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**Measuring method for volatile
organic compounds in flue gas
by analyzers**

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Foreword

This translation has been made based on the original Japanese Industrial Standard established by the Minister of Economy, Trade and Industry, through deliberations at the Japanese Industrial Standards Committee as the result of proposal of establishing a Japanese Industrial Standard by Japan Electric Measuring Instruments Manufacturers' Association (JEMIMA)/Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law.

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Measuring method for volatile organic compounds in flue gas by analyzers

1 Scope

This Japanese Industrial Standard specifies the measuring methods for the concentration of volatile organic compounds (hereafter referred to as “VOC”) in the gas exhausted from various kinds of manufacturing processes and the like in factories and facilities by using an automatic measuring instrument (hereafter referred to as “measuring instrument”). For the gas sampling, the collecting bag shall be used and the measurement principle of measuring instrument shall be based on the catalytic oxidation–non-dispersive infrared absorption method and the hydrogen flame ionization detection method.

NOTE 1 The VOC, as given in this Standard, generically refers to the organic compounds at the exhaust port which are not specified to be one of the excluded substances by the Enforcement Regulation of the Air Pollution Control Law.

NOTE 2 The measuring method for VOC concentration where the sample gas is taken by using the collecting bag is notified as the Notification No. 61 of Ministry of Environment (June 10, 2005) (see Annex B and Annex C).

NOTE 3 The handling and installation of high pressure gas cylinder used for measurement shall be in accordance with the High Pressure Gas Safety Law.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS C 1302 *Insulation resistance testers*

JIS K 0055 *General rules for calibration method of gas analyzer*

JIS K 0095 *Methods for sampling of flue gas*

JIS K 0151 *Non-dispersive infrared gas analyzer*

JIS K 0211 *Technical terms for analytical chemistry (General part)*

JIS K 0212 *Technical terms for analytical chemistry (optical part)*

JIS K 0213 *Technical terms for analytical chemistry (Electrochemistry part)*

JIS K 0215 *Technical terms for analytical chemistry (analytical instrument part)*

JIS Z 8103 *Glossary of terms used in measurement*

3 Terms and definitions

For the purposes of this Standard, the terms and definitions in **JIS K 0211**, **JIS K 0212**, **JIS K 0213**, **JIS K 0215** and **JIS Z 8103** and the following apply.

3.1 sample gas

the flue gas which is taken in the collecting bag from exhaust port

3.2 zero gas

the gas to be used for the correction of the minimum scale value of measuring instrument

3.3 span gas

the gas to be used for the correction of the maximum scale value of measuring instrument

3.4 catalytic oxidation–non-dispersive infrared absorption system

the system in which VOC is oxidized into carbon dioxide by using heated catalyst and its concentration is measured from the intensity of infrared absorption, referred to as “NDIR” in this Standard

Moreover, the non-dispersive type is the name of type of measuring instrument in which the optical filter or the detector having wavelength selectivity is used and which does not have the optical system carrying out wavelength dispersion.

3.5 flame ionization detection system

the system in which the ionic current generated when the sample gas is added to hydrogen flame is measured and the concentration of VOC is measured

It is called “FID” in this Standard.

3.6 repeatability

the degree to which the individual measured values agree when the same measurement object gas is repeatedly measured by using the same measuring instrument by the same method under the same conditions for a comparatively short time

3.7 zero drift

the variation, for a certain period, of the indicated value corresponding to the minimum scale of measuring instrument

3.8 span drift

the variation, for a certain period, of the indicated value corresponding to the maximum scale of measuring instrument

3.9 sensitivity

the percentage of the indicated value obtained when the gas containing the mass-known VOC is measured by using the measuring instrument corrected by using propane to the rated concentration (analyzed value)

3.10 set flow rate

the flow rate of sample gas, corrected gas and the like specified for the measuring instrument

3.11 volppmC

the unit of VOC concentration expressed by the number of carbon atoms as the standard

3.12 indication error

the percentage of the difference between the value indicated when the midway point gas is introduced and its concentration to the maximum scale value

