedure	D4007/API 10.3		100	Sample mixing is typically required to obtain a test portion representative of the bulk sample to be tested, but precautions shall be taken to maintain the integrity of the sample during this operation. Mixing of volatile crude petroleum containing water or sediments, or both, may result in the loss of light components. Additional information on the mixing and handling of liquid samples can be found in Practice D5854 (API MPMS Chapter 8.3)
Salts in Crude Oil (Electrometric Method)	D3230		10	The presence of water and sediment will influence the conductivity of the sample. The utmost care shall be taken in obtaining homogenized representative samples. Samples of very viscous materials may be warmed until they are reasonably fluid before they are sampled; however, no sample shall be heated more than is necessary to lower the viscosity to a manageable level.
Salt in Crude Oil (Potentiometric Method)	D6470		50	Homogenize the sample within 15 min of drawing the test sample. Mix the sample at room temperature (15 to 25°C), or less, in the laboratory sample container, and record the temperature of the sample immediately before mixing. Heat waxy samples, solid at room temperature, to 3°C above their pour point in order to facilitate test sample withdrawal. Select the type of mixer related to the quantity of crude oil in the laboratory sample container. Sample mixers are to be verified for each type of crude oil, size of sample and shape of container, and change in speed or time of mixing, following guidelines in Annex A1 of the test method. For small laboratory sample containers and volumes, 50 to 500 mL, a non-aerating, high speed (3000 r/min), shear mixer is required. Use the mixing time, mixing speed, and height above the bottom of the container found to be satisfactory to Annex A1. For larger containers and volumes, appropriate mixing conditions shall be defined by following a set of procedures similar to those outlined in Annex A1, and Practice D4177 (API MPMS Chapter 8.2) but modified for application to the larger containers and volumes. Clean and dry the mixer between samples. Record the temperature of the sample immediately after homogenization. The rise in temperature from the initial reading shall not exceed 10°C, otherwise excessive loss of volatile vapors can occur or the dispersion can become unstable. In order to ensure that crude oils with rapidly settling impurities are properly sampled, withdraw the test sample container immediately after homogenization by lowering the tip of the sample tube almost to the bottom of the container, and withdrawing the test sample as quickly as possible. Clean and dry the sample tube before and after sampling.

TABLE X1.1 Continued

Test Name	Test Method ASTM	Test Method Specific Guidance on Sample Container Use or Material for Immediate Testing ^{A,B}	Typical Sample Volume Required for Test (mL)	Special Field/Lab Handling of Sample Container ^{C,D}
Sediment by Extraction in Crude Oils and Fuel Oils by the Extraction Method	D473/API 10.1		20	Draw test portions from the laboratory samples immediately after thorough mixing. Heat viscous samples to a temperature which renders the sample liquid, and homogenize. The difficulties of obtaining a representative test portion for this determination are usually great, hence the need for great attention to the mixing and aliquoting steps. As specified in Practice D5854 (API MPMS Chapter 8.3) record the temperature of the sample before mixing. Mix the laboratory sample in its original container, using the mixing time, mixing power (speed), and position relative to the bottom of the container appropriate to the crude oil being analyzed, and as established by the verification of mixing efficiency. To avoid the loss of light ends operate the mixer through a gland in the closure of the sample container. Take the test portion for analysis immediately after mixing. Record the temperature of the sample immediately after mixing. If the rise in temperature during mixing exceeds 10°C (20°F), cool the sample, and repeat the mixing at a lower energy input. A rise in temperature greater than 10°C (20°F) may result in a decrease in viscosity that is sufficient for the sediment to settle.
Sediment in Crude Oil by Membrane Filtration	D4807/API 10.8	Analyze samples within two weeks after taking the sample. Retaining samples longer may affect the results.	20	Mixing is required to properly disperse sediment as well as any water present in the sample. Mix the sample at room temperature in the original container within 15 min of testing analysis to ensure complete homogeneity. A test sample drawn directly from a large volume dynamic mixing system shall be analyzed within 15 min or less. Analysis should follow mixing as soon as possible. The 15-min interval is a general guideline which may not apply to all crudes, especially some light crudes which do not hold water and sediment in suspension for even this short a time. Mixing of the sample should not increase the temperature of the sample more than 10°C (20°F) or a loss of water may occur affecting the sample's composition. The type of mixer depends on the quantity of crude. Before any unknown mixer is used, the specifications for the homogenization test, Practice D5854 (API MPMS Chapter 8.3), must be met. The mixer must be re-evaluated following any changes in the type of crude, quantity of crude, or shape of the sample container. For small test sample volumes, 50 to 300 mL, a non-aerating, high-speed, shear mixer is required. Use the mixing time, mixing speed, and height above the bottom of the container found to be satisfactory in Practice D5854 (API MPMS Chapter 8.3). Clean and dry the mixer between samples.
Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry	D2622		25 mL	
Sulfur Content	D4294		500 mL	
Vapor Pressure of Petroleum Products (Reid Method)	D323	Sample Container Size—The size of the sample container from which the vapor pressure sample is taken shall be 1 L (1 qt). It shall be 70 to 80 % filled with sample. In no case shall any part of the Reid (D323-specific) apparatus itself be used as the sample container prior to actually conducting the test.	1000	Test to be performed on samples from primary sample container, and not a composite. The extreme sensitivity of vapor pressure measurements to losses through evaporation and the resulting changes in composition is such as to require the utmost precaution and the most meticulous care in the handling of samples. The Reid vapor pressure determination shall be performed on the first test specimen withdrawn from the sample container. The remaining sample in the container cannot be used for a second vapor pressure determination. Protect samples from excessive temperatures prior to testing. Do not test samples in leaky containers. They should be discarded and new samples obtained. Cool the sample container and contents to 0 to 1°C (32 to 34°F) before the container is opened. Sufficient time to reach this temperature shall be ensured by direct measurement of the temperature of a similar liquid in a like container placed in the cooling bath at the same time as the sample. Refer to Practice D5854 (API MPMS Chapter 8.3).
Determination of Vapor Pressure of Crude Oil: VPCR (Expansion Method)	D6377	If crude was non-pressurized and sampled following ASTM D4057 or D4177	500	

