



Designation: D 1247 - 54  
(Reapproved 1975)

American National Standard Z77.7-1973 (R-1963)  
Approved March 21, 1973  
By American National Standards Institute

## Standard Method of SAMPLING MANUFACTURED GAS<sup>1</sup>

This Standard is issued under the fixed designation D 1247; 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.

### 1. Scope

1.1 This method covers the procedures for securing representative samples of manufactured gas, and correlates the size or type of sample with the analysis to be done subsequently on that sample.

NOTE—The values stated in U.S. customary units are to be regarded as the standard.

### 2. General Considerations

2.1 In collecting samples of manufactured gas, consideration must be given to the purposes for which the samples are to be used and the conditions under which they must be secured. Consideration must also be given to the volume of sample required for the purpose intended, to the size and length of the sampling line, and to the size and design of containers. Account must be taken of the possible constituents in the gas with reference to any action upon the material of which the lines, containers, and auxiliary equipment are constructed. The procedure used should be capable of giving results that can be duplicated. Unless a representative sample has been obtained, the results of any subsequent analytical test, regardless of the accuracy of the test itself, will be erroneous and misleading.

### 3. Volume of Sample Required

3.1 The volume of sample collected is governed by the test to be made and the method to be used. The following volumes are minimum amounts sufficient for two determinations and necessary purging:

Chemical gas analysis	1000 cm <sup>3</sup>
Fractionation gas analysis	5 to 10 ft <sup>3</sup> (0.141 to 0.283 m <sup>3</sup> )
Specific gravity	1 ft <sup>3</sup> (0.028 m <sup>3</sup> )
Heating value (Btu)	3 to 5 ft <sup>3</sup> (0.085 to 0.141 m <sup>3</sup> )

Total sulfur by Referee's apparatus	to 0.141 m <sup>3</sup> )
Determination of ammonia	10 ft <sup>3</sup> (0.283 m <sup>3</sup> )
Determination of naphthalene	10 ft <sup>3</sup> (0.283 m <sup>3</sup> )
	5 to 10 ft <sup>3</sup> (0.141 to 0.283 m <sup>3</sup> )
Hydrogen sulfide by Tutweiler method	1100 cm <sup>3</sup>
Hydrogen sulfide by cadmium precipitation	5 ft <sup>3</sup> (0.141 m <sup>3</sup> )

### 4. Description of Terms

4.1 *Manufactured Gas*—The expression "manufactured gas" as used in this method refers to fuel gas made from certain raw materials, primarily coal and oil. It may consist of one or more of such gases as carbureted water gas, coke oven gas, re-formed natural gas, producer gas, and oil gas. The principal constituents of this mixture are hydrogen, carbon monoxide, saturated and unsaturated hydrocarbons, carbon dioxide, nitrogen, and oxygen. Impurities may include organic sulfur, hydrogen sulfide, and ammonia.

4.2 *Sampling Probe* is that portion of the sampling line attached to, and possibly extending into, the main or vessel containing the gas to be sampled.

4.3 *Sampling Line* is that portion of the piping or tubing through which the sample is conducted from the sampling probe to the sample container.

4.4 *Sample Container* is the vessel in which the gas sample is collected, stored, and transported to the analytical apparatus.

### 5. Outline of Method

5.1 The method specifies the conditions for

<sup>1</sup> This method is under the jurisdiction of ASTM Committee D-3 on Gaseous Fuels.  
Current edition effective Sept. 15, 1954. Originally issued 1952. Replaces D 1247 - 52 T.

conducting a stream of gas from the sampling source into a purged container, and thus securing a representative sample. Snap samples may suffice, or a series of consecutive snap samples may be considered as an average sample. Average samples may also be obtained by continuous sampling into a portable container or direct to a recording or indicating instrument over a predetermined period of time.

5.2 Purging may be accomplished by means of the gas to be sampled, either by displacement of air or by water displacement; or by the utilization of a vacuum.

5.3 Manufactured gas will usually be under sufficient pressure to purge and fill the sample container; otherwise auxiliary mechanical equipment must be employed (see 6.1.4).

