



Designation: ~~D4057-06~~ Designation: D4057 – 06 (Reapproved 2011)



Designation: **Manual of Petroleum Measurement Standards (MPMS), Chapter 8.1**

## Standard Practice for Manual Sampling of Petroleum and Petroleum Products<sup>1</sup>

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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

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

### 1. Scope

1.1 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). 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, refer to Practice D5854 (API *MPMS* Chapter 8.3). ~~The practice does not cover sampling of electrical insulating oils and hydraulic fluids. A summary of the manual sampling procedures and their applications is presented in Table 1, 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 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.

### 2. Referenced Documents

#### 2.1 *ASTM Standards*:<sup>2</sup>

D86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure

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

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

D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis

D525 Test Method for Oxidation Stability of Gasoline (Induction Period Method)

D873 Test Method for Oxidation Stability of Aviation Fuels (Potential Residue Method)

D923 Practices for Sampling Electrical Insulating Liquids

D977 Specification for Emulsified Asphalt

D1145 Test Method for Sampling Natural Gas

D1265 Practice for Sampling Liquefied Petroleum (LP) Gases, Manual Method

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 Specification for Gas Turbine Fuel Oils

D4177 ~~Practice for Automatic Sampling of Petroleum and Petroleum Products~~ Practice for Automatic Sampling of Petroleum and Petroleum Products (API *MPMS* Chapter 8.2)

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

Current edition approved October 1, 2006; 2011. Published November 2006; August 2011. Originally approved in 1981. Last previous edition approved in 2006 as D4057-95(2000); D4057-06. DOI: 10.1520/D4057-06.10.1520/D4057-11.

<sup>2</sup> 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.

- D4306 [Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination](#)
- D4865 [Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems](#)
- D5842 [Practice for Sampling and Handling of Fuels for Volatility Measurement](#) [Practice for Sampling and Handling of Fuels for Volatility Measurement \(API MPMS Chapter 8.4\)](#)
- D5854 [Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products \(API MPMS Chapter 8.3\)](#)
- 2.2 *American National Standards:*<sup>3</sup>
- 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
- 2.3 *API Standards:*<sup>4</sup>
- 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 9.3 Thermohydrometer Test Method for Density and API Gravity of Crude Petroleum and Liquid Petroleum Products
- MPMS Chapter 10, various sections, Sediment and Water Determination
- 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

### 3. Terminology

#### 3.1 Definitions of Terms Specific to This Standard:

##### 3.1.1 *Samples:*

~~3.1.1.1 all-levels sample~~ **all-levels sample**—a sample obtained by submerging a stoppered beaker or bottle to a point as near as possible to the draw-off level, then opening the sampler and raising it at a rate such that it is approximately three-fourths full as it emerges from the liquid.

##### 3.1.1.2 *boring sample*

**boring sample**—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.

##### 3.1.1.3 *bottom sample*

**bottom sample**—a spot sample collected from the material at the bottom of the tank, container, or line at its lowest point.

##### Discussion

3.1.3.1 *Discussion*—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.

~~3.1.1.4~~ **3.1.4 bottom water sample**—a spot sample of free water taken from beneath the petroleum contained in a ship or barge compartment or a storage tank.

##### 3.1.1.5 *clearance sample*

**clearance sample**—a spot sample taken with the inlet opening of the sampling apparatus 10 cm (4 in.) (some regulatory agencies require 15 cm (6 in.)) below the bottom of the tank outlet. **Discussion**

3.1.5.1 *Discussion*—This term is normally associated with small (159 m<sup>3</sup> or 1000 Bbls or less) tanks, commonly referred to as lease tanks.

##### 3.1.1.6 *composite sample*

**composite sample**—a blend of spot samples mixed in proportion to the volumes of material from which the spot samples were obtained.

##### 3.1.1.7 *core sample*

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

##### 3.1.1.8 *dipper sample*

**dipper sample**—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.

<sup>3</sup> Withdrawn.

<sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

<sup>4</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

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

3.1.1.9 *drain sample*

3.1.9 *drain sample*—a sample obtained from the water draw-off valve on a storage tank. **Discussion**

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).

3.1.1.10 *floating roof sample*—a spot sample taken just below the surface to determine the density of the liquid on which the roof is floating.

3.1.1.11 *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.

3.1.1.12 *grab sample*

3.1.12 *grab sample*—a sample obtained by collecting equal quantities from parts or packages of a shipment of loose solids such that the sample is representative of the entire shipment.

3.1.1.13 *grease sample*

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

3.1.1.14 *lower sample*

3.1.14 *lower sample*—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. 1.

3.1.1.15 *middle sample*

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

3.1.1.16 *multiple tank composite sample*—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. **Discussion**

3.1.16.1 **Discussion**—The mixture is blended in proportion to the volume of material contained in the respective tanks or compartments.

3.1.1.17 *outlet sample*

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.

3.1.1.18 *representative sample*

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

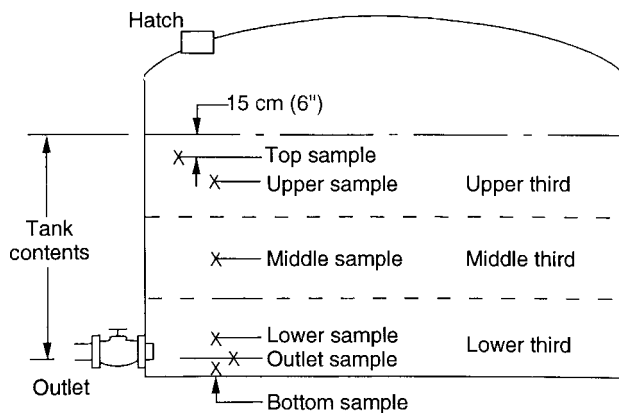
3.1.1.19 *running sample*

3.1.19 *running sample*—a sample obtained by lowering a breaker or bottle to the level of the bottom of the outlet connection or swing line and returning it to the top of the oil at a uniform rate such that the beaker or bottle is about three-fourths full when withdrawn from the oil.

3.1.1.20 *sample*

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.

3.1.1.21 *sampling*—all the steps required to obtain a sample that is representative of the contents of any pipe, tank, or



NOTE 1—The location shown for the outlet sample applies only to tanks with side outlets. It does not apply when the outlet comes from the floor of the tank or turns down into a sump. Bottom sample location must be specified.

NOTE 2—Samples should be obtained from within solid stand pipes as the materials normally not representative of the material in the tank at that point.

FIG. 1 Spot Sampling Locations

other vessel and to place that sample in a container from which a representative test specimen can be taken for analysis.

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

**3.1.1.23** *surface sample*

**3.1.23** *surface sample*—a spot sample skimmed from the surface of a liquid in a tank.

**3.1.1.24** *tank composite sample*—a blend created from the upper, middle, and lower samples from a single tank.

**Discussion**

**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 2**.

**3.1.1.25** *tap sample*

**3.1.25** *tap sample*—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.

**3.1.1.26** *top sample*

**3.1.26** *top sample*—a spot sample obtained 15 cm (6 in.) below the top surface of the liquid. See Fig. 1.

**3.1.1.27** *tube or thief sample*—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.

**3.1.1.28** *upper sample*

**3.1.28** *upper sample*—a spot sample taken from the middle of the upper one-third of the tank’s contents (a distance of one-sixth of the liquid depth below the liquid’s surface). See Fig. 1.

**3.1.2** *Other Terms:*

**3.1.2.1** *automatic sampler*

**Other Terms**

**3.1.29** *automatic sampler*—a device used to extract a representative sample from the liquid flowing in a pipe. **Discussion**

**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).

**3.1.2.2** *dissolved water* Chapter 8.2).

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

**3.1.2.3** *emulsion*

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

**3.1.2.4** *entrained water*

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

**3.1.32.1 Discussion**—Entrained water includes emulsions but does not include dissolved water.

**3.1.2.5** *free water*

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

**3.1.2.6** *intermediate container*

**3.1.34** *intermediate container*—the vessel into which all or part of the sample from a primary container/receiver is transferred for transport, storage, or ease of handling.