3.13 response time

duration consisting of the response lag time and the rise time, required for the instrument indication to reach 90 % of the final indication value from the introduction of the test gas

3.14 minimum detection limit

the percentage of the value of doubled standard deviation of the indicated value at the time of introduction of the gas for zero test to the maximum scale

4 Outline of measuring method

It is the method in which, after the flue gas is taken by suction from exhaust port in a container, the sample gas in the container is introduced into a measuring instrument and the VOC concentration is measured. The collecting bag shall be used for a container. When the VOC concentration in sample gas exceeds the measurement range of measuring instrument, the gas is diluted so as to be the concentration within the measurement range and then measured. An example of the flow chart of this measurement is as shown in figure 1.

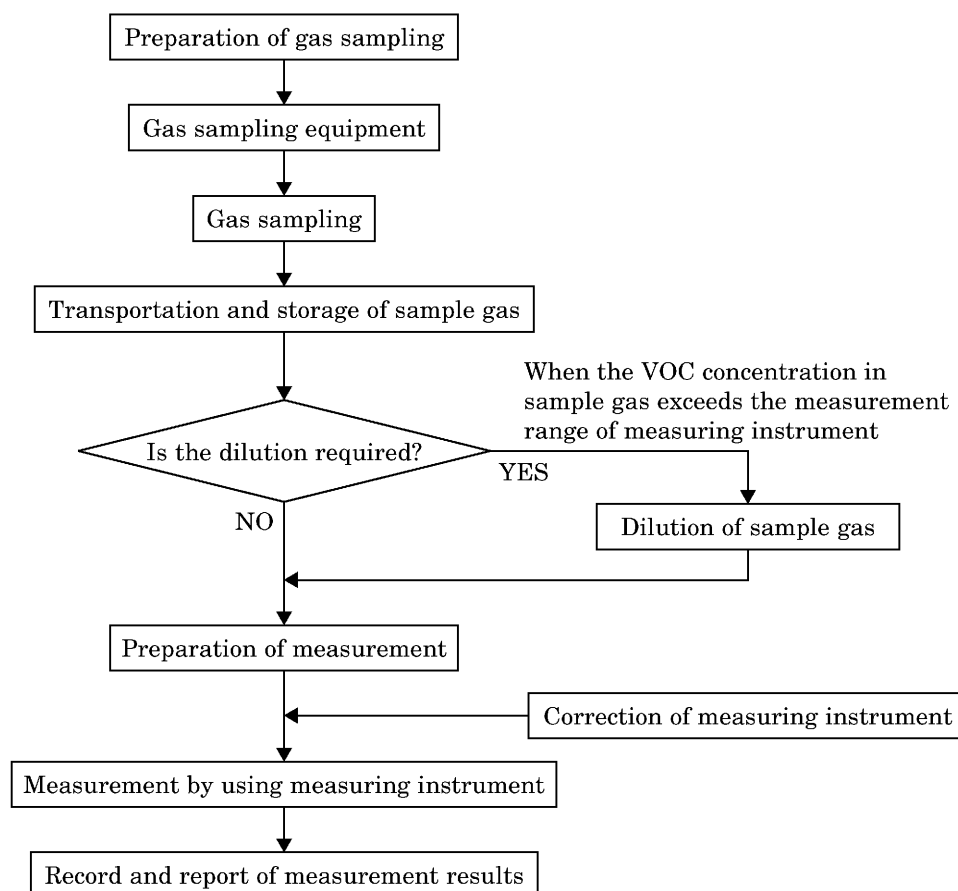


Figure 1 Example of flow chart for measurement of VOC concentration in flue gas

5 Gas sampling

5.1 Outline of gas sampling

Prior to the actual sampling, matters such as the sampling condition under which the representative VOC exhaust concentration of the facility can be determined, the necessity of the protective measures for explosion at the sampling, and the substances which are to be excluded from the VOC, etc. shall be investigated. Twenty liters or over of flue gas shall be taken by using the collecting bag, to be used as the sample gas. The sample gas shall be measured within 8 h after sampling and even if it is difficult, it shall be carried out within 24 h.