5.4 The choice of the type of sample ("snap" or "continuous"), the method of purging the kind of container, and the selection of auxiliary pressure equipment, will be determined by the analyses to be done on the sample, by the composition of the gas to be sampled, and by the available equipment.

## 6. Apparatus

6.1 The sampling equipment should guard against contamination by air, leakage, and losses by absorption or chemical reaction. The use of rubber should be kept to a minimum, and any rubber used should be of a grade that will not react with the sample and that will have low permeability characteristics. Plastic tubing may be as satisfactory where flexibility is imperative. Rubber hose should not be used where high pressures are involved.

6.1.1 *Sampling Probes* of metal are preferable because of their durability. Iron pipe,  $\frac{1}{8}$  or  $\frac{1}{4}$  in. in diameter, threaded or welded to the main or other source of sample and extending some distance into the vessel, makes a satisfactory connection. Condensate on the inside surface of the main will be avoided, and the point of average velocity of the gas, one sixth of the diameter of the main, can be reached at the same time. Figure 1 shows a simple open-end sampling tube. If unusually high temperatures are encountered, water-cooled probes (Fig. 2) may be required.<sup>2</sup>

6.1.2 *Sampling Lines*—Iron pipe  $\frac{1}{8}$  or  $\frac{1}{4}$  in. in diameter, and copper, Monel metal, or

brass tubing  $\frac{1}{2}$  to  $\frac{3}{8}$  in. (2.4 to 9.5 mm) in outside diameter, are recommended as sampling lines. Where sulfur compounds are to be determined, glass or quartz tubing should be used for the low pressures and aluminum when higher pressures are met. Any sampling line should be as short and of as small a diameter as practicable to decrease purging time, particularly in the case of a continuous sampling line direct from the source of supply to analytical apparatus in order that the test instrument may follow closely any change in composition of the supply.

6.1.3 *Sample Containers* may vary in size from 125 cm<sup>3</sup> to several cubic feet. The design and type may take many forms, such as tubes, cylinders, or tanks, which may be sealed by stopcocks or valves. They shall be made of material that will not react with the sample, usually glass, steel, or any metal or alloy sufficiently strong and gastight. Figures 3, 4, and 5 illustrate typical sample containers. Whenever necessary to ship a sample of manufactured gas under pressure, the container must meet the requirements of the Interstate Commerce Commission, and it should be correctly labeled for identification and proper handling by the analyst. If the sample is to be shipped under pressure the vessel must comply with the requirements of, and be approved by, the Interstate Commerce Commission.

6.1.4 *Aspirators and Pumps*—When the gas to be collected is not under positive pressure, some form of aspirating device (see Fig. 6) must be employed to draw the gas through the sampling probe and into the sample container. This device may be of any of the following types:

6.1.4.1 A very simple form of aspirator, which also serves as a sample container is shown in Fig. 6 (a). It consists of two interchangeable bottles of equal size. Water from the upper bottle, *A*, flows through the siphon tube, *E*, to the lower bottle, *C*, thereby creating a vacuum at tube *F*. If tube *F* is connected to the sampling probe, the gas will be aspirated into bottle *A*. When all of the water has been siphoned from the upper bottle into

<sup>2</sup> Altieri, V. J., "Gas Analysis and Testing of Gaseous Materials," Am. Gas Assn., AGACA, pp. 76-79.

the lower bottle, they may be interchanged and the aspirating continued as before.

6.1.4.2 A double-action, rubber-bulb hand pump, as shown in Fig. 6 (b), is convenient for purging sampling apparatus and filling sample containers. It may be used for either suction or pressure as it has a simple valve at each end, the two being of opposite action. In use, one end of the empty sample container is connected with the gas supply and the bulb is connected to the other end with the valves in such a position that the pressure and relief on the bulb will draw air out of the container and admit the gas. Fifty compressions of an ordinary 40 to 60 cm<sup>3</sup> capacity bulb will completely replace the air in a 250 cm<sup>3</sup> sample container. A large enough safety is provided because the bulbs become less efficient with use, although their usefulness can be fairly well determined by compressing the bulb, closing the intake end, and noting whether there is any leakage, and then repeating, endeavoring to compress the bulb with the effluent end closed. Dust sometimes collects on the ledges of the valve seats, and a seemingly defective bulb is often remedied by the removal of such dust or by wetting the valve seats with water. An added precaution against air contamination through leaky valves should be taken during sampling. This is most readily accomplished by connecting a length of rubber tubing to the effluent end of the bulb and placing the outlet of this tube under water.