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

**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.

**3.1.2.8** *stand pipes*

**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. **Discussion**

**TABLE 2 1 Sampling Instructions for Horizontal Cylindrical Tanks**

| 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    |

3.1.36.1 *Discussion*—Stand pipes may also be found on ships and barges.

3.1.2.9 *test specimen*

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. Chapter 8.3 can provide additional guidance. A summary of the manual sampling procedures and their applications is presented in Table 2.

#### 5. Significance and Use

5.1 Representative samples of petroleum and petroleum products are required for the determination of chemical and physical properties, which are used to establish standard volumes, prices, and compliance with commercial and regulatory specifications.

5.2 The following concepts must be considered when selecting a specific sampling procedure.

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.

(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)). Chapter 8.2).

#### 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

**TABLE 1-2 Typical Sampling Procedures and Applicability**

| 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    |
| Liquids of 101 kPa (14.7 psia) RVP or less                                | storage tanks with taps  | thief 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   | tap sampling       |
| Liquids of 13.8 kPa (2 psia) RVP or less                                  | storage tanks, ships, barges   | pipeline sampling  |
| Liquids of 13.8 kPa (2 psia) RVP or less                                  | free or open-discharge streams   | bottle sampling    |
| Liquids of 13.8 kPa (2 psia) RVP or less                                  | drums, barrels, cans   | dipper sampling    |
| Bottom or thief sampling of liquids of 13.8 kPa (2 psia) RVP or less      | tank cars, storage tanks   | tube 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 | thief sampling     |
| Crude petroleum   | storage tanks, ship and barge, tanks, tank cars, tank trucks, pipelines                              | dipper sampling    |
| Industrial aromatic hydrocarbons  | storage tanks, ship and barge tanks  | automatic sampling |
| Waxes, solids bitumens, other soft solids                                 | barrels, cases, bags, cakes  | thief sampling     |
| Petroleum coke; lumpy solids  | freight cars, conveyors, bags, barrels, boxes  | bottle sampling    |
| Greases, soft waxes, asphalts   | kettles, drums, cans, tubes  | tap sampling       |
| Asphaltic materials   | storage tanks, tank cars, lines, packages  | bottle sampling    |
| Emulsified asphalts   | storage tanks, tank cars, lines, packages  | boring sampling    |
|   |  | grab sampling      |
|   |  | grease sampling    |
|   |  | ...                |
|   |  | ...                |

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.

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.

6.2.4 There should be an inspection cover/closure of sufficient size to facilitate filling, inspection, and cleaning.

6.2.5 The container should be designed to allow the preparation of a homogeneous mixture of the sample while preventing the loss of any constituents which affect the representativeness of the sample and the accuracy of the analytical tests.

6.2.6 The container should be designed to allow the transfer of samples from the container to the analytical apparatus while maintaining their representative nature.

6.3 *Bottles (Glass)*—Clear glass bottles may be examined visually for cleanliness and allows visual inspection of the sample for free water cloudiness, and solid impurities. Brown glass bottles afford some protection to the samples when light may affect the test results.

6.4 *Bottles (Plastic)*—Plastic bottles made of suitable material may be used for the handling and storage of gas oil, diesel oil, fuel oil, and lubricating oil. Bottles of this type should not be used for gasoline, aviation jet fuel, kerosine, crude oil, white spirit, medicinal white oil, and special boiling point products unless testing indicates there is no problem with solubility, contamination, or loss of light components.

6.4.1 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.

6.4.2 Plastic bottles have an advantage in that they will not shatter like glass or corrode like metal containers.

6.5 *Cans*—When cans are to be used, they must have seams that 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. 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. Internal epoxy lined cans may have residual contamination and precautions should be taken to ensure its removal. Practice D4306 should be used when taking samples for aviation fuels.

