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**Methods for determination of
oxygen in flue gas**

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by Japan Environmental Measurement and Chemical Analysis Association (JEMCA)/Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14.

Consequently **JIS K 0301**:1998 is replaced with this Standard.

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Methods for determination of oxygen in flue gas

1 Scope

This Japanese Industrial Standard specifies the methods for determination of oxygen in flue gas, which is generated from the combustion of fuel and waste, metal refining, chemical reaction processes, etc. and is exhausted to flue, chimney, duct, etc. (hereafter referred to as “duct”)

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) listed below shall be applied.

- JIS B 7983 *Continuous analyzers for oxygen in flue gas*
- JIS K 0050 *General rules for chemical analysis*
- JIS K 0055 *General rules for calibration method of gas analyzer*
- JIS K 0095 *Methods for sampling of flue gas*
- JIS K 0114 *General rules for gas chromatography*
- JIS K 0211 *Technical terms for analytical chemistry (General part)*
- JIS K 0214 *Technical terms for analytical chemistry (Chromatography part)*
- JIS K 0215 *Technical terms for analytical chemistry (Analytical instrument part)*
- JIS K 0216 *Technical terms for analytical chemistry (Environmental part)*
- JIS K 0512 *Hydrogen*
- JIS K 0557 *Water used for industrial water and wastewater analysis*
- JIS K 1105 *Argon*
- JIS K 1107 *Nitrogen*
- JIS K 8150 *Sodium chloride (Reagent)*
- JIS K 8180 *Hydrochloric acid (Reagent)*
- JIS K 8574 *Potassium hydroxide (Reagent)*
- JIS K 8780 *Pyrogallol (Reagent)*
- JIS K 8893 *Methyl orange (Reagent)*
- JIS Z 8401 *Guide to the rounding of numbers*

3 Terms and definitions

For the purpose of this Standard, the terms and definitions given in **JIS K 0211**, **JIS K 0214**, **JIS K 0215** and **JIS K 0216** apply.

4 General matters

General matters shall be as follows.

- a) General matters common to chemical analysis shall be as specified in **JIS K 0050**.
- b) General matters common to sampling of flue gas shall be as specified in **JIS K 0095**.
- c) General matters common to gas chromatography shall be as specified in **JIS K 0114**.
- d) The standard gases whose traceability to the International System of Units (SI) has been guaranteed shall be used.

NOTE : The standard gases whose traceability has been guaranteed are accompanied by the jcss or JCSS calibration certificate based on the relevant national measurement standard (Article 136 or Article 144 of the Measurement Act).

- e) The apparatus and instruments satisfying the specified functions shall be used.
- f) The flue gas, etc. used for the analysis of oxygen shall be handled properly.
- g) Water of Class A2 or A3 specified in **JIS K 0557** shall be used.

NOTE : The analysis using a gas chromatograph is gas chromatography.

5 Classification and outline of analysis methods

The classification and outline of analysis methods are as given in Table 1.

Table 1 Classification and outline of analysis methods

Classification of analysis methods	Outline of analysis method			Applicable conditions
	Principle	Sampling	Determination range	
Gas absorption method (Orsat type) ^{a)}	Oxygen in sample gas is absorbed in oxygen absorbent, and oxygen concentration in sample gas is obtained from the reduced amount in volume of sample gas.	Direct sampling	0.2 vol % to 25 vol % ^{b)}	See 7.1.
		Sampling bag	0.2 vol % to 25 vol %	
Gas chromatography	Concentration of oxygen is obtained by absolute calibration curve method using gas chromatograph equipped with thermal conductivity detector (TCD) or photo ionization detector (PID) ^{c)} .	According to syringe method, sampling bag method or sampling bottle method. Amount of sample gas: about 100 ml to 10 L	TCD: 0.1 vol % to 20 vol % PID: 0.1 vol % to 20 vol % ^{d)}	See 7.2.
Automatic measuring method	a) Magnetic type b) Electrochemical type	Sampling bag method or continuous measurement	0 vol % to 5 vol % 0 vol % to 10 vol % 0 vol % to 25 vol %	See clause 8.
NOTE 1 When an approximate value of concentration is simply required, the detector tube method specified in Annex A may be used if the measurement conditions are met.				
NOTE 2 For the gas absorption method (Hempel type), see Annex B.				
Notes ^{a)} The oxygen absorbent also absorbs the carbon dioxide contained in the sample gas; therefore, it is necessary to remove the carbon dioxide by using the carbon dioxide absorbent before measuring oxygen. The reduced amount in volume of sample gas shall be measured.				
^{b)} Total volume of oxygen and carbon dioxide.				
^{c)} As the discharge type photoionization detector, helium plasma photoionization detector (HPID), barrier photo discharge ionization detector (BID), etc. are available. The lamp-type photoionization detector shall not be used.				
^{d)} In the case of high concentration, the appropriate measures such as reducing the amount of gas sample and diluting the gas sample shall be taken.				

6 Sampling method of sample gas

6.1 Sampling position

The sampling position shall be selected from among points where the typical gas can be collected, for example from the position where uniform gas velocity distribution is obtained.

