

➡ § 2296. Motor Fuel Sampling Procedures.

(a) "Scope." This method covers procedures for obtaining representative samples of motor fuel and blending components used to make motor fuel.

(b) "Summary of method." It is necessary that the samples be truly representative of the product in question. The precautions required to ensure the representative character of the samples are numerous and depend upon the tank, carrier, container or line from which the sample is being obtained, the type and cleanliness of the sample container, and the sampling procedure that is to be used. A summary of the sampling procedures and their application is presented in Table 1. Each procedure is suitable for sampling a material under definite storage, transportation, or container conditions. The basic principle of each procedure is to obtain a sample in such manner and from such locations in the tank or other container that the sample will be truly representative of the product.

(c) "Description of terms."

(1) "Average sample" is one that consists of proportionate parts from all sections of the container.

(2) "All-levels sample" is one 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 about 3/4 full (maximum 85 percent) as it emerges from the liquid. An all-levels sample is not necessarily an average sample because the tank volume may not be proportional to the depth and because the operator may not be able to raise the sampler at the variable rate required for proportionate filling. The rate of filling is proportional to the square root of the depth of immersion.

(3) "Running sample" is one obtained by lowering an unstoppered beaker or bottle from the top of the liquid to the level of the bottom of the outlet connection or swing line, and returning it to the top of the liquid at a uniform rate of speed such that the beaker or bottle is about 3/4 full when withdrawn from the liquid.

(4) "Spot sample" is one obtained at some specific location in the tank by means of a thief bottle, or beaker.

(5) "Top sample" is a spot sample obtained 6 inches (150 mm) below the top surface of the liquid (Figure 1).

(6) "Upper sample" is a spot sample taken at the mid-point of the upper third of the tank contents (Figure 1).

(7) "Middle sample" is a spot sample obtained from the middle of the tank contents (Figure 1).

(8) "Lower sample" is a spot sample obtained at the level of the fixed tank outlet or the swing line outlet (Figure 1).

(9) "Clearance sample" is a spot sample taken 4 inches (100 mm) below the level of the tank outlet (Figure 1).

(10) "Bottom sample" is one obtained from the material on the bottom surface of the tank, container, or line at its lowest point.

(11) "Drain sample" is one obtained from the draw-off or discharge valve. Occasionally, a drain sample may be the same as a bottom sample, as in the case of a tank car.

(12) "Continuous sample" is one obtained from a pipeline in such manner as to give a representative average of a moving stream.

(13) "Mixed sample" is one obtained after mixing or vigorously stirring the contents of the original container, and then pouring out or drawing off the quantity desired.

(14) "Nozzle sample" is one obtained from a motor fuel service station pump nozzle which dispenses motor fuel from a underground storage tank.

(15) "Motor fuel" shall mean, for the purpose of this sampling procedure, gasoline (including gasoline containing oxygenates), diesel fuel, or any blending components that are used to make such fuel.

(d) "Sample containers."

(1) Sample containers may be clear or brown glass bottles, or cans. The clear glass bottle is advantageous because it may be examined visually for cleanliness, and also allows visual inspection of the sample for free water or solid impurities. The brown glass bottle affords some protection from light. The only cans permissible are those with the seams soldered on the exterior surface 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.

(2) "Container closure." Cork or glass stoppers, or screw caps of plastic or metal, may be used for glass bottles; screw caps only shall be used for cans to provide a vapor-tight closure seal. Corks must be of good quality, clean and free from holes and loose bits of cork. Never use rubber stoppers. Contact of the sample with the cork may be prevented by wrapping tin or aluminum foil around the cork before forcing it into the bottle. Glass stoppers must be a perfect fit. Screw caps must be protected by a disk faced with tin or aluminum foil, or other material that will not affect petroleum or petroleum products.

(3) "Cleaning procedure." All sample containers must be absolutely clean and free of water, dirt, lint, washing compounds, naphtha, or other solvents, soldering fluxes or acids, corrosion, rust, and oil. Before using a container, rinse it with Stoddard solvent or other naphtha of similar volatility. (It may be necessary to use sludge solvents to remove all traces of sediment and sludge from containers previously used.) Then wash the container with strong soap solution, rinse it thoroughly with tap water, and finally with distilled water. Dry either by passing a current of clean, warm air through the container or by placing it in a hot dust-free cabinet at 104 degrees Fahrenheit (40 degrees centigrade) or higher. When dry, stopper or cap the container immediately.

(e) "Sampling apparatus." The sampling apparatus is described in detail under each of the specific sampling procedures. Clean, dry, and free all sampling apparatus from any substance that might contaminate the material, using the procedure described in (d)(3).

(f) "Time and place of sampling." When loading or discharging product, take samples from both shipping and receiving tanks, and from the pipeline if required.

(1) "Ship or barge tanks." Sample each product after the vessel is loaded or just before unloading.

(2) "Tank cars." Sample the product after the car is loaded or just before unloading.

Note: When taking samples from tanks suspected of containing flammable atmospheres, precautions should be taken to guard against ignitions due to static electricity. Metal or conductive objects, such as gage tapes, sample containers, and thermometers, should not be lowered into or suspended in a compartment or tank which is being filled or immediately after cessation of pumping. A waiting period of approximately one minute will generally permit a substantial relaxation of the electrostatic charge; under certain conditions a longer period may be deemed advisable.

(g) "Obtaining samples."

(1) Directions for sampling cannot be made explicit enough to cover all cases. Extreme care and good judgment are necessary to ensure samples that represent the general character and average condition of the material. Clean hands are important. Clean gloves may be worn but only when absolutely necessary, such as in cold weather, or when handling materials at high temperature, or for reasons of safety. Select wiping cloths so that lint is not introduced, contaminating samples.

(2) As many petroleum vapors are toxic and flammable, avoid breathing them or igniting them from an open flame or a spark produced by static.

(3) When sampling relatively volatile products (more than 2 pounds (0.14 kgf/cm²) RVP), the sampling apparatus shall be rinsed 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 remain in this position until the contents have been transferred so that no unsaturated air will be entrained in the transfer of the sample.

(h) "Handling samples."