TABLE X1.1 Continued

Test Name	Test Method ASTM	Test Method Specific Guidance on Sample Container Use or Material for Immediate Testing ^{A,B}	Typical Sample Volume Required for Test (mL)	Special Field/Lab Handling of Sample Container ^{C,D}
Determination of Vapor Pressure of Crude Oil:	D6377	If crude was pressurized and sampled following D3700 or D4177	500	Sampling of live crude oil shall be performed in accordance with Practice D3700 , and specific guidance in ASTM D6377 . If the sample is contained in a pressurized source like a pipeline, use a floating piston cylinder and obtain the sample directly from the source under pressure. Rinse the cylinder by opening the rinsing valve until the crude oil emerges at the second inlet. Close the rinsing valve and let the piston move slowly until at least 200 mL of sample has entered the cylinder. Close the inlet valve and apply the back pressure immediately. Check the filling of the cylinder to be at least 200 mL. The current precision statements were derived from the 2005 ILS using samples in 250 mL floating piston cylinders. Do not unnecessarily expose the samples to temperatures exceeding 30°C during sampling and storage. For prolonged storage, store the samples in an appropriate room or refrigerator. Perform the vapor pressure determination on the first test specimen withdrawn from the cylinder after the rinsing step in the test method. Do not use the remaining sample in the floating piston cylinder for more than three repeat vapor pressure determinations. Transfer the sample from the cylinder container into the measuring cell at room temperature but at least 5°C above the pour point. If the sample is contained in a pressurized floating piston cylinder, apply a back-pressure which is higher than the vapor pressure of the sample at the introduction temperature plus a minimum of 100 kPa for the piston movement. The applied back-pressure shall not exceed the maximum limit of the pressure transducer used in the vapor pressure apparatus.
Viscosity - Kinematic	D445		100	Heat in the original container, in an oven, at 60 ± 2°C for 1 h. Thoroughly stir the sample with a suitable rod of sufficient length to reach the bottom of the container. Continue stirring until there is no sludge or wax adhering to the rod. Recap the container tightly and shake vigorously for 1 min to complete the mixing. With samples of a very waxy nature or oils of high kinematic viscosity, it may be necessary to increase the heating temperature above 60°C to achieve proper mixing. The sample should be sufficiently fluid for ease of stirring and shaking. Immediately after mixing, pour sufficient sample to fill two viscometers into a 100-mL glass flask and loosely stopper.
Water in Crude Oil by Distillation	D4006 / API 10.2		200	
Water in Crude Oils by Potentiometric Karl Fischer Titration	D4377 / API 10.7		50	
Water Content by Karl Fisher (Coulometric)	D4928 / API 10.9			Mix the test sample of crude oil within 15 min before analysis to insure complete homogeneity. Mix the test sample at room temperature (25°C) in the original container. Mixing of the sample should not increase the temperature of the sample more than 10°C, or a loss of water may occur. The type of mixer depends on the quantity of crude. Before any unknown mixer is used, the specifications for the homogenization test, Annex A1 of the Test Method (or Practice D5854 (API MPMS Chapter 8.3) for Test Method D4929), must be met. The mixer must be re-evaluated for any changes in the type of crude, quantity of crude, or shape of the sample container. For small sample volumes, 50 to 500 mL, a non-aerating, high speed, shear mixer is required. Use the mixing time, mixing speed, and height above the bottom of the container found to be satisfactory in Annex A1 of the Test Method (or Practice D5854 (API MPMS Chapter 8.3)). Clean and dry the mixer between samples.

^A In addition to the specific test method, for sample container guidance for immediate testing, sample storage, and sample container reuse, refer to Practice **D5854** (API MPMS Chapter 8.3).

^B At minimum, all sample containers are to be clean and dry.

^C All sample containers should allow 15 to 30 % ullage for possible expansion. In addition, with the exception of vapor pressure test methods, samples are to be homogenized every time the sample is being transferred and immediately prior to testing.

^D The most current test method instructions will take precedence over anything contained within this matrix.

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