6.1.4.3 A hand pump equipped with double-acting valves (Fig. 6 (c)) may be used.

6.1.4.4 When running water is available, an ordinary laboratory filter pump of the "Chapman" type, as shown in Fig. 6 (d), may be used for aspirating the gas through the sampling apparatus. If a water pump of this type is not available, one may readily be improvised.

6.1.5 *Confining or Displacing Medium* used in the sample container should not react with the container or the sample. Water is the most convenient, but has more or less solvent action on all gases. The error may be minimized by slightly acidifying the water or by dissolving certain salts in it; for example, sodium chloride. Mercury is the most satisfactory liquid and should be used for precise

work as no solubility precautions are necessary; however, it has the disadvantages of its cost, excessive weight, and the fact that it reacts with hydrogen sulfide. Displacement of the sample from the container to the test apparatus may be accomplished by release of pressure if neither water nor mercury is permissible.

6.1.6 *Stopcock Lubricant*—An excessive amount of grease should be avoided when lubricating stopcocks, since many gases, particularly the hydrocarbons, are soluble in these greases. The new silicone lubricants may be superior to the older commercial products for this purpose.

## 7. Procedure for Collection of Snap Samples

7.1 Sample containers shown in Fig. 3, Type C, and in Fig. 4 are generally used for taking snap samples.

7.2 If there is an abundant supply of gas and appreciable pressure, dry procedures (air displacement) may be used. In this method, the container is purged with the gas to be sampled until the original contents are displaced, a flow of gas equivalent to ten times the volume of the sampling container being sufficient. If it is desired to use the air displacement method under negative pressure, an aspirating device of the type described in 6.1.4 may be connected to the end of the sample container and purging operations performed until the original air has been displaced.

7.3 In taking a sample when the gas supply is limited, the liquid displacement method is usually employed. The container is first filled with displacing liquid and then connected to the gas supply by means of connections that have previously been purged of air and dead gas. The latter may be done by gas displacement, that is, allowing the primary stream to flow for a short time before sampling or filling the connections with the displacing liquid before connecting the container. The container is then filled by allowing the displacing liquid to run out ahead of the gas, after which the outlet end of the container is closed and then the inlet.

7.4 When the gas to be sampled is not under pressure, one of the various types of aspirators or suction pumps previously described should be used to set up a primary stream of

gas. A satisfactory arrangement for filling containers either by water or mercury displacement from streams under supply positive pressure, neutral pressure, or a few inches of negative pressure is similar to the device shown in Fig. 7. If the gas is under appreciable positive pressure, bottle *A* may be omitted from the device and the hose connected directly to the sample container, *C*, but if the gas is at neutral pressure, and especially if it is under slight negative pressure, *A* is necessary. The container, *C*, is filled with the displacing liquid from the aspirator bottle and then connected to the sampling tube, *D*, upon which the connection is also filled with the liquid or purged by drawing a portion of the sample and rejecting it either back into the primary sampling stream if there is a continuous flow, or to the exterior by means of a three-way stopcock, as shown. After the connections have been purged, the sample container is filled by allowing the liquid to return to the aspirator bottles. In case of neutral or negative pressure, bottle *A* is partly filled with gas at the time of taking the sample and, after closing stopcock *E*, the aspiratory bottle, *B*, is raised; this forces some gas back into the container; stopcock *F* is then closed. This restores atmospheric pressure or, what is more desirable, the sample can be put under positive pressure which tends to avoid air contamination through leaks at *E* and *F* during the interim before analysis. The entire device may be mounted on a ring stand with a large clamp to receive the sample container, *C*.

#### 8. Procedures for Collection of Continuous Samples

8.1 Fig. 8 shows a simple arrangement of two 8 to 10-liter aspirator bottles connected by about 4 ft (1 m) of medium-heavy-wall rubber tubing. In use, the lower bottle is placed 2 to 4 ft (0.6 m to 1.2 m) below the upper one. The flow of water can be regulated more easily at a greater difference in the two heights of the bottles, and this results in a more uniform rate of collecting gases over long periods. In constructing the bottle, care should be taken that the "ell" inlet of *B* does not extend below the stopper and trap gas.