(1) "Volatile samples." It is necessary to protect all volatile samples of product 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. When samples of more than 16 pounds (1.12 kgf/cm²) RVP are being obtained, be sure to use containers strong enough to meet local safety regulations. After delivery to the laboratory, volatile samples should be cooled before the container is opened.

(2) "Container outage." Never completely fill a sample container, but 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.

(i) "Shipping samples." 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. The caps of metal containers must be screwed down tightly and checked for leakage. Postal and express office regulations applying to the shipment of flammable liquids must be observed.

(j) "Labeling sample containers."

(1) Label the container immediately after a sample is obtained. Use waterproof and oilproof ink or a pencil hard enough to dent the tag, since soft pencil and ordinary ink markings are subject to obliteration from moisture, oil smearing, and handling. Include the following information:

(A) Date and time (the period elapsed during continuous sampling),

(B) Name of the sample,

(C) Name or number and owner of the vessel, car, or container,

(D) Brand and grade of material, and

(E) Reference symbol or identification number.

(k) "Sampling procedures." The standard sampling procedures described in this method are summarized in Table 1. Alternative sampling procedures may be used if a mutually satisfactory agreement has been reached by the parties involved and such agreement was put in writing and signed by authorized officials.

(1) "Bottle or beaker sampling." The bottle or beaker sampling procedure is applicable for sampling liquids of 16 pounds (1.12 kgf/cm²) RVP or less in tank cars, tank trucks, shore tanks, ship tanks, and barge tanks.

(A) "Apparatus." A suitable sampling bottle or beaker as shown in Figure 2 is required. Recommended diameter of opening in the bottle or beaker is 3/4 inch (19 mm).

(B) "Procedure."

1. "All-levels sample." Lower the weighted, stoppered bottle or beaker as near as possible to the draw-off level, pull out the stopper with a sharp jerk of the cord or chain and raise the bottle at a uniform rate so that it is about 3/4 full as it emerges from the liquid.
2. "Running sample." Lower the unstoppered bottle or beaker as near as possible to the level of the bottom of the outlet connection or swing line and then raise the bottle or beaker to the top of the liquid at a uniform rate of speed such that it is about 3/4 full when withdrawn from the liquid.
3. "Upper, middle, and lower samples." Lower the weighted, stoppered bottle to the proper depths (Figure 1) as follows:

Upper sample.....	middle of upper third of the tank contents
Middle sample.....	middle of the tank contents
Lower sample.....	level of the fixed tank outlet or the swing-line outlet

Pull out the stopper with a sharp jerk of the cord or chain and allow the bottle or beaker to fill completely at the selected level, as evidenced by the cessation of air bubbles. When full, raise the bottle or beaker, pour off a small amount, and stopper immediately.

4. "Top sample." Obtain this sample (Figure 1) in the same manner as specified in (k)(1)(B)3. but at 6 inches (150 mm) below the top surface of the tank contents.
5. "Handling." Stopper and label bottle samples immediately after taking them, and deliver to the laboratory in the original sampling bottles.

(2) "Tap sampling." The tap sampling procedure is applicable for sampling liquids of 26 pounds (1.83 kgf/cm²) RVP or less in tanks which are equipped with suitable sampling taps or lines. This procedure is recommended for volatile stocks in tanks of the breather and balloon roof type, spheroids, etc. (Samples may be taken from the drain cocks of gage glasses, if the tank is not equipped with sampling taps.) When obtaining a sample for RVP or distillation analysis, use the assembly as shown in Figure 3. When obtaining a sample for other than RVP or distillation analysis, the assembly as shown in Figure 3 need not be used.

NOTE: If RVP is more than 16 pounds (1.12 kgf/cm²) but not more than 26 pounds (1.83 kgf/cm²) a cooling bath as shown in section (l)(6), Figure 5, shall be used between the tank tap and the sample container to cool the sample and prevent volatilization of low-boiling components.

(A) "Apparatus."

1. "Tank taps." The tank should be equipped with at least three sampling taps placed equidistant throughout the tank height. On tanks that are not equipped with floating roofs, each sample tap should extend into the a minimum of 10 cm (4 in.). A standard 1/4 inch pipe with suitable valve is satisfactory.
2. "Tube." A delivery tube that will not contaminate the product being sampled and long enough to reach to the bottom of the sample container is required to allow submerged filling. When a cooling bath is used while tap sampling, a similar suitable tube should be used between the tank tap and the cooler inlet.
3. "Sample containers." Use clean, dry glass bottles of convenient size and strength to receive the samples. In some cases, metal containers may be used instead of glass bottles.

(B) "Procedure."

1. Before a sample is drawn, flush the tap (or gage glass drain cock) and line until they are purged completely. Connect the clean delivery tube to the tap. Draw upper, middle, or lower samples directly from the respective taps after the flushing operation. Stopper and label the sample container immediately after filling, and deliver it to the laboratory.

2. When a sample cooler is used during the tap sampling operation, flush the tap (or gage glass drain cock). Then, using a section of clean tubing, connect the tap to the cooler inlet. Flush the cooler thoroughly, after which connect the clean delivery tube to the cooler outlet and proceed with the sampling operation.

(3) "Continuous sampling." The continuous sampling procedure is applicable for sampling liquids of 16 pounds (1.12 kgf/cm²) RVP or less and semiliquids in pipelines, filling lines, and transfer lines. The continuous sampling may be done manually or by using automatic devices.

(A) "Apparatus."

1. "Sampling probe." The function of the sampling probe is to withdraw from the flow stream a portion that will be representative of the entire stream. The apparatus assembly for continuous sampling is shown in Figure 4. Probe designs that are commonly used are as follows:

a. A tube extending to the center of the line and beveled at a 45 degree angle facing upstream (Figure 4(a)).

b. A long-radius forged elbow or pipe bend extending to the center line of the pipe and facing upstream. The end of the probe should be reamed to give a sharp entrance edge (Figure 4(b)).

c. A closed-end tube with a round orifice spaced near the closed end which should be positioned in such a way that the orifice is in the center of the pipeline and is facing the stream as shown in Figure 4(c).

