



Designation: D4296 – 83(Reapproved 2009)

Standard Practice for Sampling Pitch¹

This standard is issued under the fixed designation D4296; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers procedures for sampling pitch at points of manufacture, storage, and delivery.

1.2 The values stated in SI units are to be regarded as standard. The values in parentheses are for information only.

1.3 *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.* Specific warning statements are given in Section 11.

2. Terminology

2.1 Definitions:

2.1.1 *bulk sample*—a large sample, either from one place or made up of several incremental samples of the same material.

2.1.2 *composite sample*—a thoroughly mixed gross sample.

2.1.3 *gross sample*—a large sample made up of several portions (increments) of a mass of material.

2.1.4 *increment*—a portion of a material to be combined with other portions of the same material to provide a larger sample which will represent the whole material.

2.1.5 *representative sample*—a part of a homogeneous material, or a part of the composited and mixed portions of a material, which carries all the true properties and physical characteristics of the whole material.

2.1.6 *sample*—a part taken as representative of a whole material.

3. Summary of Practice

3.1 Samples of liquid pitches are taken from process lines, storage, and shipment containers in increments required to prepare a representative sample for testing.

3.2 Samples of solid pitches are taken from loading systems, storage, and shipment containers in increments required to prepare a representative sample for testing.

¹ This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke and Carbon Material.

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4. Significance and Use

4.1 Sampling is as important as testing. If the sampling is improperly done, the sample of the material will be in error, and the analysis will not represent the true properties of the material, and it may be impossible or impracticable to take another sample; whereas, if the sample is properly taken and the analysis is in error, another analysis can readily be made of the original sample.

4.2 Samples may be taken for either of the following two purposes:

4.2.1 To represent as nearly as possible an average of the bulk of the materials sampled, or

4.2.2 To ascertain the maximum variation in characteristics which the material possesses.

4.3 A good sampling plan has the following characteristics:

4.3.1 It protects the consumer against the acceptance of a bad batch of material.

4.3.2 It protects the producer against the rejection of a good batch of material.

4.3.3 It gives long-range protection to the consumer.

4.3.4 It encourages the producer to keep his process in control.

4.3.5 It minimizes the cost of sampling, inspection of testing, and administration.

4.3.6 It provides information concerning the quality of the product.

4.4 *Sampling Integrity*—When one takes a sample, one is attempting to represent a batch or lot of material with that sample. Different forms of pitch, and pitch in different types of containers, need to have different sampling plans and appropriate sampling equipment. Each sampling plan should be designed so that it accomplishes its objective, which is to ascertain the characteristics or quality of a batch or lot of material.

5. Selection of Samples

5.1 Pitches shall be sampled by the producer at the point of manufacture or storage, and at such time as to allow the tests controlling acceptance or rejection of quality, as specified by the consumer, to be made in advance of a shipment.

5.2 Samples of pitches shall be taken by the consumer from the shipment containers immediately upon delivery and receipt

and tests controlling acceptance or rejection of quality, as described by the consumer's specifications, shall be made as soon as possible.

6. Size of Samples

- 6.1 The sample size of liquid materials shall be as follows:
 - 6.1.1 From process lines, 1 L (1 qt).
 - 6.1.2 From bulk storage, 1 L (1 qt) or 1-L aliquot of composite sample.
 - 6.1.3 From bulk shipment containers, 1 L (1 qt) or 1-L aliquot of composite sample.
 - 6.1.4 From barrels or drums, 0.5 L (1 pt) or 0.5-L aliquot of composite sample.
- 6.2 The sample size of solid materials shall be as follows:
 - 6.2.1 From storage area, 4 L (1 gal) or 4-L aliquot of composite sample.
 - 6.2.2 From bulk shipment containers, 4 L (1 gal) aliquot of composite sample.
 - 6.2.3 From barrels or drums, 0.5-L (1 pt) or 0.5-L aliquot of composite sample.

7. Collection of Gross Sample

- 7.1 A collection of gross sample represents a lot or batch of material and is composed of a number of increments.
 - 7.1.1 Collect the increments regularly and systematically, so that the entire quantity of pitch sampled will be represented proportionately in the gross sample, and with such frequency that a gross sample of the required amount will be collected.

