Chapter 2　Triangular odor bag method

1. Equipment and apparatus

1-1 Sampling equipment

1-1-1 Sampling equipment for collecting an ambient air sample

No. 2　Equipment and apparatus
Equipment and apparatus are as given as follows:
1. Sampling equipment
(1) Sampling equipment for collecting an ambient air sample
Equipment is given from “a” to “d,” as in the following.

There is a large variation over time of the offensive odor of the ambient air sample to be collected, so samples should be collected in a short period of time (6 to 30 seconds), at the peak of the variation. In general, the concentration of the collected sample is low, and a large sample amount (more than 10 L) is necessary to carry out the measurement of the triangular odor bag method. Because of this, techniques such as odor sampling method using a vacuum bottle or a suction bottle have been developed.

There are four methods of collecting an ambient air sample: 1. Vacuum bottle method (a method of using a vacuum bottle with an already reduced internal pressure); 2. Suction bottle method (a method using a suction bottle with an already reduced internal pressure and a sampling bag attached to the inside); 3. Direct sampling method (a method of collecting a sample directly in a sampling bag through a pump); and 4. Indirect sampling method (a method of collecting a sample indirectly in a sampling bag by reducing the internal pressure of the vacuum container using a pump).

Methods 1 and 2 can carry out sampling in a short period of time, and have the advantage in collecting samples at the variation peak of the offensive odor. Both of these methods have another advantage of not having to prepare a power supply, because a pump is not used on-site. Various strategies that do not require a power supply, such as using a battery-operated handy pump or stirrup pump, have been developed for methods 3 and 4.

In methods 1 and 2, one bottle is used to collect one sample, in principle. On the other hand, methods 3 and 4 have the advantage of collecting many samples. However, since the sample passes through the inside of a pump in method 3, it is necessary to use a pump in which this part can be replaced.

When selecting a method for collecting samples, it is also necessary to consider the characteristics of each sampling method, including those such as easy operation and maintenance conditions such as washing, and then adopt a suitable method..

1-1-1 (A) Vacuum bottle method

No. 2 Equipment and apparatus

1 Sampling equipment

(1) Sampling equipment for collecting an ambient air sample

a. An airtight sampling container made of glass, with a valve made of fluoride plastic, glass sliding part, and an inner volume of about 10 L.

1) Vacuum bottle

The vacuum bottle, similar to the one shown in Figure 6, should have an inner volume of about 10 L, and should be made of borosilicate glass. One side of the vacuum bottle is equipped with a cock made of polyfluoride plastic, and the other side has a glass sliding part. This bottle can also be made airtight by using an O-ring. A large silicone rubber stopper (No. 30) and a vacuum bottle bag (75 cm long, approx. 30 cm wide) are necessary as the accessories for this vacuum bottle.

These should be used after thoroughly washing with a highly fragrance-free detergent and letting it dry.

2) Vacuum pump, etc.

A vacuum pump is necessary to clear the air out from the inside of the vacuum bottle. It is efficient if the ultimate vacuum achieved is less than 1.3 kPa (10 mmHg).

A vacuum pressure gauge (mercury manometer) which can measure at less than 26 kPa (200 mmHg) is necessary to check the degree of vacuum.