6.6 *Container Closures*—Cork stoppers, or screw caps of plastic or metal may be used for glass bottles. Corks must be of good quality, clean, and free from holes and loose bits of cork. Never use rubber stoppers. Prevent the sample from contacting the cork by wrapping tin or aluminum foil around the cork before forcing it into the bottle. Screw caps providing a vapor tight closure seal shall be used for cans. Screw caps must be protected by a disk faced with material that will not deteriorate and contaminate the sample. Containers used to take samples that will be tested for density or gravity shall have screw caps.

6.7 *Cleaning Procedure*—Sample containers must be clean and free from all substances which might contaminate the material being sampled (such as water, dirt, lint, washing compounds, naphtha and other solvents, soldering fluxes, acids, rust, and oil). Prior to further use, reusable containers such as cans and bottles should be rinsed with a suitable solvent. Use of sludge solvents to remove all traces of sediments and sludge may be necessary. Following the solvent wash, the container should be washed with a strong soap solution, rinsed thoroughly with tap water, and given a final rinse using distilled water. Dry the container either by passing a current of clean warm air through the container or by placing it in a hot dust-free cabinet at 40°C (104°F) or higher. When dry, stopper or cap the container immediately. Normally, it is not necessary to wash new containers.

6.7.1 Depending on service, receivers used in conjunction with automatic samplers may need to be washed with solvent between uses. In most applications, it is not desirable or practical to wash these receivers using soap and water as outlined above for cans and bottles. The cleanliness and integrity of all sample containers/receivers must be verified prior to use.

6.7.2 When sampling aviation fuel, Practice D4306 should be consulted for recommended cleaning procedures for containers that are to be used in tests for the determination of water separation, copper corrosion, electrical conductivity, thermal stability, lubricity, and trace metal content.

6.8 *Sample Mixing Systems*—The sample container should be compatible with the mixing system for remixing samples that have stratified to ensure that a representative sample is available for transfer to an intermediate container or the analytical apparatus. This is especially critical when remixing crude, some black products, and condensates for sediment and water analysis to ensure a representative sample. The requirements governing the amount of mixing and type of mixing apparatus differ depending upon the petroleum or petroleum product and the analytical test to be performed. Refer to Practice D5854 (API *MPMS* Chapter 8.3) for more detailed information.

6.8.1 When stratification is not a major concern, adequate mixing may be obtained by such methods as shaking (manual or mechanical), or use of a shear mixer.

6.8.2 Manual and mechanical shaking of the sample container are not recommended methods for mixing a sample for sediment

and water (S&W) analysis. Tests have shown it is difficult to impart sufficient mixing energy to mix and maintain a homogeneous representative sample. Practice D5854 (API MPMS Chapter 8.3) contains more detailed information.

6.9 *Other Equipment*—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.

6.10 *Sampling Devices*—Sampling devices are described in detail under each of the specific sampling procedures. Sampling devices shall be clean, dry, and free of all substances that might contaminate the material being sampled.

## 7. Manual Sampling Considerations

7.1 The following factors must be considered in the development and application of manual sampling procedures:

7.1.1 *Physical and Chemical Property Tests*—The physical and chemical property tests to be performed on a sample will dictate the sampling procedures, the sample quantity required, and many of the sample handling requirements.

7.1.2 *Sampling Sequence*:

7.1.2.1 Any disturbance of the material in a tank that is to be sampled may adversely affect the representative character of the sample(s). Therefore, the sampling operation should be conducted before innage gaging, the associated temperature determination, and any other similar activity that could disturb the tank contents.

7.1.2.2 To avoid contamination of the oil column during the sampling operation, the order of precedence for sampling should start from the top and work downward, according to the following sampling sequence: surface, top, upper, middle, lower, outlet, clearance, all-levels, bottom, and running sample.