6.2 Classification of sampling methods

The sampling methods are as given in Table 1.

6.3 Sampling apparatus and instruments

The sampling apparatus and instruments shall have the features and meet the requirements as described below.

- a) The sampling tube made of the material which is not corroded by the coexisting component in flue gas, such as borosilicate glass tube, stainless steel tube, quartz glass tube and tetrafluoroethylene resin tube shall be used.
- b) To prevent ingress of dust into the sample gas, a filtering material shall be packed at the tip of the sampling tube. When this is impracticable, it shall be packed at a suitable position.
- c) When moisture could be condensed in tubes, the sampling tube shall be heated to 120 °C or higher and a condensate trap shall be provided in the middle of the conduit. In this case, for preventing the bubbling of sample gas in the gathered condensate, the trap having a large capacity shall be used.
- d) For the connection of tubes at a heating part, a commonly ground connecting tube, silicone rubber tube or tetrafluoroethylene resin tube shall be used.

6.4 Construction of sampling apparatus and sampling operation

6.4.1 Gas absorption method (Orsat type)

The sampling tube shall be installed as shown in Figure 1, and the sample gas sucked by a suction pump or twin bulbs for spray shall be sent directly to the analyzer¹⁾.

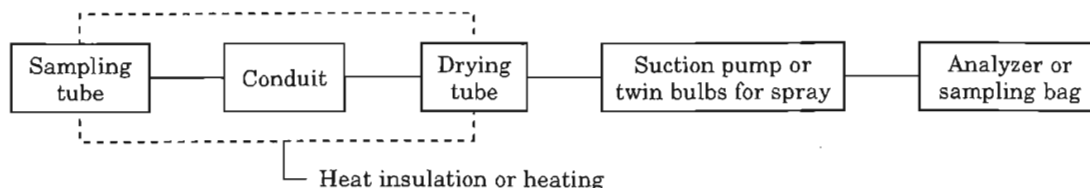


Figure 1 Sampling method of sample gas

Note ¹⁾ If the resistance of the filtering material packed in the sampling tube is large or the pressure in the duct is negative, care should be taken to the leak-in of air.

6.4.2 Gas chromatography

6.4.2.1 Sampling apparatus and instruments

Examples of the sampling apparatus are shown in Figure 2, Figure 3 and Figure 4.

6.4.2.2 Container of sample gas

The following containers of sample gas are used.

- a) **Syringe**, a syringe type sampler exemplified in Figure 2 as H shall be used, and the sample gas shall be introduced by pulling back the inner cylinder. The ground joint of syringe shall be modestly greased, as needed.
- b) **Sampling bag**, made of tetrafluoroethylene resin, polypropylene, etc. having lower gas permeability of not less than 1 L in inner capacity used for the gas sampling. No leakage from a hole, etc. shall be checked before use.

- c) **Sampling bottle**, the glass container to collect the sample gas in a gaseous state. The flow-through type sampling bottle with two stopcocks (300 ml to 500 ml) or the vacuum type sampling bottle with one stopcock (100 ml to 1 000 ml) shall be used.

6.4.2.3 Sampling operation

The sampling operation shall be one of the following.

- a) **Syringe method** The sampling operation by the syringe method shall be as follows.

The symbols of the apparatus herein are as shown in Figure 2.

- 1) Open the three-way stopcock for flow path switch (G) in the horizontal direction, and connect to the pump.
- 2) Connect the syringe (H) from which a needle has been removed to the three-way stopcock for flow path switch (G) by a rubber tube.
- 3) Open the three-way stopcock for flow path switch (G) in all the directions (horizontal and upward directions).
- 4) Repeat the pull-back/push-out operation of the syringe (H) several times to sufficiently purge the conduit, trap (F) and syringe with the sample gas.
- 5) Collect the sample gas into the syringe (H)²⁾. During sampling, it should be constantly checked that the pointer or the counter of the gas meter (K) is in operation.
- 6) Close the three-way stopcock for flow path switch (G) and remove the syringe (H). Quickly attach the needle to the syringe (H) and cover the needle tip with a rubber piece.

Note ²⁾ When the inside pressure of flue is negative, care should be taken to the failure of the collection of sample gas into the syringe.