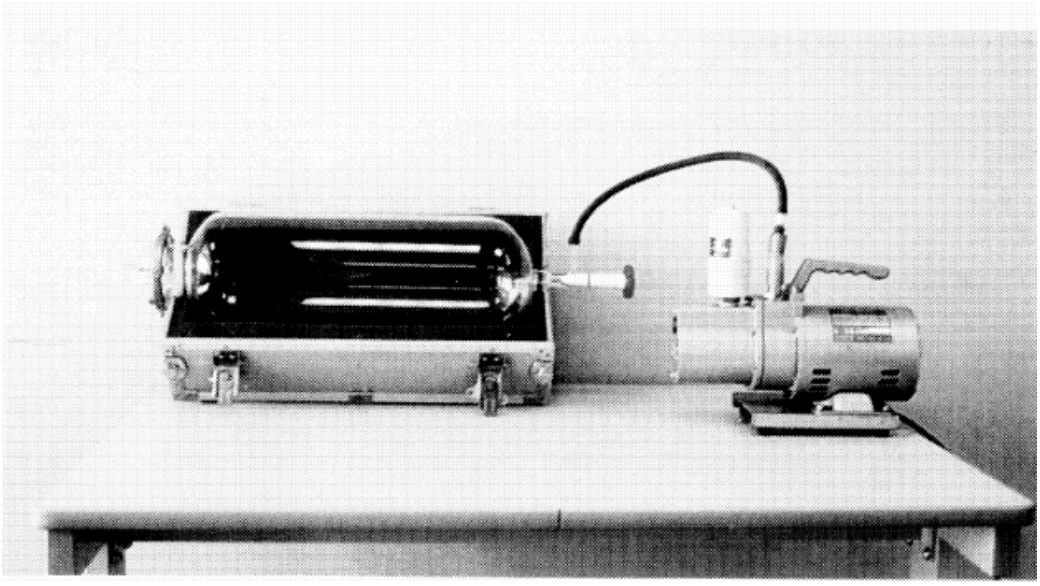


Figure 6: Deaerator of the vacuum bottle

1-1-1 (B) Suction bottle method

No. 2 Equipment and apparatus

1 Sampling equipment

(1) Sampling equipment for collecting an ambient air sample

b. The equipment in which a sampling bag is connected to the inside of the suction bottle. The following requirements should be fulfilled:

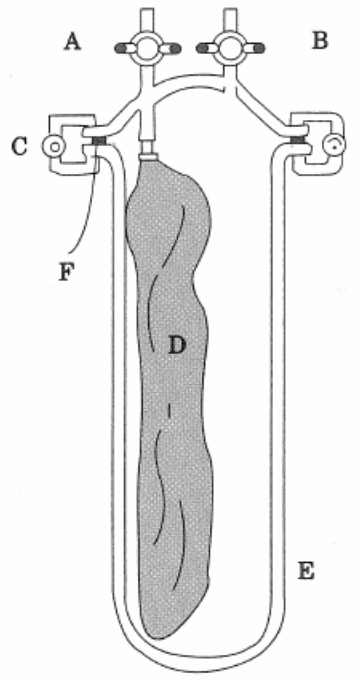
a) The suction equipment for collecting sample gas should have an airtight structure in which the inner sampling bag can be viewed, and have an inner volume of about 10 L.

b) The material of a sampling bag should be polyester (compound name: polyethylene terephthalate) film that is odor-free and has low odor adsorption, or an equivalent or better material with a storing capability. The sampling bag should have an inner volume of about 10 L and should match the shape of the suction equipment for collecting sample gas.

1) Suction bottle for sampling

An airtight bottle that has an inner volume of about 10 L and is made of glass, similar to the one shown in Figure 7, is commonly used. The lid of this bottle is equipped with two cocks made of polyfluoride plastic. Accessories such as O-rings and clamps are necessary to make this bottle airtight.

The requirements of the suction bottle are that enough negative pressure in the bottle can be maintained until taken to the sampling site, and that the inside can be viewed in order to check whether sampling was successful. If these requirements are satisfied, the bottle does not need to be made of glass. Currently, impact-resistant suction bottles made of vinyl chloride are being developed and are commercially available.



A, B: Cock made of Teflon
C: Clamp
D: Sampling bag
E: Suction bottle for sampling
F: O-ring

Figure 7: Sampling equipment using a suction bottle

2) Sampling bag

This sampling bag should have an inner volume of about 10 L and should match the shape of the suction bottle. It is required that the sampling bag be made of polyester or an equivalent or better material that is odor-free and has a storing capability.

Currently, odor-free bags are commercially available, and these bags can be used. Before using a sampling bag, any odors inside the bag should be removed by methods such as filling it with odor-free air through activated carbon and leaving it to stand for several hours, or filling the bag with odor-free air for a given length of time, before confirming that they are odorless.

3）Vacuum pump

The same pump as the vacuum pump given in 2) of 1-1-1 (A) “Vacuum bottle method” should be used.