7.1.3 *Equipment Cleanliness*—The sampling equipment should be clean prior to commencing the sampling operation. Any residual material left in a sampling device or sample container from a previous sample or cleaning operation may destroy the representative character of the sample. It is good practice with light petroleum products to rinse the container with the product to be sampled prior to drawing samples.

7.1.4 *Compositing of Individual Samples*:

7.1.4.1 If the sampling procedure requires that several different samples be obtained, physical property tests may be performed on each sample or on a composite of the various samples. When the respective tests are performed on individual samples, which is the recommended procedure, the test results are averaged generally.

7.1.4.2 When a multiple tank composite sample is required, such as on board ships and barges, a composite tank sample may be prepared from the samples from different tanks when they contain the same material. In order for such a composite tank sample to be representative of the material contained in the various tanks, the quantity from the individual samples used to prepare the composite tank sample must be proportional to the volumes in the corresponding tanks. In most other compositing situations, equal volumes from the individual samples must be used. The method of compositing should be documented and care taken to preserve the integrity of the samples. It is recommended that a portion of each tank sample be retained separately (not composited) for retesting if necessary.

7.1.4.3 When compositing samples, exercise care to ensure sample integrity. Refer to Practice D5854 (API MPMS Chapter 8.3) for guidance on mixing and handling of samples.

7.1.4.4 Samples taken at specific levels, for example, upper-middle-lower capping will require a small portion of the sample to be poured out to create an ullage in the container before capping. All other samples shall be capped immediately and taken to the laboratory.

7.1.5 *Sample Transfers*—The number of intermediate transfers from one container to another between the actual sampling operation and testing should be minimized. The loss of light hydrocarbons as the result of splashing, loss of water due to clingage, or contamination from external sources, or both, may distort test results, for example, density, sediment and water, product clarity. The more transfers between containers, the greater the likelihood one or both of these problems may occur. See Practice D5854 (API MPMS Chapter 8.3) for additional information concerning the handling and mixing of samples.

7.1.6 *Sample Storage*—Except when being transferred, samples should be maintained in a closed container in order to prevent loss of light components. Samples should be protected during storage to prevent weathering or degradation from light, heat, or other potential detrimental conditions.

7.1.7 *Sample Handling*—If a sample is not uniform (homogeneous) and a portion of the sample must be transferred to another container or test vessel, the sample must be thoroughly mixed in accordance with the type of material and appropriate test method, in order to ensure the portion transferred is representative. Exercise care to ensure mixing does not alter the components within the sample, for example, loss of light ends. See Practice D5854 (API MPMS Chapter 8.3) for more detailed instructions.

## 8. Special Precautions

8.1 This practice does not purport to cover all safety aspects associated with sampling. However, it is presumed that the personnel performing sampling operations are adequately trained with regard to the safe application of the procedures contained herein for the specific sampling situation.

8.2 A degree of caution is required during all sampling operations, but in particular when sampling certain products. Crude oil may contain varying amounts of hydrogen sulfide (sour crude), an extremely toxic gas. Annex A1 provides precautionary statements that are applicable to the sampling and handling of many of these materials.

8.3 When taking samples from tanks suspected of containing flammable atmospheres, precautions should be taken to guard

against ignitions from static electricity. Conductive objects, such as gage tapes, sample containers, and thermometers, should not be lowered into or suspended in a compartment or tank that is being filled, or immediately after cessation of pumping. Conductive material such as gage tape should always be in contact with gage tube until immersed in the fluid. A waiting period (normally 30 min or more after filling cessation) will generally be required to permit dissipation of the electrostatic charge. In order to reduce the potential for static charge, nylon or polyester rope, cords, or clothing should not be used. Refer to Test Method D4865.

## 9. Special Instructions for Specific Materials

### 9.1 *Crude Petroleum and Residual Fuel Oils:*

9.1.1 Crude petroleum and residual fuel oils usually are nonhomogeneous. Tank samples of crude oil and residual oils may not be representative for the following reasons:

9.1.1.1 The concentration of entrained water is 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.

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

9.1.1.3 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.