8. Size of Increments

8.1 To collect samples, use a shovel or specially designed tool or mechanical means for taking equal portions or increments. When samples are collected from the surface of loaded shipments, the gross sample shall consist of nine increments of approximately equal quantity. When sampling from piles, conveyer belts, and so forth, the gross sample shall consist of not less than 25 nor more than 50 increments of approximately equal quantity.

9. Protection and Preservation of Samples

- 9.1 Sample containers shall be new or reusable containers which can be thoroughly cleaned with a solvent and wiped dry with a clean dry cloth.
- 9.2 Care shall be taken to prevent the sample from becoming contaminated. Immediately after filling, hot liquid sample containers shall be positively covered, but not tightly sealed. Containers of solid materials shall be tightly and positively sealed.
- 9.3 While sampling during inclement weather, attention shall be paid to prevent water from dropping into the hot liquid sample or samples of solid materials.
- 9.4 Immediately after filling and sealing, the sampling containers shall be properly marked for identification with a suitable pencil on the container itself, not on the lid.

10. Apparatus

10.1 *Sampling Equipment for Liquid Materials:*

10.1.1 If so equipped, pipe lines and storage tanks may be sampled from valves installed in the lines or up the side of the tanks. A typical tank valve is shown in Fig. 1, and suitable line valves are illustrated in Fig. 2 and Fig. 3.

10.1.2 For sampling tank trucks, a pipe connection with attached sample valve similar to the one shown in Fig. 2 may be inserted between the outlet pipe and unloading line.

10.1.3 A weighted sample bottle similar to those shown in Fig. 4 and Fig. 5, or a "thief sampler" as shown in Fig. 6, or a "dip sampler" similar to that shown in Fig. 7, may be used to sample production and storage tanks, tank cars, and tank trucks.

10.1.4 A "tube" or "thief" sampler may be used to sample materials in drums or barrels.

10.2 Sampling Equipment for Solid Materials:

10.2.1 Samples from loading systems may be taken with a shovel, metal or plastic scoop, or metal can.

10.2.2 A shovel may be used to sample storage bins, pitch piles, pitch bays, hopper cars, and dump trucks.

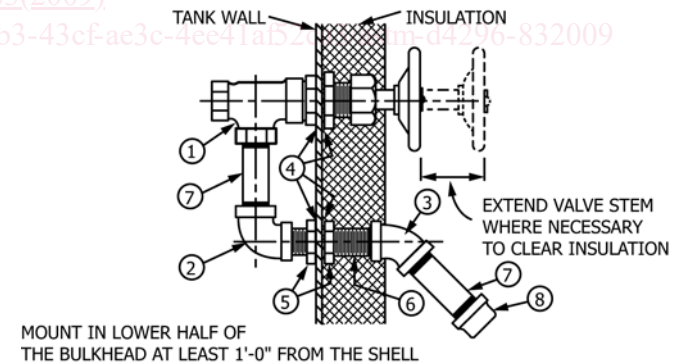
10.2.3 A sampling spear as shown in Fig. 8 may be used to sample materials in drums, barrels, bags, and silos.

10.3 Sampling Containers:

10.3.1 Containers for liquid materials shall be wide-mouth cans with line screw caps or triple-seal friction-top cans.

10.3.2 Containers for formed pitches or crushed solid pitch shall be triple-seal friction-top cans, sealable plastic or paper cartons with inert inside coating, plastic sacks placed in other containers suitable for handling, or substantial strength paper bag which can be stapled closed.

10.4 Mixing Equipment for Solid Materials:



REF. NO.	DESCRIPTION	NO. REQ.
1	¾" "VOGT" P-9844 STEEL ANGLE VALVE OR SIMILAR, PANEL MOUNTED	1
2	¾" STEEL OR MALL. IRON 90° ELBOW	1
3	¾" STEEL OR MALL. IRON 45° ELBOW	1
4	ASBESTOS GASKETS SNUG ON THREAD OR WOUND WITH YARN	4
5	¾" 150# SCREWED M. I. LOCKNUT	2
6	¾" × 3½" ± PARALLEL THREADED STEEL PIPE NIPPLE (CUT FROM ¾" STD. TANK NIPPLE IF OTHERWISE UNOBTAINABLE)	1
7	¾" × 3" THREADED STEEL PIPE NIPPLE	2
8	¾" MALL. IRON PIPE CAP	1