2. Since the fluid pumped may not in all cases be homogeneous, the position and size of the sampling probe should be such as to minimize stratification or dropping out of heavier particles within the tube or the displacement of the product within the tube as a result of variation in gravity of the flowing stream. The sampling probe should be located preferably in a vertical run of pipe and as near as practicable to the point where the product passes to the receiver. The probe should always be in a horizontal position.

a. The sampling lines should be as short as practicable and should be cleared before any samples are taken.

b. A suitable device for mixing the fluid flow to ensure a homogeneous mixture at all rates of flow and to eliminate stratification should be installed upstream of the sampling tap. Some effective devices for obtaining a homogeneous mixture are as follows: Reduction in pipe size; a series of baffles; orifice or perforated plate; and a combination of any of these methods.

c. The design or sizing of these devices is optional with the user, as long as the flow past the sampling point is homogeneous and stratification is eliminated.

3. To control the rate at which the sample is withdrawn, the probe or probes should be fitted with valves or plug cocks.

4. "Automatic sampling devices" that meet the standards set out in (3)(A)5. may be used in obtaining samples of gasoline. The quantity of sample collected must be of sufficient size for analysis, and its composition should be identical with the composition of the batch flowing in the line while the sample is being taken. An automatic sampler installation necessarily includes not only the automatic sampling device that extracts the samples from the line, but also a suitable probe, connecting lines, auxiliary equipment, and a container in which the sample is collected. Automatic samplers may be classified as follows:

a. "Continuous sampler, time cycle (nonproportional) types." A sampler designed and operated in such a manner that it transfers equal increments of liquid from the pipeline to the sample container at a uniform rate of one or more increments per minute is a continuous sampler.

b. "Continuous sampler, flow-responsive (proportional) type." A sampler that is designed and operated in such a manner that it will automatically adjust the quantity of sample in proportion to the rate of flow is a flow-responsive (proportional) sampler. Adjustment of the quantity of sample may be made either by varying the frequency of transferring equal increments of sample to the sample container, or by varying the volume of the increments while maintaining a constant frequency of transferring the increments to the sample container. The apparatus assembly for continuous sampling is shown in Figure 4.

c. "Intermittent sampler." A sampler that is designed and operated in such a manner that it transfers equal increments of liquid from a pipeline to the sample container at a uniform rate of less than one increment per minute is an intermittent sampler.

5. "Standards of installation." Automatic sampler installations should meet all safety requirements in the plant or area where used, and should comply with American National Standard Code for Pressure Piping, and other applicable codes (ANSI B31.1). The sampler should be so installed as to provide ample access space for inspection and maintenance.

a. Small lines connecting various elements of the installation should be so arranged that complete purging of the automatic sampler and of all lines can be accomplished effectively. All fluid remaining in the sampler and the lines from the preceding sampling cycle should be purged immediately before the start of any given sampling operation.

b. In those cases where the sampler design is such that complete purging of the sampling lines and the sampler is not possible, a small pump should be installed in order to circulate a continuous stream from the sampling tube past or through the sampler and back into the line. The automatic sampler should then withdraw the sample from the sidestream through the shortest possible connection.

c. Under certain conditions, there may be a tendency for water and heavy particles to drop out in the discharge line from the sampling device and appear in the sample container during some subsequent sampling period. To circumvent this possibility, the discharge pipe from the sampling device should be free of pockets or enlarged pipe areas, and preferably should be pitched downward to the sample container.

d. To ensure clean, free-flowing lines, piping should be designed for periodic cleaning.

6. "Field calibration." Composite samples obtained from the automatic sampler installation should be verified for quantity performance in a manner that meets with the approval of all parties concerned, at least once a month and more often if conditions warrant. In the case of time-cycle samplers, deviations in quantity of the sample taken should not exceed + 5 percent for any given setting. In the case of flow-responsive samplers, the deviation in quantity of sample taken per 1,000 barrels of flowing stream should not exceed +5 percent. For the purpose of field-calibrating an installation, the composite sample obtained from the automatic sampler under test should be verified for quality by comparing on the basis of physical and chemical properties, with either a properly secured continuous nonautomatic sample or tank sample. The tank sample should be taken under the following conditions:

a. The batch pumped during the test interval should be diverted into a clean tank and a sample taken within one hour after cessation of pumping.

b. If the sampling of the delivery tank is to be delayed beyond one hour, then the tank selected must be equipped with an adequate mixing means. For valid comparison, the sampling of the delivery tank must be completed within eight hours after cessation of pumping, even though the tank is equipped with a motor-driven mixer.

c. When making a normal full-tank delivery from a tank, a properly secured sample may be used to check the results of the sampler if the parties mutually agree to this procedure.

7. "Receiver." The receiver must be a clean, dry container of convenient size to receive the sample. All connections from the sample probe to the sample container must be free of leaks. Two types of container may be used, depending upon service requirements.

a. "Atmospheric container." The atmospheric container shall be constructed in such a way that it retards evaporation loss and protects the sample from extraneous material such as rain, snow, dust, and trash. The construction should allow cleaning, interior inspection, and complete mixing of the sample prior to removal. The container should be provided with a suitable vent.

b. "Closed container." The closed container shall be constructed in such a manner that it prevents evaporation loss. The construction must allow cleaning, interior inspection and complete mixing of the sample prior to removal. The container should be equipped with a pressure-relief valve.

(B) "Procedure."

1. "Nonautomatic sample."

a. Adjust the valve or plug cock from the sampling probe so that a steady stream is drawn from the probe. Whenever possible, the rate of sample withdrawal should be such that the velocity of liquid flowing through the probe is approximately equal to the average linear velocity of the stream flowing through the pipeline. Measure and record the rate of sample withdrawal as gallons per hour. Divert the sample stream to the sampling container continuously or intermittently to provide a quantity of sample that will be of sufficient size for analysis.

2. "Automatic sampling." Purge the sampler and the sampling lines immediately before the start of a sampling operation. If the sample design is such that complete purging is not possible, circulate a continuous stream from the probe past or through the sampler and back into the line. Withdraw the sample from the side stream through the automatic sampler using the shortest possible connections. Adjust the sampler to deliver not less than 1 and not more than 40 gallons (151 liters) of sample during the desired sampling period. For time-cycle samplers, record the rate at which sample increments were taken per minute. For flow-responsive samplers, record the proportion of sample to total stream. Label the samples and deliver them to the laboratory in the containers in which they were collected.

