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Manual of Petroleum Measurement Standards (MPMS), Chapter 8.1

Standard Practice for Manual Sampling of Petroleum and Petroleum Products[1]

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 ~~covers procedures for manually obtaining representative samples of petroleum products of a liquid, semi-liquid, or solid state whose vapor pressure at ambient conditions is below 101 kPa (14.7 psia).~~ 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 of samples,~~ handling, refer to Practice D5854 (API *MPMS* Chapter 8.3). ~~The practice does not cover sampling of electrical insulating oils and hydraulic fluids.~~

~~NOTE 1—The procedures described in this practice may also be applicable in sampling most noncorrosive liquid industrial chemicals, provided that all safety precautions specific to these chemicals are strictly followed.~~

~~NOTE 2—The procedure for sampling liquefied petroleum gases is described in Practice D1265; the procedure for sampling fluid power hydraulic fluids~~

[1] This practice is under the jurisdiction of ASTM Committee D02 on Petroleum ~~Products~~ Products, Liquid Fuels, and Lubricants and the API Committee on Petroleum Measurement and is the direct responsibility of Subcommittee D02.02 /COMQ ~~the joint ASTM-API committee~~ on Hydrocarbon Measurement for Custody Transfer (Joint ASTM-API). This ~~test method~~ practice has been approved by the sponsoring committees and accepted by the Cooperating Societies in accordance with established procedures. This ~~test method~~ practice was issued as a joint ASTM-API standard in 1981.

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~~is covered in ANSI B93.19 and B93.44; the procedure for sampling insulating oils is described in Practice D923; and the procedure for sampling natural gas is described in Test Method D1145.~~

~~NOTE 3—The procedure for special fuel samples for trace metal analysis is described in an appendix to Specification D2880.~~

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.

1.6 *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
~~D525~~D445 Test Method for ~~Oxidation Stability of Gasoline (Induction Period Method)~~Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
~~D873~~D473 Test Method for ~~Oxidation Stability of Aviation Fuels (Potential Residue Method)~~Sediment in Crude Oils and Fuel Oils by the Extraction Method (API *MPMS* Chapter 10.1)
~~D923~~D664 ~~Practices for Sampling Electrical Insulating Liquids~~Test Method for Acid Number of Petroleum Products by Potentiometric Titration
D977 Specification for Emulsified Asphalt
~~D1145 Test Method for Sampling Natural Gas (Withdrawn 1986)~~[3]
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
~~D2880~~D2622 ~~Specification for Gas Turbine Fuel Oils~~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

[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.

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)
~~D4865~~D5762 ~~Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems~~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)
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 *American National Standards:*~~[4]
~~B93.19 Standard Method for Extraction Fluid Samples from the Lines of an Operating Hydraulic Fluid Power System (for Particulate Contamination Analysis)~~
~~B93.44 Method for Extracting Fluid Samples from the Reservoir of an Operating Hydraulic Fluid Power System~~
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 ~~Thermohydrometer~~Standard Test Method for ~~Density~~ Density, Relative Density, and API Gravity of Crude Petroleum and Liquid Petroleum Products by Thermohydrometer Method (ASTM Test Method D6822)
MPMS Chapter ~~10,~~10.1 ~~various sections, Sediment and Water Determination~~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

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

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 Titration[5]
49 CFR 173 Shippers—General Requirements for Shipments and Packagings[6]

3. Terminology

3.1 ~~*Definitions of Terms Specific to This Standard:*~~*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.

[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.

3.1.3.1 *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.

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.

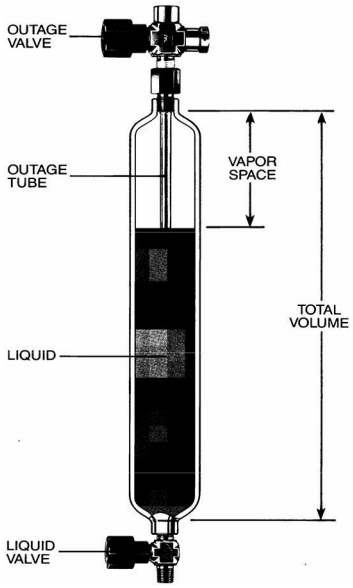


FIG. 1 Example of a Fixed Volume Cylinder with an Outage Tube

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.