9.1.2 Automatic sampling in accordance with Practice D4177 (API MPMS Chapter 8.2) is recommended whenever samples of these materials are required for custody transfer measurements. However, tank samples may be used when agreed to by all parties to the transaction.

9.2 *Gasoline and Distillate Products*— Gasoline and distillate products are usually homogeneous, but they are often shipped from tanks that have clearly separated water on the bottom. Tank sampling, in accordance with the procedures outlined in Section 13, is acceptable under the conditions covered in 5.2.2.

9.3 *Industrial Aromatic Hydrocarbons*— For samples of industrial aromatic hydrocarbons (benzene, toluene, xylene, and solvent naphthas), proceed in accordance with 5.2.1, Sections 6 and 10, 12.2-12.5, and Section 13, with particular emphasis on the procedures pertaining to the precautions for care and cleanliness. See Annex A1 for details.

### 9.4 *Lacquer Solvents and Diluents:*

9.4.1 When sampling bulk shipments of lacquer solvents and diluents which are to be tested using Guide D268, observe the precautions and instructions described in 9.4.2 and 9.4.3.

9.4.2 *Tanks and Tank Cars*—Obtain upper and lower samples (see Fig. 1) of not more than 1 L (qt) each by the thief or bottle spot sampling procedures outlined in 13.4.2. In the laboratory, prepare a composite sample of not less than 2 L/2 qt by mixing equal parts of the upper and lower samples.

9.4.3 *Barrels, Drums, and Cans*—Obtain samples from the number of containers per shipment as mutually agreed. In the case of expensive solvents, which are purchased in small quantities, it is recommended that each container be sampled. Withdraw a portion from the center of each container to be sampled using the tube sampling procedure (see 9.4.3) or bottle sampling procedure (see 13.4.2, although a smaller bottle may be used). Prepare a composite sample of at least 1 L (1 qt) by mixing equal portions of not less than 500 mL (1 pt) from each container sampled.

9.5 *Asphaltic Materials*—When sampling asphaltic materials that are to be tested using Test Method D1856 or Test Method D2172, obtain samples by the boring procedure in Section 17 or the grab procedure in Section 18. A sample of sufficient size to yield at least 100 g (¼ lb) of recovered bitumen is required. About 1000 g (2 lb) of sheet asphalt mixtures usually will be sufficient. If the largest lumps in the sample are 2.5 cm (1 in.), 2000 g (4 lb) will usually be required, and still larger samples if the mixture contains larger aggregates.

9.6 *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 the bottle sampling procedure outlined in 13.4.2 using a bottle that has a 4-cm (1½-in.) diameter or larger mouth. Refer to Fig. 1 and Table 2-Table 1 for sampling locations. Use the dipper procedure in Section 15 to obtain samples for fill or discharge lines. Sample packages in accordance with Table 3. If the material is solid or semisolid, use the boring sampling procedure described in Section 17. Obtain at least 4 L (1 gal) or 4.5 kg (10 lb) from each lot or shipment. Store the samples in clean, airtight containers at a temperature of not less than 4°C (40°F) until the test. Use a glass or black iron container for emulsified asphalts of the RS-1 type.

**TABLE 3 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                     | 6850 and greater | 20                     |



## 10. Special Instructions for Specific Tests

10.1 *General*—Special sampling precautions and instructions are required for some ASTM test methods and specifications. Such instructions supplement the general procedures of this practice and supersede them if there is a conflict.

10.2 *Distillation of Petroleum Products*—When obtaining samples of natural gasoline that are to be tested using Test Method D86, the bottle sampling procedure described in 13.4.2 is the preferred technique, with the exception that pre-cooled bottles and laboratory compositing is required. Before obtaining the sample, pre-cool the bottle by immersing it in the product, allowing it to fill, and discard the first filling. If the bottle procedure cannot be used, obtain the sample by the tap procedure and with the use of the cooling bath, as described in 13.6. Do not agitate the bottle while drawing the sample. After obtaining the 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).