The sampling method for sample gas shall be in accordance with **JIS K 0095**.

5.2 Preparation of gas sampling

The following items shall be included in the advance preparation.

- a) **Exhaust port for gas sampling** When some exhaust ports are present in one facility, it shall be in accordance with either of the following.
- 1) When the flue gas system of facility is investigated and the exhaust port exhausting the highest concentration of VOC can be specified, the gas sampling is carried out at that exhaust port.
 - 2) The gas sampling is carried out at every exhaust port.

However, when the concentration of VOC can be represented by some exhaust ports because of the structure of the facility and the like, the gas sampling is carried out at those exhaust ports.

- b) **Time of gas sampling** The gas sampling is carried out at the period in which the exhaust of VOC is confirmed to stabilize in one process, based on the investigation of the operating conditions of the facility. In the case of gas sampling in storage tank, the sampling time shall be the injecting time of VOC.

Where a flue gas treatment equipment is installed, the sampling shall be carried out avoiding the time when the high concentration of VOC is temporarily exhausted, such as the operation starting time of the treatment equipment.

- c) **Explosion-proof measure** It is confirmed whether the explosion-proof measure is required for the mechanical equipment and materials to be used for sampling of exhaust gas or not. And, for the sample gas taken, it is confirmed whether the explosion-proof measure is required for its transportation and storage or not.

5.3 Gas sampling equipment

The sampling equipment for sample gas, as exemplified in figure 2 and figure 2 e) of **JIS K 0095**, consist of sampling tube, conduit, filter, drain pot, stop valve, air-tight container, collecting bag, drying tube, flow rate control valve, suction pump, flowmeter and the like.