(4) "Nozzle sampling." The nozzle sampling procedure is applicable for sampling product from a service station underground storage tank.

(A) "Apparatus." Sample containers conforming with (d)(1) should be used. A spacer, as shown in Figure 6, shall be used, if appropriate. When obtaining a sample for RVP or distillation analysis, an ice water bath and nozzle extension, as shown in Figure 7, shall be used. When obtaining a sample for other than RVP or distillation analysis, neither the ice water bath nor the nozzle extension need to be used.

(B) "Procedure."

1. When obtaining a sample for RVP or distillation analysis, conduct the sampling in the following manner: Immediately after gasoline has been delivered from pump and pump has been reset, deliver a small amount of product into the sample container, using spacer (Figure 6), if needed, on the pump nozzle (vapor recovery type). Rinse sample container and dump product into waste container. Insert nozzle extension (Figure 7) into sample container and insert pump nozzle into extension with slot over air bleed hole (if the extension is equipped with a slot). Replace sample container in chilling medium and fill slowly through nozzle extension to 70-80 percent full (Figure 8). Remove nozzle extension. Cap container at once. Check for leaks. Discard container and resample if leak occurs. If container is leak tight, place container in a cold chest of ice water.

2. When obtaining a sample for other than RVP or distillation analysis, the following procedure may be used instead of the procedure in (k)(4)(B)1: Immediately after product has been delivered from pump and pump has been reset, deliver a small amount of product into the sample container, using spacer (Figure 6), if needed, on the pump nozzle (vapor recovery type). Rinse sample container and dump product into waste container. Fill slowly with the nozzle to 70-80 percent full. Cap container at once. Check for leaks. Discard container and resample if leak occurs.

(l) "Special precautions and instructions for RVP Sampling."

(1) "Precautions." Vapor pressures are extremely sensitive to evaporation losses and to slight changes in composition. When obtaining, storing, or handling samples, observe the necessary precautions to ensure samples representative of the product and satisfactory for RVP tests. Official samples should be taken by, or under the immediate supervision of a person of judgment, skill, and sampling experience. Never prepare composite samples for RVP testing. Make certain that containers which are to be shipped by common carrier conform to Interstate Commerce Commission, state, or local regulations. When flushing or purging lines or containers, observe the pertinent regulations and precautions against fire, explosion, and other hazards.

(2) "Sample containers." Use containers of not less than 1 quart (1 liter) nor more than 2 gallons (7.5 liters) capacity, of sufficient strength to withstand the pressures to which they may be subjected, and of a type that will permit replacement of the cap or stopper with suitable connections for transferring the sample to the gasoline chamber (if applicable) of the vapor pressure apparatus. Open-type containers have a single opening which permits sampling by immersion. Closed-type containers have two openings, one in each end (or the equivalent thereof), fitted with valves suitable for sampling by water displacement or by purging.

(3) "Transfer connections." The transfer connection for the open-type container consists of an air tube and a liquid delivery tube assembled in a cap or stopper. The air tube extends to the bottom of the container. One end of the liquid delivery tube is flush with the inside face of the cap or stopper and the tube is long enough to reach the bottom of the gasoline chamber while the sample is being transferred to the chamber. The transfer connection for the closed-type container consists of a single tube with a connection suitable for attaching it to one of the openings of the sample container. The tube is long enough to reach the bottom of the gasoline chamber while the sample is being transferred.

(4) "Sampling open tanks." Use clean containers of the open type when sampling open tanks and tank cars. An all-level sample obtained by the bottle procedure, (k)(1) is recommended. Before taking the sample, flush the container by immersing it in the product to be sampled. Then obtain the sample immediately. Fill to 70-80 percent and close it promptly. Label the container and deliver it to the laboratory.

(5) "Sampling closed tanks." Containers of either the open or closed type may be used to obtain samples from closed or pressure tanks. If an open type container is used, follow the cooling bath procedure described in (l)(7) or (l)(10). If the closed type is used, obtain the sample using the water displacement procedure, (l)(8), or the purging procedure, (l)(9). The water displacement procedure is preferable because the flow of product involved in the purging procedure may be hazardous.

(6) "Cooling bath." A bath (Figure 5) of sufficient size to hold the sample container and a cooling coil of about 25 feet (8 m) of copper tubing (3/8 inch (9 mm) or less outside diameter) shall be required when using the procedure described in (l)(7). One end of the coil is provided with a connection for attaching it to the tank sampling tap or valve. The other end is fitted with a suitable valve (outlet) of good quality. A removable copper tube of 3/8 inch or less outside diameter and of sufficient length to reach the bottom of the sample container shall be connected to the open end of the outlet valve.

(7) "Cooling bath procedure." When using a cooling bath and a container of the open type, keep it at a temperature of 32 degrees to 40 degrees Fahrenheit (0 degrees to 4.5 degrees centigrade) during the sampling operation by using the cooling bath (Figure 5). Connect the coil to the tank sampling tap or valve and flush it with a sufficient amount of product to ensure complete purging. When obtaining a sample, throttle the outlet valve so that the pressure in the coil will be approximately the same as that

in the tank. Fill the container once to wash and cool it, and discard the wash product. Then draw the sample immediately. Pour off enough so that the container will be 70-80 percent full and close it promptly. Label the container and deliver it to the laboratory.

(8) "Water displacement procedure." Completely fill the closed-type container with water and close the valves. The water should be at the same temperature or lower than that of the product to be sampled. While permitting a small amount of product to flow through the fittings, connect the top or inlet valve of the container to the tank sampling tap or valve. Then open all valves on the inlet side of the container. Open the bottom or outlet valve slightly to allow the water to be displaced slowly by the sample entering the container. Regulate the flow so that there is no appreciable change in pressure within the container. Close the outlet valve as soon as gasoline discharges from the outlet; then in succession close the inlet valve and the sampling valve on the tank. Disconnect the container and withdraw enough of the contents so that it will be 70-80 percent full. If the vapor pressure of the product is not high enough to force liquid from the container, open both the upper and lower valves slightly to remove the excess. Promptly seal and label the container, and deliver it to the laboratory.