1-1-1 (C) Direct sampling method

No. 2　Equipment and apparatus

1　Sampling equipment

(1) Sampling equipment for collecting an ambient air sample

c. The equipment that collects a sample in a sampling bag through a sampling pump. The following requirements should be fulfilled.

a) A sampling pump should have an ability to suck air over 20 L/min, and should be odor-free and have low odor adsorption.

b) The material of the sampling bag should be the same as the one specified in No. 2-1-(1)-b-(b), and should have an inner volume of about 10 L.

1) Sampling pump

An ambient air sample has to be collected at a rate of over 10 L within 6 to 30 seconds; the pump used for sampling should have a suction ability of over 20 L/min.

Currently, portable sampling pumps, such as the one shown in Figure 8, are used widely. The part of the sampling pump that the sample gas passes through is a cassette and can be exchanged, which can be convenient when collecting various types of samples. These sampling pumps are also battery operated, so it is not necessary to prepare a power supply.

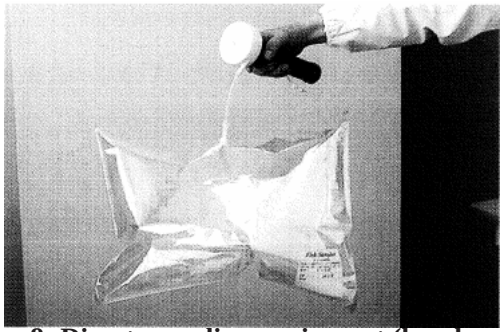


Figure 8: Direct sampling equipment (handy pump)

2) Sampling bag

It is necessary that the sampling bag has an inner volume of about 10 L, and made of polyester or an equivalent or better material that is odor-free and has a storing capability.

Currently, odor-free bags are commercially available, and these bags can be used. The sampling bag should be used after washing it with odor-free air and confirming that it is odorless. For instructions on washing, refer to 2) of 1-1-1 (B), “Suction bottle method.”

3) Tube

An odor-free tube made of polyfluoride plastic is commonly used as the tube to connect the pump to the sampling bag.

1-1-1 (D) Indirect sampling method

No. 2 Equipment and apparatus

1 Sampling equipment

(1) Sampling equipment for collecting an ambient air sample

d. The equipment in which a sampling bag is connected to the inside of a vacuum container with a suction pump. The following requirements should be fulfilled.

a) The vacuum container should have an airtight structure in which the inner sampling bag can be viewed.

b) The suction pump should have an ability to suck air over 20 L/min.

c) The material of the sampling bag should be the same as the one specified in No. 2-1-(1)-b-(b), and should have an inner volume of about 10 L.

1）Vacuum container for sampling

This is a container, similar to the one shown in Figure 9, which should be airtight and made of material that is hard to break.

It is required that one part of the container be transparent, in order to be able to view the internal state of the container, since the sampling bag may burst by sucking an amount of air that exceeds the inner volume of the sampling bag.

Containers that are made of transparent acrylic or vinyl chloride plastic and structured so that the lid can be removed are commonly used. Cubic- shaped containers are also commercially available but the container can be self-made.

A sampling bag can be used regardless of the position of a sampling outlet by placing two =two-way cocks made of polyfluoride plastic on the upper lid of the vacuum container symmetrically, which can be useful.

It is favorable to use column-shaped containers made with vinyl chloride tubes that have a large diameter in order to make a structure that is resistant to negative pressure. Cylindrical vacuum containers that can be

also be used for the suction bottle method are commercially available.
Containers with an inner volume of about 15 to 35 L are commonly used. It is preferable to prepare two sizes of containers in accordance with the sampling volume.