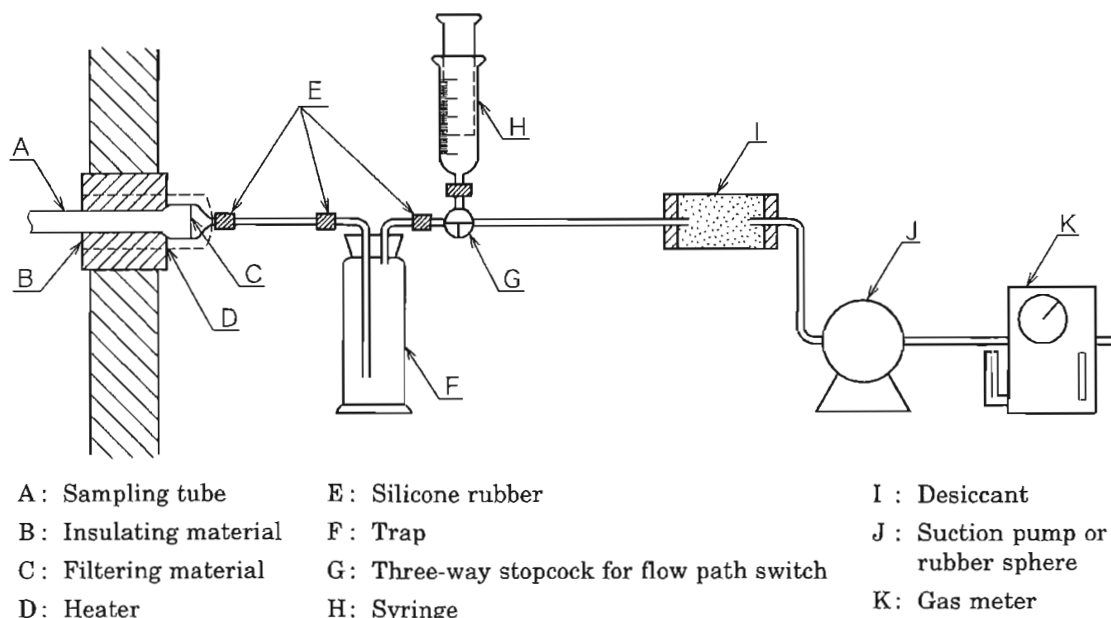
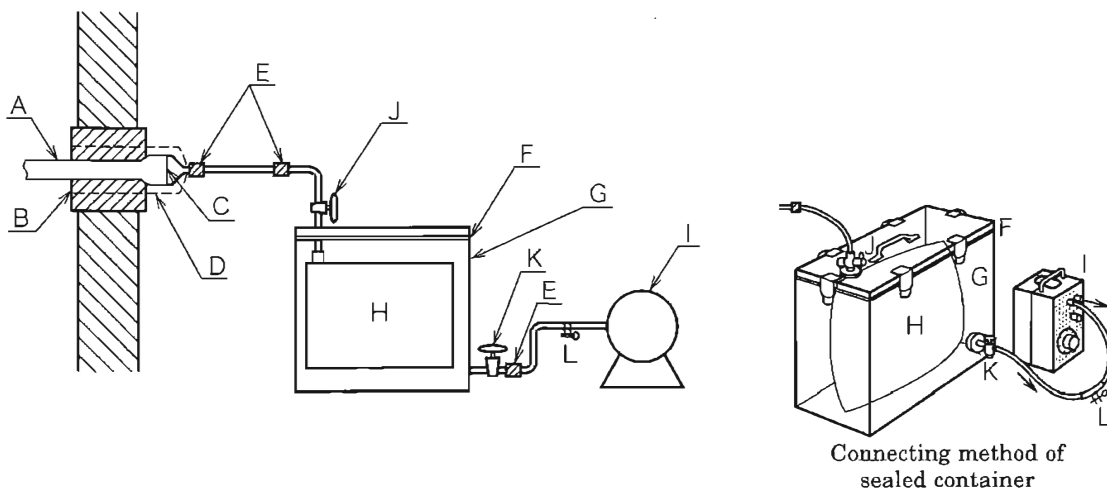


Figure 2 Example of sampling apparatus (syringe method)

- b) **Sampling bag method** The sampling operation by the sampling bag method shall be as follows.

The symbols of the apparatus herein are as shown in Figure 3.

- 1) Connect the sampling bag (H) with the suction pump (I), deaerate the inside of sampling bag (H) and stopper it.
- 2) Connect the conduit from the sampling tube (A) to the stopcock (J) as shown in Figure 3, connect it with the suction pump (I), purge the sampling tube inside with the sample gas and close the stopcock (J).
- 3) Put the sampling bag (H) in the sealed container (G), connect it to the stopcock (J) and close the lid of sealed container tightly. Connect the suction pump (I) as shown in Figure 3, open the stopcocks (J and K), operate the suction pump (I), reduce the inside pressure of sealed container (G) and collect the sample gas into the sampling bag (H).
- 4) Close the stopcock (J), stop the suction pump (I), take out the sampling bag (H) from the sealed container (G), and stopper it with a silicone rubber plug.



A: Sampling tube	E: Silicone rubber	I : Suction pump
B: Insulating material	F: Rubber packing	J : Stopcock made of polytetrafluoroethylene resin
C: Filtering material	G: Sealed container made of methacrylic resin	K: Stopcock
D: Heater	H: Sampling bag	L: Screw cock

Figure 3 Example of sampling apparatus (sampling bag method)

- c) **Sampling bottle method** In the gas sampling bottle exemplified in Figure 4, pass the sample gas of 10 times or more the bottle capacity and collect the sample gas. Alternatively, make the bottle vacuum by using a vacuum pump, and introduce the sample gas in the sampling bottle. In these cases, the sampling tube shall be sufficiently purged with the sample gas beforehand.