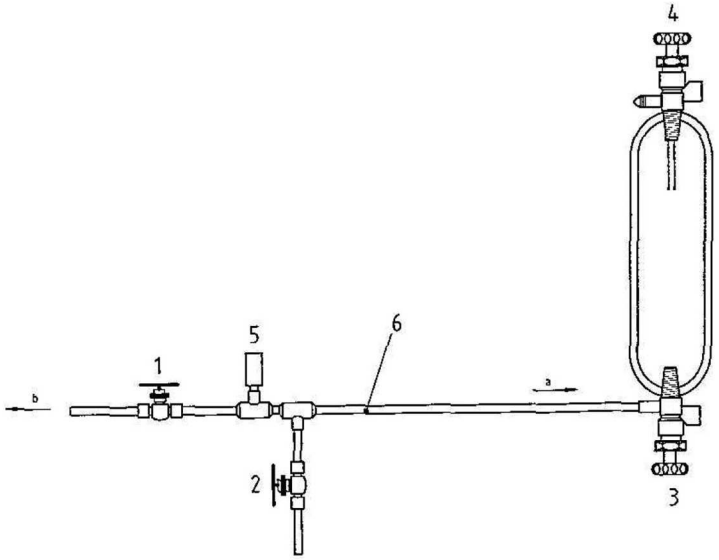
3.1.27 *ullage (outage), n*—the volume of available space in a container unoccupied by contents.

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.



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

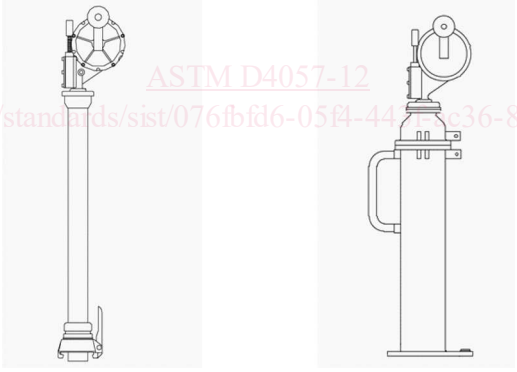


FIG. 3 Examples of a Small Volume (5 cm (2 in.)) and a Large Volume PSU (10 cm (4 in.))

~~Samples~~Sample Types

3.1.30 *all-levels ~~sample—~~sample, n—*a sample obtained by ~~submerging a stoppered beaker or bottle to a point as near as possible to the draw-off level,~~ 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 ~~approximately three-fourths full as it emerges from the liquid.~~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.

3.1.30.1 *Discussion—*

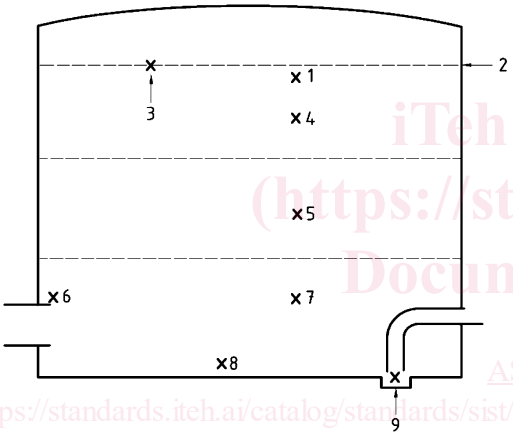
If required by the test method, the sampler may be greater than 85 % full when withdrawn but in no case shall it be completely full. In these cases, take special handling precautions to consider the hazards associated with product thermal expansion.