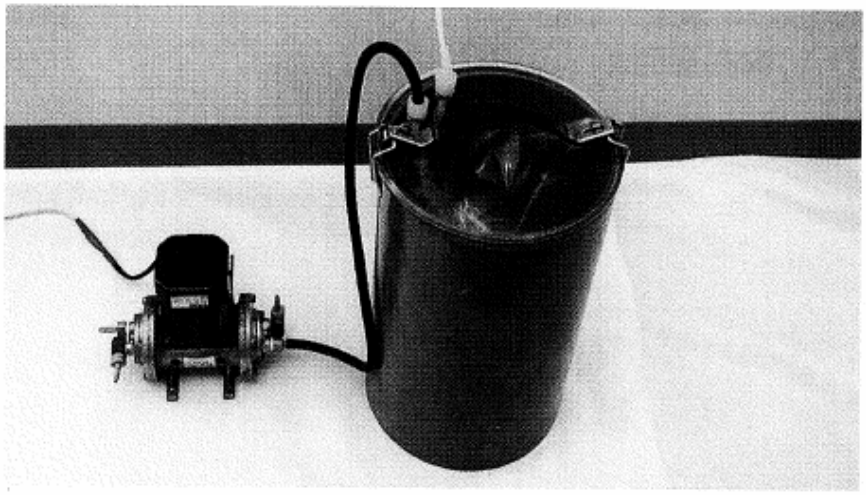


Figure 9: Indirect sampling equipment

2) Sampling pump

It is required that the sampling pump has a flow rate of at least 20 L/min.
The sampling pump used for indirect sampling is different from the one used for direct sampling, and though a sample never passes directly through inside of the pump, using a pump with a generator as a power supply or the vacuum pump that uses oil, the exhaust gas of the pump may have an affect on the sample to be collected. Therefore, a sampling pump that is odor-free and has low odor adsorption, such as a diaphragm pump, should be used.
There is another method of reducing the internal pressure of the container by using a stirrup pump, which is commercially available. The stirrup pump is hand-operated and can suck the required volume in a short period of time without a power supply.

3) Sampling bag, tube, etc.

The sampling bag and tube should be similar to the ones described in 2) and 3) of 1-1-1 (C), “Direct sampling method.”

1-1-2 Sampling equipment for exhaust port

No. 2 Equipment and apparatus

1 Sampling equipment

(2) Sampling equipment for collecting a exhaust port sample

The equipment that can collect a sample in a sampling bag through a sampling pump, or the equipment in which a sampling bag is connected to the inside of a vacuum container with a suction pump. The following requirements should be fulfilled.

(a) The sampling pump should have an ability to suck the sample gas at a rate of at least 4 L/min, and should be odor-free and have low odor adsorption.

(b) The vacuum container should have an airtight structure in which the inner sampling bag can be viewed.

(c) The suction pump should have an ability to suck the sample gas at a rate of at least 4 L/min.

(d) The material of the sampling bag should be the same as the one specified in No. 2-1-1-b-(b), and have an inner volume of about 3 to 20 L.

(e) If the gas temperature is high, heat-resistant material should be used for the sampling probe from the sampling port. If the sample has a high water content, a gas washing bottle with a volume of about 250 mL should be used as a condenser.

Because the concentration of exhaust port sample is comparatively high and has little fluctuations, the requirements for collecting exhaust port sample are less strict than those for collecting an ambient air sample. Consequently, the sampling time and the sampling volume can be selected from the ranges of 1 to 3 min and 3 to 20 L, respectively. When sampling from a tall smoke stack, it is hard to carry out sampling using a large glass bottle. For this reason, a sampling method of using a pump on-site is used.

There are two methods for collecting exhaust port sample: 1. Direct sampling method (a method of collecting a sample directly in a sampling bag through a pump): and 2. Indirect sampling method (a method of collecting a sample indirectly in a sampling bag by reducing the internal pressure of the vacuum container using a pump). The odor sampling methods of using a vacuum bottle and a suction bottle, used for collecting ambient air samples, are not commonly used.

The decision of whether to use the direct sampling method or indirect sampling method should be determined by considering the sampling conditions (such as the temperature and natural properties), as well as the conditions of the sampling place (such as a location of the sampling port and the gas pressure inside the duct).

1-1-2 (A) Direct sampling method

1) Sampling pump

The flow rate of the sampling pump should be at least 4 L/min. The sampling pump, such as the diaphragm pump, should be odor-free and have low odor adsorption. A vacuum pump that uses oil should not be used.