8.1.1 To collect a sample, connect the bottles to the secondary gas outlet as shown, except that *F* shall be connected to *G*. With

screw clamps open, force the confining liquid to the point *E*. Then, by lowering *D*, draw enough gas into *B* to purge the connection (about one fourth the volume of *B*) and immediately discharge it back through *E*. Repeat the purging procedure and when the liquid reaches *E* the second time, close screw clamps *D* and *G*. Remove the connection to *G* and replace it in the stopper of bottle *C*, upon which the apparatus is ready for collecting a sample. The screw clamp *D* may be adjusted to any desired flow of solution and, consequently, collection of sample. The correct adjustment of the regulating pump can be made easily by observing the amount of water dripping from *F*. The size of the drop for the particular tube in *F* should be determined and used as a basis for calculating rates of collection of sample.

8.1.2 After the gas has been collected, close screw clamp *D*, then *A*, and again connect *F* to *G* (being careful to avoid entrapping air bubbles). Place the sample under slight positive pressure by raising *C* until the level of the liquid is about an inch or two above that in *B* and open *D* for a minute or two. The sample may now be taken to the laboratory or a snap sample taken for analysis

8.2 An improved and more desirable method for collecting accumulative samples, using aspirating bottles, is shown in Fig. 9. In the diagram, *A* and *B* are aspirator bottles of suitable size for collecting the samples. If 24-h samples are to be collected, the capacity of the bottles should be not less than 10 liters each. Bottle *A* is connected to the gas line with glass or metal tubing, but may be connected to *B* with rubber tubing. Bottle *B* should be connected to the gas line by flexible tubing. A flow indicator, *H*, of the bubble bottle type serves to give visual control of the flow of gas through the primary gas line. A pressure regulator, *F*, and a flow meter, *G*, are provided for regulating the rate of collection. The sample container, *E*, is not connected until the sample is to be collected.

8.2.1 To collect a sample, open stopcock *D* and allow the primary stream of gas to flow through the bubble bottle to a bleeder connection. Fill bottle *B* with the confining liquid and, with all stopcocks closed, except screw clamp *L* at the outlet of *A*, break the connection between bottle *A* and the flow meter, *G*.

Open stopcock *C* to admit the gas to bottle *B*. Raise *B* so that the gas pressure forces the liquid back into *A* and the air, or gas from the previous sample, contained in *A* is forced out through the tube to where the connection is broken at *G*. When *A* is filled with the liquid as far as the broken connection, close stopcock *C* and the screw clamp *L*. Now, open stopcock *C* and allow the gas to purge through *F* and *G*. Connect the flowmeter *G* to *A* again and lower bottle *B* until the liquid flows into it. Open the outlet stopcock at *M* and connect to a burner or purge line, and adjust the screw clamp *L* at the bottom of *A* to give the desired rate of flow as indicated by the flowmeter.

8.2.2 At the end of the sampling period, close stopcock *C*, the screw clamp *L*, and the stopcock, *M*, at the outlet of *B*, and take the sample from *A* by attaching a sample container as shown in Fig. 10. Transfer a portion of the gas to the sample container by raising the level of bottle *B*.

8.3 The sample container illustrated in *F*, Fig. 3, is used for collecting continuous samples by mercury displacement, as shown in Fig. 10. The container should have a capacity of about 250 cm.<sup>3</sup>

8.3.1 To take a sample, open stopcocks *A* and *B*. As the mercury flows from the lower outlet, the gas will be drawn into the container and will collect above the mercury. As long as the surface of the mercury remains above the gas inlet tube, the flow of mercury will be constant and the same volume of gas will be collected in each equal interval of sampling period. The time required for the mercury to flow from the container may be varied by attaching a glass orifice to the outlet end of the container. The rate of sampling is fixed by the stopcock and by the distance *C-D* between the end of the inlet tube and the end of the stopcock. This sampler may also be used with water or salt solution.