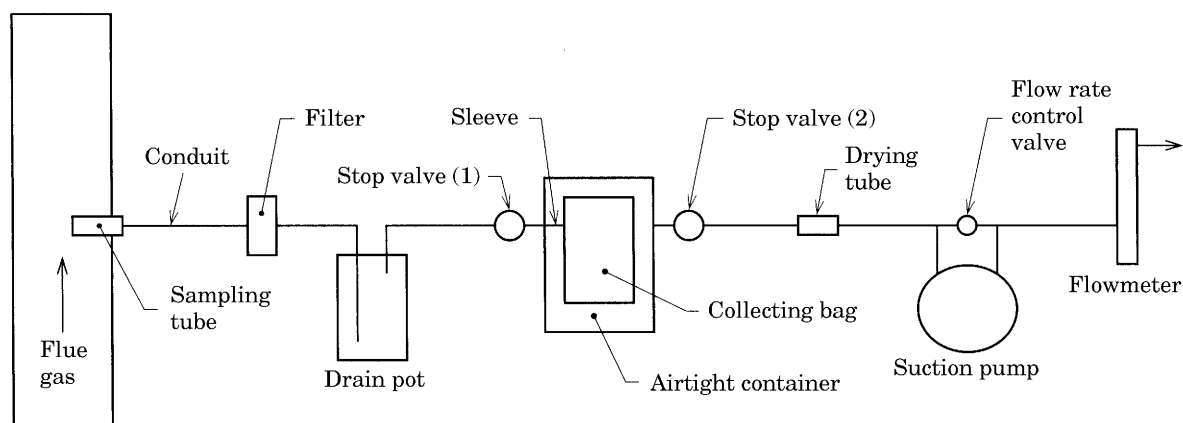


Figure 2 Example of flue gas sampling equipment

- a) **Sampling tube** It is the tube inserted into a sampling opening for taking flue gas, which has the sufficient heat resistance and mechanical strength to bear the temperature and the flow rate of the flue gas and is made of materials which will not cause the adsorption of VOC in sample or deterioration, for example, fluorocarbon resin, silica glass, etc.
- b) **Conduit** It is the tube for connecting a sampling tube to a sampling equipment, which is made of materials not causing the adsorption of VOC or deterioration, for example, fluorocarbon resin, silica glass, etc. The conduit shall be as short as possible and, in addition, its inside diameter shall be such that the contact time to flue gas is minimal, in consideration of the capability of the suction pump.
- c) **Filter** It is a filter removing dust and mist during sampling of flue gas, and made of materials which are good in removal efficiency, small in pressure loss and which do not produce the adsorption of VOC or deterioration, for example, fluorocarbon resin.
- d) **Drain pot** It is a pot for carrying out dehumidification so that the moisture in sample is not condensed in the inside of the collecting bag, and is used as required.
- e) **Stop valve (1)** It is a valve used for opening and closing the flow path of conduit and the flow path of airtight container, made of materials of little adsorption of VOC, for example, fluorocarbon resin, stainless steel.
- f) **Stop valve (2)** It is a valve used for opening and closing the airtight container and the flow path of suction pump.
- g) **Drying tube** It is a tube to protect a suction pump, for which desiccant such as silica gel, etc. are used.
- h) **Airtight container** It is a container to take the sample gas into a collecting bag by decompressing the container and is constructed such that a collecting bag 20 L or over is capacity can be placed, airtightness is kept and the collecting bag in the container is visible from outside.

- i) **Collecting bag** It is the bag which is made of fluorocarbon resin film or polyester resin film, does not cause the adsorption, permeation of VOC and deterioration or has the performance at least equal to it, is 20 L or over in inside capacity attached with a plastic sleeve.

Moreover, the collecting bag shall not be reused.

- j) **Suction pump** It is the pump to decompress the airtight container. An electric pump is used. When the explosion-proof measures are required, a manual pump or an explosion-proof electric pump is used.
- k) **Flow rate control valve** It is the valve capable of controlling the flow rate of gas sampling at 0.5 L/min to 5 L/min.
- l) **Flowmeter** It is a device to monitor the flow rate of gas sampling into a collecting bag, capable of measuring the flow rate of 0.5 L/min to 5 L/min.

5.4 Gas sampling

The gas sampling shall be in accordance with the following procedures.

- a) **Exhaust port for measurement** The exhaust port selected in **5.2 a)** shall be the exhaust port for measurement.
- b) **Sampling position** The sampling position at the exhaust port shall be as specified in **JIS K 0095**. However, the sampling position of storage facility shall be at a vent hole.
- c) **Attachment of sampling tube and conduit** It shall be in accordance with the method specified in **JIS K 0095**. However, when the attachment of sampling tube to the duct for exhaust is difficult, the conduit is directly inserted into the duct.
- d) **Timing and duration of sampling** The sampling shall be carried out for 20 min at the time of sampling confirmed in **5.2 b)**. When the exhaust time of one process is shorter than 20 min, the sampling duration shall be the time of one process.
- e) **Gas sampling** When the equipment exemplified in figure 2 e) of **JIS K 0095** is used, it shall be in accordance with **7.6.2** and **7.6.5** of **JIS K 0095**.

When the equipment exemplified in figure 2 is used, it shall be as follows.

- 1) Place a collecting bag in an airtight container and connect a sleeve to the outlet tube of stop valve (1).
- 2) Detach the suction pump at the outlet of stop valve (2), and connect it to the inlet tube of stop valve (1).
- 3) Open stop valve (1) to operate the suction pump, and after deairing the inside of collecting bag, close stop valve (1).
- 4) Replace to connect stop valve (1) and suction pump as shown in figure 2, and open stop valve (1) and stop valve (2) to operate the suction pump, so that a small amount of flue gas is taken into the collecting bag.
- 5) Repeat several times the operations of **2)** to **4)** alternately, and replace sufficiently the inside of piping and the inside of collecting bag with flue gas.

Next, operate the flow rate control valve of suction pump and take flue gas into the collecting bag at a definite flow rate.

- 6) Close stop valve (1) and stop the suction pump. Take out the collecting bag from the airtight container and stopper the sleeve with silicone rubber stopper.
- f) During transportation and storage, the collecting bag shall be shielded from light and maintained at room temperature.

5.5 Record of gas sampling

The following items shall be recorded. The photograph shall be taken at the job site as required.