FIG. 4 Examples of Closed/Restricted Sampling Equipment

3.1.31 *boring* ~~*sample—*~~*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—*~~*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.

~~3.1.3.1 *Discussion—*~~



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. ~~1~~5 ~~Spot Sampling Locations~~Illustration of Common Spot Sample Positions

~~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 from the bottom) should be specified when using this term.~~

3.1.33 *bottom water* ~~*sample—*~~*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—*~~*sample, n—*a spot sample taken with the inlet opening of the sampling ~~apparatus~~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 m^3 (1000 barrels) or less) tanks, commonly referred to as lease tanks.

~~3.1.5.1 *Discussion—*~~

~~This term is normally associated with small (159 m^3 or 1000 Bbls or less) tanks, commonly referred to as lease tanks.~~

3.1.35 *composite ~~sample—~~sample, n—*a ~~blend of spot samples mixed in proportion to the volumes of material from which the spot samples were obtained.~~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—~~sample, n—*a sample of uniform ~~cross sectional~~ 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.

3.1.39 *dipper ~~sample—~~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—~~sample, n—*a sample obtained from the water draw-off valve on a storage ~~tank.~~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).

~~3.1.9.1 *Discussion—*~~

~~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—~~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.

~~3.1.11 *flow proportional sample—*a sample taken from a pipe such that the rate of sampling is proportional throughout the sampling period to the flow rate of the fluid in the pipe.~~

3.1.42 *grab ~~sample—~~sample, n—(a) solid*—a sample obtained by collecting equal quantities from parts or packages of a shipment of loose solids ~~such~~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.

3.1.43 *grease ~~sample—~~sample, n—*~~a sample~~ 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—~~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~~liquid's surface). See Fig. ~~1~~5.

3.1.46 *middle ~~sample—~~sample, n—*a spot sample taken from the middle of a tank's contents (a distance of ~~one-half~~ one half of the depth of liquid below the ~~liquid's~~liquid's surface). See Fig. ~~1~~5.

3.1.47 *multiple tank composite ~~sample—~~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.

~~3.1.16.1 *Discussion—*~~

~~The mixture is blended in proportion to the volume of material contained in the respective tanks or compartments.~~

~~3.1.17 *outlet sample—*a spot sample taken with the inlet opening of the sampling apparatus at the level of the bottom of the tank outlet (fixed or floating). See Fig. 1.~~

3.1.48 *representative ~~sample—~~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—~~sample, n—*a sample obtained by lowering ~~a breaker or bottle~~an open sampling device to the ~~level of the~~ bottom of the outlet ~~connection or swing line~~ suction level, but always above free water, and returning it to the top of the ~~oil~~product at a uniform rate such that the ~~beaker or bottle is about three-fourths~~ sampling device is between 70 and 85 % full when withdrawn from the ~~oil.~~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.

TABLE ~~6 Sample Tap Specifications~~

~~colwidth="1.46in"> colname="col2" colwidth="1.06in"> colname="col3" colwidth="0.96in">~~

~~Tank Capacity~~	~~1590 m³ (10 000 bbls) Or Less~~	~~Greater than 1590 m³ (10 000 bbls)~~
~~Number of Sets~~	~~1~~	~~2[A]~~
~~Number of taps per set, min~~	~~3~~	~~5~~
~~Vertical location~~		
~~—Upper tap~~	~~45 cm (18 in.) from top of shell~~	
~~—Lower tap~~	~~even with bottom of outlet~~	
~~—Middle tap(s)~~	~~equally spaced between upper and lower tap~~	
~~Circumferential location~~		
~~—From inlet~~	~~2.4 m (8 ft), min~~	
~~—From outlet/drain~~	~~1.6 m (6 ft), min~~	

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

~~[A] The respective sets of taps should be located on opposite sides of the tank.~~

~~3.1.20 *sample*—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.~~

~~3.1.21 *sampling*—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.~~

3.1.50 *spot ~~sample—~~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—~~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—~~sample, n—*a blend created from a single tank, as an example combining the upper, middle, and lower ~~samples from a single tank.~~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.