10.3 *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). Chapter 8.4).

10.4 *Oxidation Stability*:

10.4.1 When sampling products that are to be tested for oxidation stability in accordance with Test Method D525, Test Method D873, or equivalent methods, observe the precautions and instructions that follow.

10.4.2 *Precautions*—Very small amounts (as low as 0.001 %) of some materials, such as inhibitors, have a considerable effect upon oxidation stability tests. Avoid contamination and exposure to light while taking and handling samples. To prevent undue agitation with air, which 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.

10.4.3 *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. Clean the bottles by the procedure described in 6.7. Rinse thoroughly with distilled water, dry, and protect the bottles from dust and dirt.

10.4.4 *Sampling*—A running sample obtained by the procedure in 13.5 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.

## 11. Special Instructions for Specific Applications

11.1 *Marine Cargoes of Crude Oils*:

11.1.1 Samples of ship or barge cargoes of crude petroleum may be taken by mutual agreement by the following methods:

11.1.1.1 From the shore tanks before loading and both before and after discharging as in Section 13.

11.1.1.2 From the pipeline during discharging or loading. Pipeline samples may be taken either manually or with an automatic sampler. If the pipeline requires displacement or flushing, exercise care that the pipeline sample includes the entire cargo and none of the displacement. Separate samples may be required to cover the effect of the line displacement on the prior or following transfer.

11.1.1.3 From the ship's or barge's tanks after loading or before discharging. An all-levels sample, running sample, upper-middle-lower sample, or spot samples at agreed levels may be used for sampling each cargo compartment of a ship or barge.

11.1.2 Ship and barge samples may be taken either through open hatches or by use of equipment designed for closed systems.

11.1.3 Normally, when loading a marine vessel, the shore tank sample or the pipeline sample taken from the loading line is the custody transfer sample. However, ship's/barge's tank samples may also be tested for sediment and water (S&W) and for other quality aspects, when required. The results of these ship's/barge's tank sample tests, together with the shore tank sample tests, may be shown on the cargo certificate.

11.1.4 When discharging a ship/barge, the pipeline sample taken from a properly designed and operated automatic line sampler, in the discharge line, should be the custody transfer sample. Where no proper line sample is available, the ship's/barge's tank sample can be the custody transfer sample except where specifically exempted.

11.1.5 Samples of ship/barge cargoes of finished products are taken from both shipping and receiving tanks and from the pipeline, if required. In addition, the product in each of the ship/barge tanks should be sampled after the vessel is loaded or just before unloading.

NOTE 4—Refer to MPMS Chapter 17 for additional requirements associated with sampling materials in marine vessels.

11.2 *Crude Oil Gathered By Truck*—Refer to MPMS Chapter 18.1 for additional sampling requirements when gathering crude oil by tank truck.

11.3 *Tank Cars*—Sample the material after the car has been loaded or just before unloading.

11.4 *Package Lots (Cans, Drums, Barrels, or Boxes)*—Take samples from a sufficient number of the individual packages to prepare a composite sample that will be representative of the entire lot or shipment. Select at random the individual packages to be sampled. The number of random packages will depend on several practical considerations, such as (1) the tightness of the product specifications; (2) the 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. In most cases, the number specified in Table 3 will be satisfactory.

## 12. Sampling Procedures (General)

12.1 The standard sample procedures described in this practice are summarized in ~~Table 1~~Table 2. Alternative sampling procedures may be used if a mutually satisfactory agreement has been reached by the parties involved. It is recommended that such agreements be put in writing and signed by authorized officials.

### 12.2 Precautions:

12.2.1 Extreme care and good judgment are necessary to ensure that samples are obtained that represent the general characteristics and average condition of the material. Clean hands are important.

12.2.2 Since many petroleum vapors are toxic and flammable, avoid breathing them, igniting them from an open flame, burning embers, or a spark produced by static electricity. All safety precautions specific to the material being sampled should be followed.