(9) "Purging procedure." Connect the inlet valve of the closed-type container to the tank sampling tap or valve. Throttle the outlet valve of the container so that the pressure in it will be approximately equal to that in the container being sampled. Allow a volume of product equal to at least twice that of the container to flow through the sampling system. Then close all valves, the outlet valve first, the inlet valve of the container second, and the tank sampling valve last, and disconnect the container immediately. Withdraw enough of the contents so that the sample container will be 70-80 percent full. If the vapor pressure of the product is not high enough to force liquid from the container, open both the upper and lower valves slightly to remove the excess. Promptly seal and label the container and deliver it to the laboratory.

(10) "Nozzle sampling procedure." When using a container of the open type, keep it at a temperature of 32 degrees to 40 degrees Fahrenheit (0 degree to 4.5 degrees centigrade) when sampling by the nozzle sampling procedure. The container may be chilled by placing it into an ice chest containing ice (frozen water). The sampling is accomplished following the procedure in (k)(4).

Table 1
Summary of Sampling Procedures and Applicability

Type of container	Procedure	Paragraph
Storage tanks, ship and barge tanks, tank cars, tank trucks	Bottle sampling	(k)(1)
Storage tanks with taps	Tap sampling	(k)(2)
Pipes and lines	Continuous line sampling	(k)(3)
Service station underground storage tanks	Nozzle sampling	(k)(4)

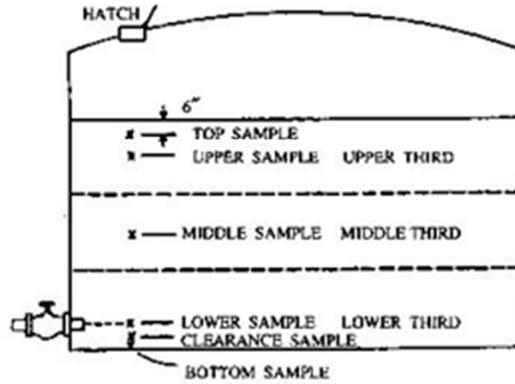


Figure 1. Sampling Depths

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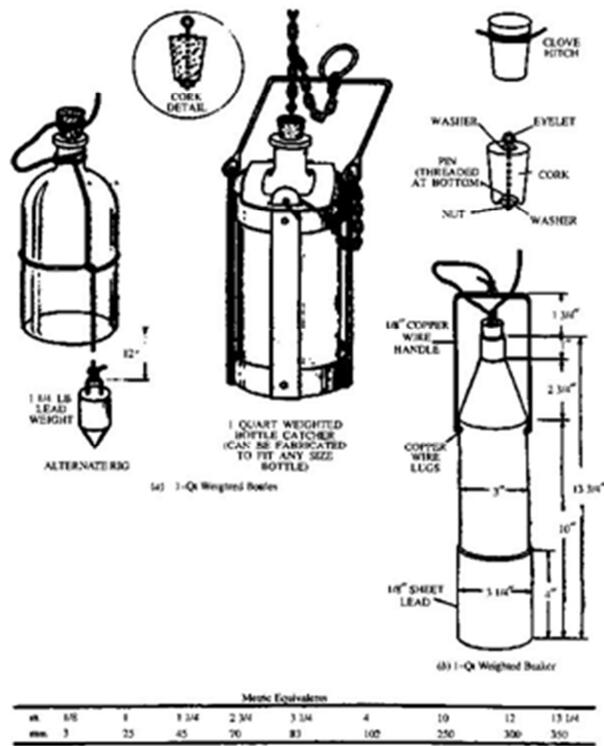


Figure 2. Assembly for Bottle Sampling

Metric Equivalents

in.	1/8	1	1 1/4	2 3/4	3 1/4	4	10	12	13 1/4
mm	3	25	45	70	83	102	250	300	350

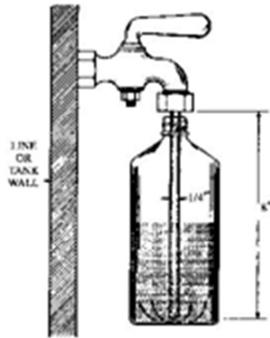


Figure 3. Assembly for Tap Sampling

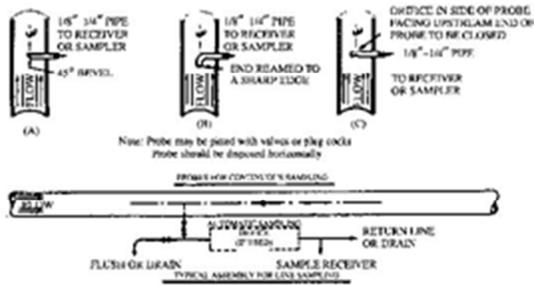


Figure 4. Probes for Continuous Sampling

Figure 3. Assembly for Tap Sampling

Figure 4. Probes for Continuous Sampling

Note: Probe may be pitted with valves or plug cocks. Probe should be disposed horizontally

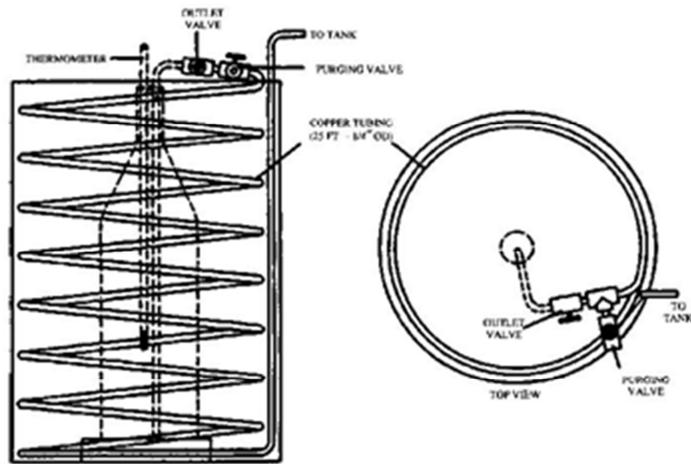
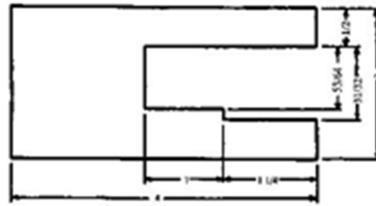


Figure 5. Cooling Bath

Figure 5. Cooling Bath

Figure 6. Spacer for Nozzle Sampling



Make from 1/4 inch flat steel
All dimensions in inches
Break all edges and corners

Figure 6. Spacer for Nozzle Sampling



Use 3/4 in. Schedule 40 Black Iron Pipe
All dimensions in inches
All tolerances = ±1/32 inch
A = Recommended 3/8"
B = Inside diameter Schedule 40 Black Iron Pipe



All dimensions in inches (not in notes)
All diameters represents maximum and minimum
Tolerances for all other dimensions in ±1/32"
Made of non-ferrous material, finished by passivate.