8.3.2 After the sample of gas has been collected (stop the flow when the level of the mercury is about the level of *C*) and close stopcocks *A* and *B*; then fill the tube above *A* with mercury from a small funnel whose stem has been drawn out to a capillary. Invert the container and, by means of a rubber tube attached to an aspiratory bottle or levelling bulb reservoir filled with mercury, admit mer-

cury through *A* (using precaution not to entrap air in the connection) and put the gas under a pressure of about 100 mm of mercury, as observed by the height of the level of the mercury in the reservoir and the height of the mercury at *C*. Close stopcock *A* and return the vessel to its original position, as shown in the illustration. The mercury will then be well above the level of *C* and the stopcocks *A* and *B* will be mercury sealed against leaks.

When it is desired to remove the sample for analysis, again invert the container and attach *D* to the buret. By means of a mercury reservoir, mercury may be admitted to displace the gas while filling the buret.

8.4 For collecting continuous samples of several cubic feet (or cubic meters), a metal gas holder as shown in Fig. 11 is recommended. Adjust the pressure regulator outlet and orifice size to permit continuous flow of gas into the holder to completely fill it in the time desired for taking an average sample. Purge the sample line and holder thoroughly with the gas being sampled; then start the sampling period with the holder either completely filled with water, or the small section of the holder crown sheet, which may extend above the water, filled with the same gas as it is intended to sample.

## 9. Procedure for Collecting Samples for the Determination of Suspended Particles

9.1 A sampling tube of the type shown in Fig. 12 is suitable for collecting samples of gas for the determination of suspended particles. These sampling probes should be located ordinarily in the vertical main and at some distance (usually about 15 ft (5 m)) from any bend or obstruction. The probe should be inserted parallel to and against the gas flow so that the open-end faces the approaching stream of solid particles. The result will then be theoretically correct, assuming uniform distribution of solid particles and a velocity in the sampling tube equal to that in the main. Moreover, the sample should be withdrawn from the region of the main where the mean velocity of the gas is found to exist. Although it may be necessary to investigate the distribution of the velocity for each individual case, it is generally accepted that the point of average velocity is approximately one sixth of the diameter from the inside surface of the main.

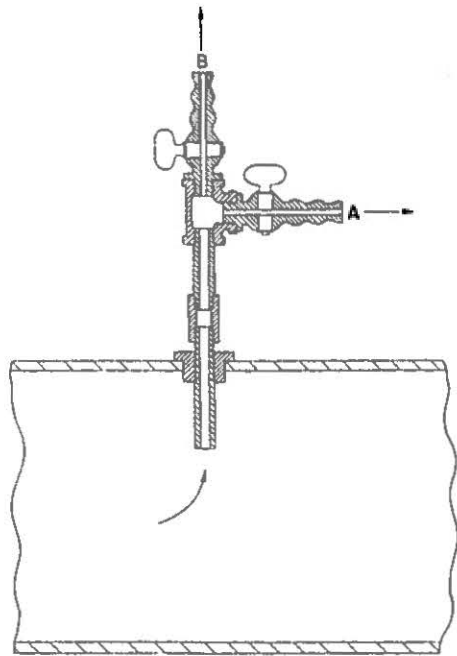


FIG. 1 Simple Open-End Sampling Tube.

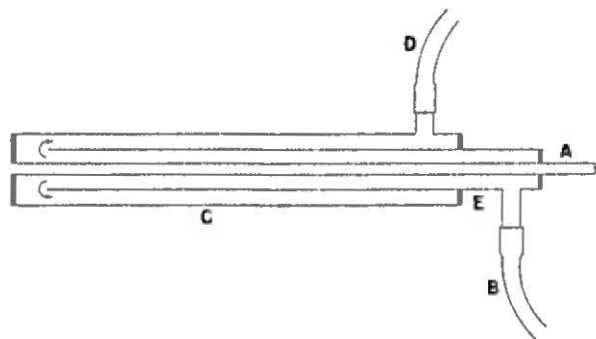


FIG. 2 Water-Cooled Sampling Tube.



Type C



FIG. 4 Metal Gas Sample Container.



Type F

FIG. 3 Glass Gas Sample Containers.