12.2.3 When sampling relatively volatile products more than 13.8 kPa (2 psia) RVP, the sampling apparatus shall be filled and allowed to drain before drawing the sample. If the sample is to be transferred to another container, this container shall also be rinsed with some of the volatile product and then drained. When the actual sample is emptied into this container, the sampling apparatus should be upended into the opening of the sample container and should remain in this position until the contents have been transferred so that no unsaturated air will be entrained in the transfer of the sample.

12.2.4 When sampling nonvolatile liquid products, 13.8 kPa (2 psia) RVP or less, sampling apparatus shall be filled and allowed to drain before drawing the actual sample. If the actual sample is to be transferred to another container, the sample container shall be rinsed with some of the product to be sampled and drained before it is filled with the actual sample.

12.2.5 The transfer of crude oil samples from the sample apparatus/receiver to the laboratory glassware in which they will be analyzed requires special care to maintain their representative nature. The number of transfers should be minimized. Mechanical means of mixing and transferring the samples in the sample receiver are recommended.

### 12.3 Sample Handling:

12.3.1 *Volatile Samples*—All volatile samples of petroleum and petroleum products shall be protected from evaporation. Transfer the product from the sampling apparatus to the sample container immediately. Keep the container closed except when the material is being transferred. After delivery to the laboratory, volatile samples should be cooled before the containers are opened.

12.3.2 *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, tetraethyl lead and inhibitor contents, sludge forming characteristics, stability tests, or neutralization value. Brown glass bottles may be used. Wrap or cover clear glass bottles immediately.

12.3.3 *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.

12.3.4 *Container Outage*—Never fill a sample container completely. Allow adequate room for expansion, taking into consideration the temperature of the liquid at the time of filling, and the probable maximum temperature to which the filled container may be subjected. Adequate sample mixing is difficult if the container is more than 80 % full.

12.4 *Sample Labeling*—Label the container immediately after a sample is obtained. Use waterproof and oil proof ink or a pencil hard enough to dent the tag. Soft pencil and ordinary ink markers are subject to obliteration from moisture, oil smearing, and handling. Include the following information on the label:

12.4.1 Date and time (the period elapsed during continuous sampling and the hour and minute of collection for dipper samples),

12.4.2 Name of the sampler,

12.4.3 Name and number and owner of the vessel, car, or container,

12.4.4 Grade of material, and

12.4.5 Reference symbol or identification number.

12.5 *Sample Shipment*—To prevent loss of liquid and vapors during shipment and to protect against moisture and dust, cover the stoppers of glass bottles with plastic caps that have been swelled in water, wiped dry, placed over the tops of the stoppered bottles, and allowed to shrink tightly in place. Before filling metal containers, inspect the lips and caps for dents, out-of-roundness, or other imperfections. Correct or discard the cap or container, or both. After filling, screw the cap tightly and check for leaks. Appropriate governmental and carrier regulations applying to the shipment of flammable liquids must be observed.

## 13. Tank Sampling

13.1 Samples should not be obtained from within solid stand pipes as the material is normally not representative of the material in the tank at that point. Stand pipe samples should only be taken from pipes with at least two rows of overlapping slots. See Fig. 2.

13.2 When sampling crude oil tanks with diameters in excess of 45 m (150 ft), additional samples should be taken from any other available gaging hatches located around the perimeter of the tank roof, safety requirements permitting. All the samples should be individually analyzed using the same test method and the results should then be averaged arithmetically.

13.3 *Composite Sample Preparation*—A composite spot sample is a blend of spot samples mixed volumetrically proportional for testing. Some tests may also be made on the spot samples before blending and the results averaged. Spot samples from crude oil tanks are collected in the following ways:

13.3.1 *Three-way*—On tanks larger than 159 m<sup>3</sup> (1000 bbls) capacity, which contain in excess of 4.5 m (15 ft) of oil, equal