Figure 7. Nozzle Extensions for Nozzle Sampling

Make from 1/4 inch flat steel

All dimensions in inches

Break all edges and corners

Figure 7. Nozzle Extensions for Nozzle Sampling

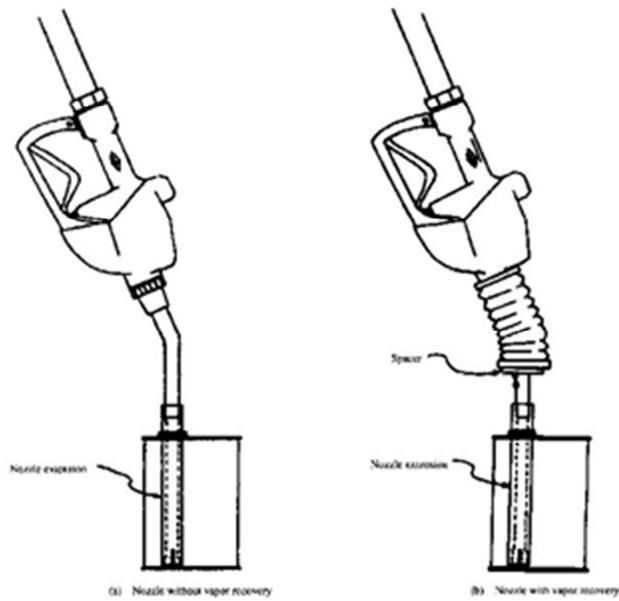


Figure 8. Assembly for Nozzle Sampling

Use 3/4 in. schedule 80 Black Iron Pipe

All dimensions in inches

All tolerances +1/128 inch

A - Recommend 30

B - Inside diameter Schedule 80 Black Iron Pipe

All dimensions in inches (not to scale).

All decimal dimensions represent minimum and maximum.

Tolerance for all other dimensions is +1/32".

Made of non-ferrous material, unaffected by gasoline.

Figure 8. Assembly for Nozzle Sampling

► **§ 2297. Test Method for the Determination of the Reid Vapor Pressure Equivalent Using an Automated Vapor Pressure Test Instrument.**

(a) Scope.

(1.0) This test method covers the determination of the total pressure, exerted in vacuum, by air-containing, volatile, petroleum products. The test method is suitable for testing samples with boiling points above 0° C (32° F) that exert a vapor pressure between 7 and 130 kPa (1.0 and 19 psi) at 37.8° C (100° F) at a vapor-to-liquid ratio of 4:1. The test method is suitable for testing gasoline samples which contain oxygenates. No account is made of dissolved water in the sample. (Samples can also be tested at other vapor-to-liquid ratios, temperatures and pressures, but the Precision and

Bias as described in section (k) do not necessarily apply.)

(2.0) This test method covers the use of automated vapor pressure instruments that perform measurements on liquid specimen sizes in the range from 1 to 10 ml.

(3.0) Standard values are specified in SI units (International System of Units). The values given in parentheses are provided for information purposes only.

(4.0) This test method may involve hazardous materials, operations, and equipment. This test method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see section (g)(5.0).

(b) Summary of Test Method.

(1.0) A known volume of chilled, air-saturated sample is introduced into a thermostatically controlled test chamber, the internal volume of which is five times that of the total test specimen introduced into the chamber. A vacuum is applied to the chamber in accordance with the manufacturer's instructions. After introduction into the test chamber the test specimen is allowed to reach thermal equilibrium at the test temperature, 37.8° C (100° F). The resulting rise in pressure in the chamber is measured using a pressure transducer sensor and indicator.

(2.0) Only the sum of the partial pressure of the sample and the partial pressure of the dissolved air (commonly known as the total pressure) are used in this test method. Note that some instruments may call this pressure measurement by another term. Also note that some instruments are capable of measuring the absolute pressure of the specimen as well.

(3.0) The measured total vapor pressure is converted to a Reid vapor pressure equivalent (RVPE) by use of a calibration equation (section (i)(1.0)). This calculation converts the measured total pressure to the Reid vapor pressure (RVP) expected from the American Society of Testing and Materials (ASTM) Test Method D 323-58.

(c) Apparatus.

(1.0) Vapor Pressure Apparatus - An appropriate instrument, designed for the intended use should be selected. The minimum performance level for the automated vapor pressure test instrument is that the instrument shall perform as well as, or better than, the precision criteria set forth in the ASTM D323- 58, which is incorporated herein by reference. The ASTM D323-58 states a repeatability value of 0.2 psi and a reproducibility value of 0.3 psi. The instrument shall provide accurate results which are comparable to the RVP measured by the ASTM 323-58. Typically, the type of apparatus suitable for use in this test method employs a small volume test chamber incorporating a transducer for pressure measurements and associated equipment for thermostatically controlling the chamber temperature and for evacuating the test chamber.

(1.1) The test chamber shall be designed to contain between 5 and 50 ml of liquid and vapor and be capable of maintaining a vapor-to-liquid ratio between 3.95 to 1.00 and 4.05 to 1.00.

(1.2) The pressure transducer shall have a minimum operational range from 0 to 177 kPa (0 to 25.6 psi) with a minimum resolution of 0.1 kPa (0.01 psi) and a minimum accuracy of ± 0.3 kPa (± 0.05 psi). The pressure measurement system shall include associated electronics and readout devices to display the resulting pressure reading.