FIG. 1 Typical Submerged Sampling Device



FIG. 2 In-Line Sampling Device

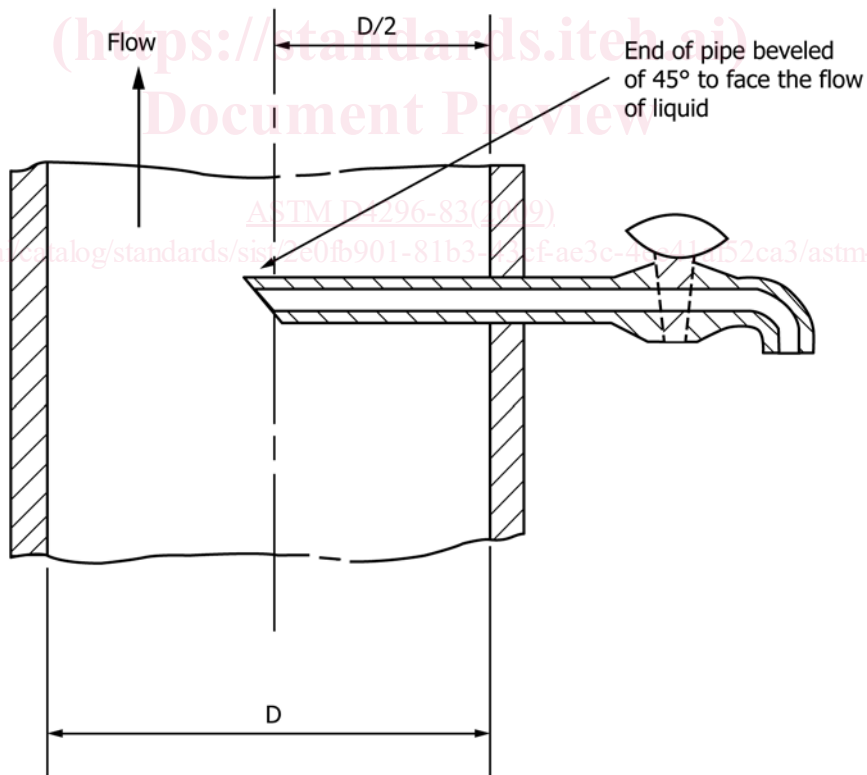
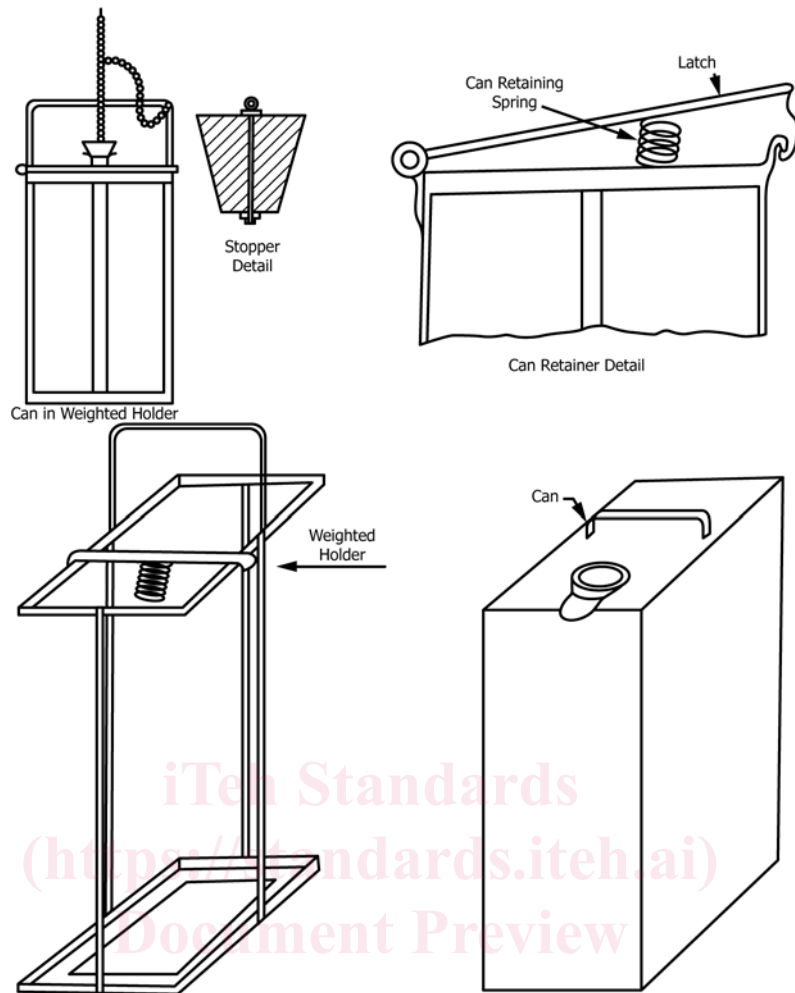