At present, sampling pumps in which the part that the sample gas passes through is a cassette and can be easily exchanged are commercially available, making it convenient to collect many samples. Battery operated pumps are also commercially available.

If the internal pressure of the duct is negative, be careful not to use a pump with weak static pressure because it may not be able to suck the sample sufficiently.

2) Sampling bag

The sampling bag should be odor-free, have low odor adsorption and low permeability, with an inner volume of about 3 to 20 L. Polyester is commonly used as the material for the sampling bag.

3) Tube

A tube made of polyfluoride plastic is commonly used as the tube connecting an exhaust pipe to the pump, and the pump to a sampling bag. However, a silicone rubber tube can also be used if odor concentration is high. If the exhaust gas temperature is low, a sampling probe (refer to Figure 10), which is the tip of the tube, should be used as is. If the exhaust gas temperature is high (over 250 °C), however, a glass tube or a stainless steel tube should be used.

4) Other

A condenser is necessary if the water content of the exhaust gas is high. A gas washing bottle (250 mL) is suitable for this condenser.

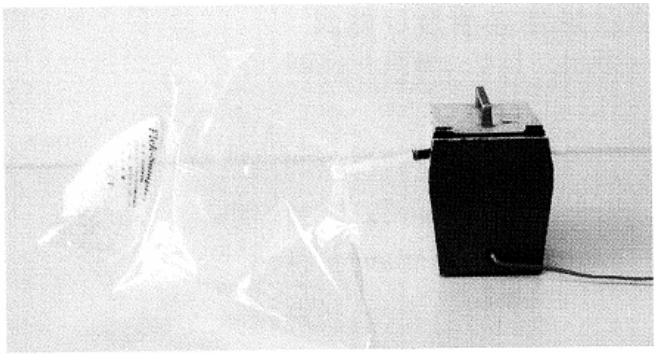


Figure 10: Direct sampling equipment from an outlet port (Diaphragm pump)

1-1-2 (B) Indirect sampling method

1) Sampling pump, etc.

A sampling pump, sampling bag, tube, and condenser similar to those described in the direct sampling method (exhaust port sample) of 1-1-2 (A) should be used.

2) Vacuum container for sampling

A vacuum container similar to the one described in the 1) of 1-1-1 (D), “Indirect sampling method,” should be used. (The volume of the container should be one that can hold the amount of the sample to be collected.)

1-2 Equipment and apparatus for a sensory test

No. 2 Equipment and apparatus

2 Equipment and apparatus for a sensory test

a. Pump for supplying air

A pump that has an ability to supply air at over 30 L/min.

b. Odor-free air distributor

An apparatus that can remove odors from the air to be supplied, as well as from a pump for supplying air, when transferring odor-free air into an odor bag.

c. Syringe

A syringe made of glass. A gastight syringe should be used if the volume is less than 1 mL. A syringe made of plastic that is airtight, odor-free and has low odor adsorption, similar to the syringe made of glass or the gastight syringe, can also be used.

d. Odor bag

An odor bag made of polyester film that is odor-free, has low odor adsorption and low permeability, or made of plastic film that has similar or better capabilities. The odor bag should also be equipped with a glass tube that has an inner diameter of 10 mm and a length of 6 cm as a sample outlet port. The inner volume should be 3 L.

e. Sniffing mask

A sniffing mask that is odor-free and made of plastics, which connects to the outlet of an odor bag and is structured to cover a nose.

f. Silicone rubber stopper

A silicone rubber stopper that should be able to be used as a tight stopper for the outlet port of an odor bag.

Note: The tube used for connecting equipment where the sample passes through should be made of polyvinyl fluoride with low odor adsorption, or of other material with similar or better capabilities.

1) Pump for supplying air

A pump for supplying air that transfers odor-free air into an odor bag and has a flow rate of over 30 L/min. It is necessary for this pump to be odor-free, such as a diaphragm pump.

2) Odor-free air distributor

An apparatus used to remove odors from pumps, or to supply odor-free air into an odor bag, consisting of an activated carbon tank, filter, and distributor, as shown in Figure 11. A distributor with nine cocks (which can prepare nine odor bags at a time) and six cocks are commercially available.