~~3.1.24.1 *Discussion*—~~

~~For a tank of uniform cross section, such as an upright cylindrical tank, the blend consists of equal parts of the three samples. For a horizontal cylindrical tank, the blend consists of three samples in the proportions shown in Table 1.~~

3.1.55 *tank tap ~~sample—~~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—~~sample, n—*a spot sample obtained 15 cm (6 in.) below the top surface of the liquid. See Fig. ~~1~~5.

3.1.58 *tube ~~or thief sample—~~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—~~sample, n—*a spot sample taken from the middle of the upper ~~one-third~~ one third of the ~~tank's~~tank's contents (a distance of one-sixth of the ~~liquid depth~~ depth of the liquid below the liquid's surface). See Fig. ~~1~~5.

TABLE 1 Sampling Instructions for Horizontal Cylindrical Tanks

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Liquid Depth (% of Diameter)	Sampling Level (% of Diameter Above Bottom)			Composite Sample (Proportionate Parts Of)		
	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

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.

Other Terms

3.1.60 *automatic sampler—volumetric composite sample, n—*a device used to extract a representative sample from the liquid flowing in a pipe.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.

3.1.29.1 *Discussion—*

The automatic sampler generally consists of a probe, a sample extractor, an associated controller, a flow measuring device, and a sample receiver. For additional information on an automatic sampler, see Practice D4177 (API *MPMS* Chapter 8.2).

3.1.30 *dissolved water—*water in solution in an oil.

3.1.31 *emulsion—*an oil/water mixture that does not readily separate.

3.1.32 *entrained water*—water suspended in the oil.

3.1.32.1 *Discussion*—

Entrained water includes emulsions but does not include dissolved water.

3.1.33 *free water*—the water that exists as a separate phase.

3.1.61 *intermediate container*—*zone sample, n*—the vessel into which all or a sample taken as that part of the sample from a primary container/receiver is transferred for transport, storage, or ease of handling.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.

3.1.35 *primary sample receiver/receptacle*—a container in which a sample is initially collected.

3.1.35.1 *Discussion*—

Examples of primary sampler containers include glass and plastic bottles, cans, core-type thief, and fixed and portable sample receivers.

3.1.36 *stand pipes*—vertical sections of pipe or tubing extending from the gaging platform to near the bottom of tanks that are equipped with external or internal floating roofs.

3.1.36.1 *Discussion*—

Stand pipes may also be found on ships and barges.

3.1.37 *test specimen*—the representative sample taken from the primary or intermediate sample container for analysis.

4. Summary of Practice

4.1 This practice provides procedures for manually obtaining samples of petroleum and petroleum products of a liquid, semi-liquid or solid state from tanks, pipelines, drums, barrels, cans, tubes, bags, kettles and open-discharge streams. It addresses, in detail, the various factors which need to be considered in obtaining a representative sample. These considerations include the analytical tests to be conducted on the sample, the types of sample containers to be used and any special instructions required for special materials to be sampled. Test Method D5854 (API *MPMS* Chapter 8.3) can provide additional guidance. A summary of the manual sampling procedures and their applications is presented in Table 2.

4. Significance and Use

4.1 Representative samples Samples of petroleum and petroleum products are required for obtained for many reasons, including the determination of chemical and physical properties, which are used to establish standard volumes, prices, and compliance with commercial and regulatory specifications.properties. These properties may be used for: calculating standard volumes; establishing product value; and often safety and regulatory reporting.

4.2 The following concepts must be considered when selecting a specific sampling procedure.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 samples, the sample only represents the column of material from which it was taken.

5.2.1 *Objective of Manual Sampling:*

5.2.1.1 The objective of manual sampling is to obtain a small portion (spot sample) of material from a selected area within a container that is representative of the material in the area or, in the case of running or all-level samples, a sample whose composition is representative of the total material in the container. A series of spot samples may be combined to create a representative sample.