FIG. 3 Pipeline Sampler

10.4.1 Section of clean canvas or plastic sheet of sufficient size to contain a large bulk sample which is to be reduced by mixing and quartering.

10.4.2 A “riffle” as shown in Fig. 9 may be used for reducing gross samples to smaller sizes for testing.



NOTE 1—This type sampler is lowered into the tank with the stopper in place. When the desired depth is reached, the stopper is removed by means of the attached wire, cord, or chain and the container allowed to fill. Complete filling is indicated by the cessation of bubbles of air from the can at the surface of the liquid. The sampler is then withdrawn from the tank and the contents poured into the clean sampler container. A clean can (bottle) must be used for each sample taken.

FIG. 5 Throw-Away Container Sampler

bottom. A recommended design of the sample valve is shown in Fig. 1. Draw a minimum of 4 L (1 gal) of the product from each sample valve for test or composite.

12.1.2 *Thief Sampling Method*—Take samples at the top, middle and lower sections of the tank by lowering a thief sampler into the material. A satisfactory type with instructions for use is shown in Fig. 6. The samples may be tested separately or combined and mixed thoroughly for a composite analysis.

12.1.3 *Weighted Sample Bottle Method*—Take samples at the top, middle, and lower sections of the tank by lowering the bottle into the material. Two types of weighted sampling bottles are illustrated in Fig. 4 and Fig. 5.

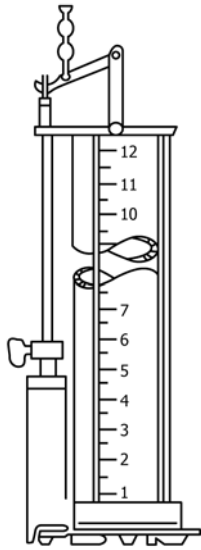
12.1.3.1 The essential feature of the method is to drop the stoppered bottle into the material to the desired depth, and then pull the stopper by means of an attached chain. When the material fills the bottle, lift out of the liquid and empty into a clean container. Drain the bottle as completely as possible, re-insert the stopper, lower into the material to the next sampling depth.

12.1.3.2 Normally, there is not enough undrained residue from the first sample to affect the properties of the next sample taken from a different level. However, if substantial material difference is suspected at various levels of the material, discard the first of successive samples to rinse the bottle and then return the bottle to the same level for another sample.

12.1.3.3 The three samples may be tested separately for consistency to detect stratification or they may be combined, thoroughly mixed, and a 1 to 4-L (¼ to 1-gal) aliquot of the composite taken for determining the average characteristics of the material.

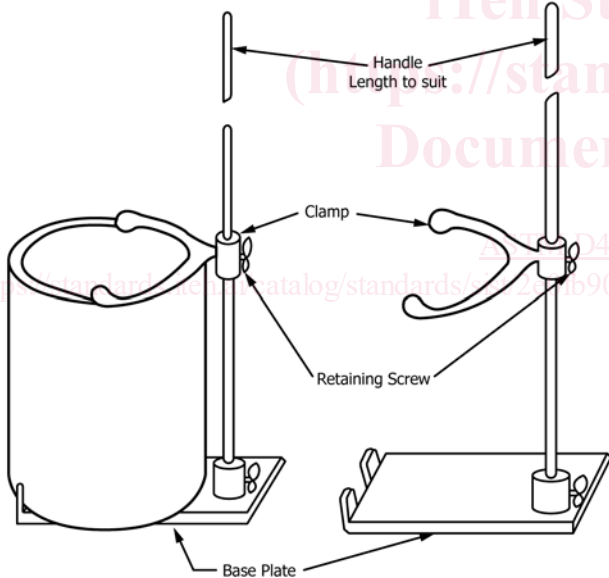
12.2 *Sampling Bulk Storage Tanks Equipped with Mechanical Agitators or Recirculating Pumps*—When the tank is equipped with operating mechanical agitators or recirculating pumping systems which are performing adequate mixing of the tank contents, take a single sample by any of the methods described in 12.1.1, 12.1.2, or 12.1.3 to use for test purposes.