(1.3) The thermostatically controlled heater shall be used to maintain the test chamber at $37.8 \pm 0.1^\circ$ C ($100 \pm 0.2^\circ$ F) for the duration of the test.

(1.4) A platinum resistance thermometer shall be used for measuring the temperature of the test chamber. The minimum temperature range of the measuring device shall be from ambient to 60° C (140° F) with a resolution of 0.1° C (0.2° F) and accuracy of 0.1° C (0.2° F).

(1.5) The vapor pressure apparatus shall have provisions for introduction of the test specimen into the test chamber and for the cleaning or purging of the chamber following the test.

(2.0) A vacuum pump (if required by the manufacturer's instructions) shall be capable of reducing the pressure in the test chamber to less than 0.01 kPa (0.001 psi) absolute.

(3.0) A syringe (optional, depending on sample introduction mechanism employed with each instrument) shall be gas-tight. The syringe shall be 1 to 20-ml capacity with a $\pm 1\%$ or better precision. The capacity of the syringe should not exceed two times the volume of the test specimen being dispensed.

(4.0) Ice Water Bath or Refrigerator (Air Bath): for chilling the samples and syringe to temperatures between and 1°C (32 to 34°F).

(5.0) Mercury Barometer (if required by the manufacturer's instructions): in the 0 to 120 kPa (0 to 17.4 psi) range.

(6.0) McLeod Vacuum Gage (if required by the manufacturer's instructions): to cover at least the range from 0 to 0.67 kPa (0 to 5mm Hg).

(d) Sampling.

(1.0) Obtain a sample in accordance with title 13, California Code of Regulations, section 2296.

(2.0) The extreme sensitivity of vapor pressure measurements to losses through evaporation and the resulting changes in composition is such as to require the utmost precaution and most meticulous care in the handling of samples.

(3.0) Protect samples from excessive high temperatures prior to testing. This can be accomplished by storage in an appropriate ice water bath or refrigerator.

(4.0) Do not test samples stored in leaky containers. Discard and obtain another sample if leaks are detected.

(e) Preparation of Apparatus.

(1.0) Prepare the instrument for operation in accordance with the manufacturer's instructions.

(2.0) Clean and prepare the test chamber as required to avoid contamination of the test specimen.

(3.0) For instruments that require that the test chamber be evacuated prior to the introduction of the test specimen: Prior to specimen introduction, visually determine from the instrument display that the test chamber pressure is stable and does not exceed 0.1 kPa (0.01 psi). When the pressure is not stable or exceeds this value, check that the chamber is clean of volatile materials remaining in the chamber from a previous specimen or check the calibration of the transducer.

(4.0) If a syringe is used for introduction of the specimen, chill it to between 0 and 4.5°C (32 and 40°F) in an ice water bath or a refrigerator before drawing in the specimen. Avoid water contamination of the syringe reservoir by suitably sealing the outlet of the syringe during the cooling process.

(5.0) For instruments using a pre-heated test chamber: Prior to introduction of the test specimen check that the temperature of the test chamber is within the required range from $37.8 \pm 0.1^{\circ}\text{C}$ ($100 \pm 0.2^{\circ}\text{F}$).

(f) Calibration.

(1.0) Pressure Transducer:

(1.1) Check the calibration of the pressure transducer on a monthly basis or when needed as indicated from the quality control checks (section (g)). The calibration of the pressure transducer is checked using two reference points, zero pressure (<0.1kPa) and the ambient barometric pressure.

(1.2) Connect a McLeod gage to the vacuum source in line with the test chamber. Apply a vacuum to the test chamber. When the McLeod gage registers a pressure less than 0.1 kPa (0.8mm Hg, or 0.01 psi), adjust the pressure transducer control to zero or to the actual reading on the McLeod gage as dictated by the instrument design and manufacturer's instructions.

(1.3) Open the test chamber to the atmosphere and observe the pressure transducer reading. If the pressure reading is not equal to the ambient barometric pressure, then adjust the pressure transducer span control until the appropriate reading is observed. Ensure that the instrument is set to display the total pressure and not a calculated or corrected value.

(1.4) Repeat steps (f)(1.2) and (f)(1.3) until the zero and barometric pressures read correctly without further adjustments.

(2.0) Thermometer - Check the calibration of the platinum resistance thermometer used to monitor the temperature of the test chamber at least every six months against a National Institute on Standards and Technology (NIST) traceable thermometer.

(g) Quality Control Checks.

(1.0) Check the performance of the instrument each day it is in use by running a quality control sample consisting of a pure solvent of known vapor pressure similar to the vapor pressure of the samples to be tested. Treat the pure solvent quality control check sample in the same manner as a sample (section (h)). Record the total vapor pressure (do not calculate a Reid vapor pressure equivalent) in a log for the purpose of tracking the instrument's performance. If the total vapor pressure differs from the previous entry (for the same pure solvent) in the log by more than +/- 1.0 kPa (0.15 psi), then check the instrument calibration (section (f)). If the trend of the log shows variations of more than +/- 1.0 kPa (0.15 psi) (for the same pure solvent), also check the instrument calibration.

(2.0) Some of the possible reference pure materials and their corresponding absolute vapor pressures include:

cyclohexane	22.5 kPa	(3.27 psi)
cyclopentane	68.3 kPa	(9.92 psi)
2,2-dimethylbutane	67.9 kPa	(9.86 psi)
2,3-dimethylbutane	51.1 kPa	(1.41 psi)
2-methylpentane	46.7 kPa	(6.77 psi)
toluene	7.1 kPa	(1.03 psi)

(The total pressure values cited were obtained from Phillips Petroleum Co., Bartlesville, OK, or the Table of Physical Constants, National Gas Producer Association.)

(3.0) Purity of Reagents - Use chemicals of at least 99% purity for quality control checks. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. ("Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co, Inc., New York, NY and the "United States Pharmacopeia.") Lower purities can be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

(4.0) The chemicals in this section are suggested for use in quality control procedures; not for instrument calibration.

(5.0) WARNING -Cyclohexane, cyclopentane, 2,2-dimethylbutane, 3,2- dimethylbutane, 2-methylpentane, and toluene are extremely flammable. They are an aspiration hazard and are harmful if inhaled. They are also a skin irritant on repeated contact.