- a) Date and hour of sampling
- b) Designation of facility, type of facility, type of main VOC to be used, the type of excluded substance when used
- c) Circumstances of sampling place: schematic diagram, sampling position, capacity of exhauster and blower, the existence of VOC processing equipment and processing system, flue gas temperature, etc.
- d) Operational circumstances of facility
- e) Sampling conditions: constitution of sampling equipment, suction time, amount of suction gas, etc.

5.6 Dilution of sample gas

When the concentration of sample gas exceeds the measurement concentration range of measuring instrument, the dilution of sample gas is used.

- a) **Dilution equipment** A part of sample gas is taken from the collecting bag by using a syringe, etc., and injected into the collecting bag containing a definite amount of highly purified air. It is then diluted so that the concentration is within the measurement range of measuring instrument. The introduction of the highly purified air for dilution into the collecting bag shall be carried out by using the dilution equipment exemplified in figure 3. The dilution equipment consists of a zero gas purification equipment, a zero gas flow rate control valve, an integrating flowmeter and the like.

- 1) **Zero gas purification equipment** It is the equipment to generate the purified air for the dilution of sample gas, which removes the VOC in atmosphere by combustion or similar procedure and is capable of making the VOC content 1 volppmC or under.

For the purified air, the highly purified air or synthetic air filled in a high pressure cylinder may be used.

- 2) **Zero gas flow rate control valve** It is the valve to regulate the purified air for dilution at the specified flow rate. A needle valve of the material of less VOC release and the like is used.
- 3) **Integrating flowmeter** It is the device to measure the amount of purified air for dilution, and the dry gas meter of 1 L per one rotation, in which

no release of VOC is confirmed, is used. The dry gas meter shall be used after the correction of flow rate is carried out by using a wet gas meter.

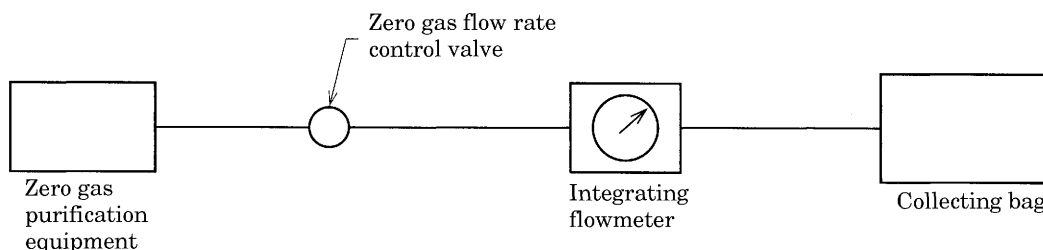


Figure 3 Example of constitution of dilution equipment

b) **Procedures of dilution** The dilution of sample gas shall be carried out in accordance with the following procedures.

- 1) **Calculation of dilution rate of sample gas** Calculate the dilution rate so as to include in the measurement concentration range of measuring instrument, and obtain the taken amount of the sample gas to be diluted and the purified air for dilution.

Obtain the dilution rate according to the following formula.

$$D = (S + A) / S$$

where, D : dilution rate

S : amount of gas sampling (L)

A : injection amount of purified air for dilution (L)

- 2) **Pre-treatment of dilution equipment** Operate the zero gas purification equipment and replace the inside of equipment with purified air.
- 3) **Pre-treatment of collecting bag for dilution of sample gas** After filling about a half of the amount of purified air for dilution obtained in 1) into the collecting bag by using the dilution equipment, take an aliquot of a half of sampling amount of sample gas obtained in 1) by a syringe and inject it into the collecting bag. Stopper the sleeve with a silicone rubber stopper, and after leaving it to stand for several minutes, deair it by using a suction pump and the like.
- 4) **Preparation of diluted sample gas** After filling the amount of purified air for dilution obtained in 1) in the deaired collecting bag in 3) by using the dilution equipment, take an aliquot of taken amount of sample gas obtained in 1) by using a syringe to inject into the collecting bag and stopper the sleeve with a silicone rubber stopper.

6 Measuring instrument

6.1 Type and measurement range of measuring instrument

The type (classification by measurement system) of measuring instrument and its measurement range (hereafter referred to as “range”) shall be in accordance with table 1.