12.3 *Sampling Tank Cars and Tank Trucks:*



NOTE 1—This type sampler is lowered into the tank with bottom valve open (there is no top closure). When the desired depth is reached, the lowering chain is given a snap tug which closes the bottom valve. The sampler is then withdrawn from the tank and the contents transferred to the sample container. This sampler may be used for repetitive sampling in the same tank.

FIG. 6 Thief Sampler



NOTE 1—Sampler with can secured in place is rapidly lowered into tank to desired depth and allowed to fill with sample. The sampler is then withdrawn from the tank and the contents transferred to the sample container. A clean container *must* be used to take each sample.

FIG. 7 Dip Sampler

12.3.1 Samples of hot liquid materials may be taken by the dip method using a clean wide-mouth or friction-top can in a suitable holder or firmly attached to a metal rod. One such dip apparatus is shown in Fig. 7. Quickly lower the dip sample about 0.3 m (1 ft) below the surface of the liquid and carefully remove. Use a clean container to take each sample, and then transfer the material sampled to another clean container for retention and testing.

12.3.2 If incremental samples are required for subsequent composite, samples may be taken as described in 12.1.2 or 12.1.3.

12.3.3 When tank cars or tank trucks are being loaded or unloaded, samples may conveniently be taken from the pipe line through which material is flowing by insertion of a sampling pipe into the rising section of the pipe line on the discharge side of the pump, or in a completely filled line in which gravity creates the material's flow.

12.3.3.1 A suitable in-line sampling pipe is shown in Fig. 2 and Fig. 3. Turn the opening of the sampling pipe to face the flow of the liquid. Provide the pipe with a valve or plug cock and discharge into a sample receiver. Before the sample is taken, draw 4 L (1 gal) of the material from the pipe valve and discard.

12.3.3.2 Take samples of 1 L (1 qt) at such frequency during the loading or unloading to provide a 4 to 8-L (1 to 2-gal) composite of the material. At the completion of the loading or unloading, thoroughly mix the collected sample and take a 0.5 to 4-L (1/8 to 1-gal) portion for testing.

12.4 Sampling from Drums or Barrels:

12.4.1 High-softening point pitches poured into drums for storage and shipment will solidify at ambient temperatures and sampling should be made in accordance with the procedure in 12.5.

12.4.2 Some soft pitches stored or shipped in drums or barrels will be semi-solid at ambient temperatures, and will permit the insertion of drum heaters. Drum "Band Heaters" are also useful in heating materials in semisolid condition. After thorough heating, the material can then be sampled.

12.4.3 Secure samples of liquid pitch from barrels or drums by "Tube" or "Thief" sampling by taking 1 L (1 qt) of material from the packages. When the lot of material to be sampled is obviously from a single run or batch, select one or more packages at random for sampling according to the following table:

Packages in Batch or Shipment	Packages to be Sampled
1 to 10	1
10 to 30	2
30 to 50	3
50 to 100	4

12.4.3.1 Take samples from at least 80 mm (3 in.) below the surface and at least 80 mm (3 in.) from the side of the container.

12.4.4 Reheat all samples, and if more than one sample is taken, combine them into a composite from which a 1 L (1 qt) portion shall be taken for examination.

12.4.5 If more than one batch is encountered, and it is not possible to differentiate between batches, it may be necessary to examine each sample separately, or take a higher proportionate number of samples per number of packages for individual or composite analysis.

13. Sampling Solid Pitch

13.1 Sampling Formed Pitches (Rod, Pencil, Prell, Pellet, Flake, or Chipped).

13.1.1 From Discharge Media (Shute, Pipe, Conveyer, etc.) to Container—Take incremental samples from discharge at a