3) Syringe

It is necessary for the syringe, which is used to inject a sample from the sampling bag to the odor bag, to be odor-free. For this reason, a syringe made of glass is commonly used. It has also become possible to use disposable syringes made of plastic, which have become commercially available recently, if they are odor-free. A gastight syringe should be used if the volume is less than 1 mL.

A syringe made of glass should be used after thoroughly washing it with a highly fragrance-free detergent and letting it dry. Regardless of the material, the syringe should also be washed with a sample several times.

Do not use a syringe made of glass in which odor is adsorbed to the glass sliding part.

At the very least, 200 mL, 100 mL, 10 mL, 1 mL, 100μL syringes should be prepared. A 300 mL syringe must be specially ordered from a manufacturer, but it can be useful in preparing three odor bags at a time, and is highly recommended.

It is useful to prepare a standard type (approx. 0.5 mm diameter: 23G) and a large-hole type (approx. 1.0 mm diameter: 18G) needle.

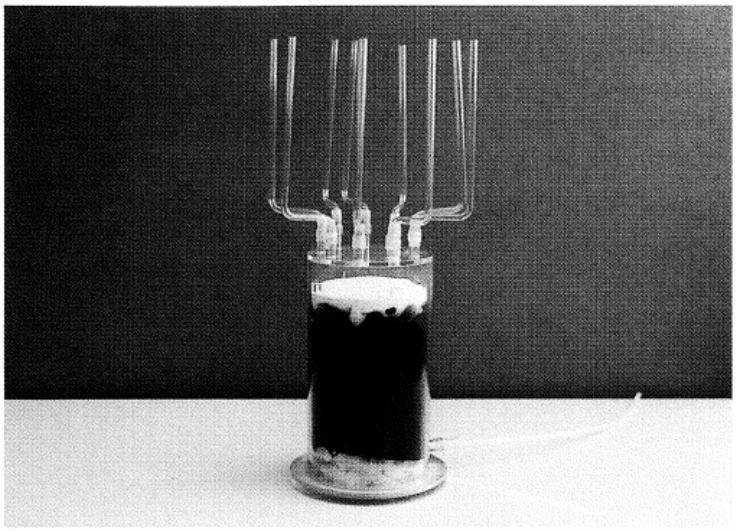


Figure 11: Odor-free air distributor (an example of a distributor with nine cocks)

4) Odor bag

An odor bag equipped with a glass tube with an inner diameter of about 10 mm, an external diameter of about 12 mm, and a length of 6 cm should be odor-free, and have low adsorption and low permeability. Its inner volume should be 3 L. Currently, only polyester is commercially available and used as the material of odor bags. It would be preferable not to use odor bags a year after production, because deterioration and adsorption of odor would occur.

5) Sniffing mask

A sniffing mask should be connected to the glass tube of an odor bag and should be structured to cover a nose. Sniffing masks made of rigid polyvinyl chloride are commercially available.

6) Silicone rubber stopper

The No.03 size is standard and is used as a stopper for the odor bag.

2. Measurement methods

No. 3 Measurement methods

Measurement is carried out according to the following procedures. A person who conducts the following measurement should be recognized as having normal olfaction through the panel screening method as described in No. 1-2, and should also have advanced knowledge and skills for odor index measurement.

A person who conducts measurement (such as for the odor index) by using a panel (hereinafter called the "operator") takes charge of a series of measurement operations, such as implementing an olfactory test for a panel, preparing the samples to be supplied to a panel, controlling the sensory test room, and summarizing test results. Therefore, it is necessary for the operator to be cooperative and independent, and be familiar with the triangular odor bag method. The operator should also have normal olfaction in order to judge the presence of odor inside the sensory test room, and to determine the first concentration step with which to start the sensory test. Therefore, it is necessary that the operator also pass the same olfactory test as the panel.

If private testing agencies are commissioned by local authorities to carry out odor index measurement based on the Offensive Odor Control Law, an operator who engages in odor index measurement related to the commission should be Odor Judgment Technician, in compliance with the provisions of Article 12 of the Offensive Odor Control Law.