(h) Procedure.

(1.0) Sample Temperature - Cool the sample container and contents in an ice water bath or refrigerator to the 0 to 1° C (32 to 34° F) range prior to opening the sample container. Allow sufficient time to reach this temperature.

(2.0) Verification of Sample Container Filling - After the sample reaches thermal equilibrium at 0 to 1° C, take the container from the ice water bath or refrigerator, wipe dry with an absorbent material, unseal and examine the ullage. With a suitable gage, determine that the liquid content in the container is between 70 to 80% of the volume of the container capacity.

(2.1) Discard the sample if the liquid content of the container is less than 70% of the volume of the container capacity.

(2.2) If the liquid content of the container is more than 80% of the volume of the container capacity, pour out enough sample to bring the liquid contents within the 70 to 80% volume range.

(3.0) Air Saturation of Sample in Sample Container

(3.1) After determining that the liquid content in the sample container is between 70 to 80% full, reseal the container and shake vigorously. Return the container to the ice water bath or refrigerator for a minimum of 2 minutes.

(4.0) Remove the sample from the ice water bath or refrigerator, dry the exterior of the container with absorbent material, uncap, insert a transfer tube or syringe (section (e)(4.0)). Draw a bubble-free aliquot of sample into a gas tight syringe or transfer tube and deliver this test specimen to the test chamber as rapidly as possible. The total time between opening the chilled sample container and inserting/securing the syringe into the sealed test chamber shall not exceed 1 minute.

(5.0) The vapor pressure determination shall be performed on the first test specimen withdrawn from a sample container. Successive vapor pressure determinations can be made on the remaining test material in the same container if the container had been tightly sealed immediately after the previous vapor pressure determination.

(6.0) Follow the manufacturer's instructions for the introduction of the test specimen into the test chamber, and for the operation of the instrument to obtain a total vapor pressure result for the test specimen.

(7.0) Set the instrument to read the result in terms of total vapor pressure. If the instrument is capable of calculating a Reid vapor pressure equivalent value, ensure that only the parameters described in section (i)(2.0) are used.

(8.0) Verification of Single Phase - After drawing a test specimen and introducing it into the instrument for analysis, check the remaining sample for phase separation. If the sample is contained in a glass container, this observation can be made prior to sample transfer. If the sample is contained in a non-transparent container, mix the sample thoroughly and immediately pour a portion of the remaining sample into a glass container and observe for evidence of phase separation. If the sample is not clear and bright or if a second phase is observed, discretion shall be used to determine if the sample is truly representative.

(9.0) Record the total vapor pressure reading from the instrument to the nearest 0.1 kPa (0.01 psi). For instruments that do not automatically record or display a stable pressure value, manually record the pressure indicator reading every minute to the nearest 0.1 kPa; and, when three successive readings agree to within 0.1 kPa, record the result to the nearest 0.1 kPa (0.01 psi).

(i) Calculation.

(1.0) Calibration Equation - Calculate the Reid vapor pressure equivalent (RVPE) using the following calibration equation. Ensure that the instrument reading used in this equation corresponds to the total pressure and has not been corrected by an automatically programmed correction factor.

Equation 1:

$$RVPE = aX - b$$

where:

"RVPE" is the vapor pressure value (in psi) that would be expected from test method ASTM D323-58;

"a" is the correlative relationship of test data from the specific automated vapor pressure test instrument and test data from ASTM D323-58;

"X" is the total vapor pressure value (in psi) as determined by the specific automated vapor pressure test instrument;

"b" is the offset of the test data between the specific automated vapor pressure test instrument and the test data from ASTM D323-58.

The data used for determining the calibration equation for each instrument shall be obtained during an Air Resources Board vapor pressure test program. The data shall consist of test results obtained from the analysis of identical samples by the automated instrument and by ASTM D323-58. Vapor pressure test programs may be conducted on a periodic basis as needed. The Air Resources Board conducted such a program and determined that the following automated vapor pressure test instruments meet the requirements of section (c). The data from the test program were used to arrive at the calibration equations for these instruments. The calibration equations are as follows:

1. Grabner Instruments,

Model: CCA-VP (laboratory Grabner) $RVPE = (.965) x - .304$

2. Grabner Instruments,

Model: CCA-VPS (portable Grabner) $RVPE = (.972) x - .715$

3. Stanhope-Seta Limited,

Model: Setavap $RVPE = (.961) x - .577$

(2.0) The calculation described in section (i)(1.0), above, can be accomplished automatically by the instrument, if so equipped, and in such cases the user shall not apply any further corrections.

(j) Report.

(1.0) Report the Reid vapor pressure equivalent to the nearest 0.1 kPa (0.01 psi).

(k) Precision and Bias.

(1.0) Precision - The precision of this test method as determined by the statistical examination of interlaboratory test results is as follows:

(1.1) Repeatability - The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the

long run, in the correct operation of the test method exceed the following value only in one case in twenty. The repeatability values for the specific automated vapor pressure test instruments listed in section (i)(1.0) are:

1. Grabner Instruments, Model: CCA-VP (laboratory Grabner) 0.084 psi 2. Grabner Instruments, Model: CCA-VPS (portable Grabner) 0.084 psi 3. Stanhope-Seta Limited Model: Setavap 0.10 psi

(1.2) Reproducibility - The difference between two single and independent test results obtained by different operators working in different laboratories using the same make and model test instrument on identical test material would, in the long run, in the correct operation of the test method exceed the following value only in one case in twenty. The reproducibility values for the specific automated vapor pressure test instruments listed in section (i)(1.0) are:

1. Grabner Instruments, Model: CCA-VP (laboratory Grabner) 0.13 psi 2. Grabner Instruments, Model: CCA-VPS (portable Grabner) 0.21 psi 3. Stanhope-Seta Limited Model: Setavap 0.32 psi

(2.0) Bias - A relative bias was observed between the total pressure obtained using this test method and the Reid vapor pressure obtained using ASTM Test Method D323-58. This bias is corrected by the use of the calibration equation in section (i)(1.0) which calculates a Reid vapor pressure equivalent value from the observed total pressure.