5.2.2 *Required Conditions for the Application of Manual Sampling:*

5.2.2.1 Manual sampling may be applied under all conditions within the scope of this practice, provided that the proper sampling procedures are followed.

5.2.2.2 In many liquid manual sampling applications, the material to be sampled contains a heavy component (such as free water) which tends to separate from the main component. In these cases, manual sampling is appropriate under the following conditions.

(1) Sufficient time must have elapsed for the heavy component to adequately separate and settle.

(2) It must be possible to measure the level of the settled component in order to stay well above that level when drawing representative samples, unless all or part of the heavy component will be included in the portion of the tank contents to be identified.

~~**TABLE 2 Typical Sampling Procedures and Applicability**~~

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~~Application~~	~~Type of Container~~	~~Procedure~~
~~Liquids of more than (13.8 kPa) and not more than 101 kPa (14.7 psia) RVP~~	~~storage tanks, ship and barge tanks, tank cars, tank trucks~~	~~bottle sampling~~
		~~thief sampling~~
~~Liquids of 101 kPa (14.7 psia) RVP or less~~	~~storage tanks with taps~~	~~tap sampling~~
~~Bottom sampling of liquids of 13.8 kPa (2 psia) RVP or less~~	~~storage tanks with taps~~	~~tap sampling~~
~~Liquids of 101 kPa (14.7 psia) RVP or less~~	~~pipes or lines~~	~~pipeline sampling~~
~~Liquids of 13.8 kPa (2 psia) RVP or less~~	~~storage tanks, ships, barges~~	~~bottle sampling~~
~~Liquids of 13.8 kPa (2 psia) RVP or less~~	~~free or open discharge streams~~	~~dipper sampling~~
~~Liquids of 13.8 kPa (2 psia) RVP or less~~	~~drums, barrels, cans~~	~~tube sampling~~
~~Bottom or thief sampling of liquids of 13.8 kPa (2 psia) RVP or less~~	~~tank cars, storage tanks~~	~~thief sampling~~
~~Liquids and semi-liquids of 13.8 kPa (2 psia) RVP or less~~	~~free or open discharge streams; open tanks or kettles with open heads; tank cars, tank trucks, drums~~	~~dipper sampling~~
~~Crude petroleum~~	~~storage tanks, ship and barge, tanks, tank cars, tank trucks, pipelines~~	~~automatic sampling~~
		~~thief sampling~~
		~~bottle sampling~~
		~~tap sampling~~
~~Industrial aromatic hydrocarbons~~	~~storage tanks, ship and barge tanks~~	~~bottle 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~~
~~Asphaltic materials~~	~~storage tanks, tank cars, lines, packages~~	~~...~~
~~Emulsified asphalts~~	~~storage tanks, tank cars, lines, packages~~	~~...~~

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	

~~*(3)* When one or more of these conditions cannot be met, sampling is recommended and is accomplished by means of an automatic sampling system (see Practice D4177 (API *MPMS* Chapter 8.2)).~~

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,

FIG. 26 ~~Stand Pipe (with overlapping slots)~~Typical Sample Carrier

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 ~~Sample containers come in a variety of shapes, sizes, and materials. To be able to select the right container for a given application one must have knowledge of the material to be sampled to ensure that there will be no interaction between the sampled material and the container which would affect the integrity of the other. Additional considerations in the selection of sample containers is the type of mixing required to remix the contents before transferring the sample from the container and the type of laboratory analyses that are to be conducted on the sample. 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 without exceeding 80 % of the container capacity. The additional capacity is required for thermal expansion of the sample and enhances sample mixing.~~*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;

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 *General Container Design Considerations*—Following are general design considerations for sample containers:~~

~~6.2.1 The bottom of the container should be sloped continuously downward to the outlet to ensure complete liquid withdrawal.~~

~~6.2.2 There should be no internal pockets or dead spots.~~

~~6.2.3 Internal surfaces should be 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.