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**Methods for determination of
hydrogen sulfide 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 The Japan Society For Analytical Chemistry (JSAC)/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 0108:1983** is replaced with this Standard.

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Methods for determination of hydrogen sulfide in flue gas

1 Scope

This Japanese Industrial Standard specifies the methods for determination of hydrogen sulfide in flue gas which is generated by combustion or chemical reaction or the like, and exhausted into flue, chimney, duct, etc.

Warning: Persons performing analysis based on this Standard should be familiar with normal laboratory practice. This Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. For standards with the year indication, only the editions of the indicated year shall be applied and the revisions (including amendments) made thereafter shall not be applied. For those without the indication of the year, the most recent edition (including amendments) shall be applied.

- JIS K 0050:2005 *General rules for chemical analysis*
- JIS K 0095 *Methods for sampling of flue gas*
- JIS K 0114 *General rules for gas chromatographic analysis*
- JIS K 0115 *General rules for molecular absorptiometric analysis*
- JIS K 0122 *General rules for ion selective electrode method*
- 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 0214 *Technical terms for analytical chemistry (Chromatography part)*
- JIS K 0215 *Technical terms for analytical chemistry (Analytical instrument part)*
- JIS K 0512 *Hydrogen*
- JIS K 0557 *Water used for industrial water and wastewater analysis*
- JIS K 1101 *Oxygen*
- JIS K 1105 *Argon*
- JIS K 1107 *Nitrogen*
- JIS K 8005 *Reference materials for volumetric analysis*
- JIS K 8107 *Disodium dihydrogen ethylenediamine tetraacetate dihydrate (Reagent)*
- JIS K 8142 *Iron (III) chloride hexahydrate (Reagent)*
- JIS K 8180 *Hydrochloric acid (Reagent)*

JIS K 8193	<i>N,N-Dimethyl-para-phenylenediammonium dichloride (Reagent)</i>
JIS K 8295	<i>Glycerin (Reagent)</i>
JIS K 8576	<i>Sodium hydroxide (Reagent)</i>
JIS K 8625	<i>Sodium carbonate (Reagent)</i>
JIS K 8637	<i>Sodium thiosulfate pentahydrate (Reagent)</i>
JIS K 8659	<i>Starch, soluble (Reagent)</i>
JIS K 8913	<i>Potassium iodide (Reagent)</i>
JIS K 8920	<i>Iodine (Reagent)</i>
JIS K 8949	<i>Sodium sulfide nonahydrate (Reagent)</i>
JIS K 8951	<i>Sulfuric acid (Reagent)</i>
JIS K 8953	<i>Zinc sulfate heptahydrate (Reagent)</i>
JIS K 8960	<i>Ammonium sulfate (Reagent)</i>
JIS K 9005	<i>Phosphoric acid (Reagent)</i>
JIS K 9502	<i>L(+)-Ascorbic acid (Reagent)</i>
JIS Z 8401	<i>Guide to the rounding of numbers</i>
JIS Z 8808	<i>Methods of measuring dust concentration in flue gas</i>

3 Terms and definitions

For the purpose of this Standard, the terms and definitions given in **JIS K 0211**, **JIS K 0212**, **JIS K 0213**, **JIS K 0214** and **JIS K 0215** apply.

4 General matters

The general matters shall be as follows.

- a) General matters of chemical analysis shall be in accordance with **JIS K 0050:2005**.
- b) General matters of flue gas sampling shall be in accordance with **JIS K 0095**.
- c) General matters of gas chromatograph shall be in accordance with **JIS K 0114**.
- d) General matters of absorptiometry shall be in accordance with **JIS K 0115**.
- e) General matters of ion selective electrode method shall be in accordance with **JIS K 0122**.
- f) Water to be used in analysis shall be of A3 or A4 among the classification and qualities as specified in clause 4 of **JIS K 0557** or Annex 1 of **JIS K 0050:2005**, or any water equivalent to these.
- g) Reagents to be used shall be of the highest grade or of an appropriate quality as specified in a relevant Japanese Industrial Standard, if applicable. If the reagent does not have a relevant specification in Japanese Industrial Standard, that of a quality adequate for the analysis shall be used.
- h) Apparatus and instrument to be used shall be provided with the specified functions.

- i) When disposing flue gas, or absorbing solution of the flue gas used for the analysis of hydrogen sulfide, it shall be done with utmost care.

5 Types and summaries of analysis methods

The types and summaries of analysis methods are as shown in table 1.

Table 1 Type and summary of analysis methods^{a)}

Analysis method	Summary of analysis method			
	Outline	Sampling	Determination range ^{b)}	Sampling of sample gas
Gas chromatography (see clause 8)	Flue gas is introduced into gas chromatograph and separated by packed column or capillary column, then hydrogen sulfide is determined from the chromatogram obtained by the detector.	The syringe method, gas sampling bag method, gas collecting bottle method or gas collecting can method. Sampling amount of sample gas: about 1 ml to 1 L	In the case of sample introduction amount 100 µl: Thermal conductivity detector: 200 vol ppm to 20 vol % Flame photometric detector: 0.2 vol ppm to 50 vol ppm Atomic emission detector: 0.05 vol ppm to 50 vol ppm	(Gas sampler) In the case of analyzing immediately after sampling: a gas sampling syringe made of hard glass, 1 ml to 5 ml in capacity, or a gas sample introducing apparatus attached to the apparatus, etc. is used. In the case of transferring to analysis room after sampling: a gas sampling bag 1 L or over in capacity or a gas collecting bottle 1 L or over in capacity is used.
Methylene blue absorptiometry (see clause 9)	After flue gas is allowed to be absorbed in the absorbing solution, the absorbance of the methylene blue generated by <i>N,N</i> -dimethyl- <i>p</i> -phenylenediammonium and iron (III) is measured and hydrogen sulfide is determined.	Absorbing bottle method Absorbing solutions: in accordance with 6.3.2.3 a) Sampling amount of sample gas: 1 L to 20 L	1.7 vol ppm to 6.9 vol ppm (volume concentration)	(Absorbing bottle of sample gas) In the case of sampling amount of sample gas 1 L or over: two absorbing bottles as shown in figure 4 are used. In the case of sampling amount of sample gas less than 1 L: one absorbing bottle as shown in figure 6 is used.
Ion selective electrode method (see clause 10)	After flue gas is allowed to be absorbed in the absorbing solution, potential difference is measured by means of an ion selective electrode and hydrogen sulfide is determined.	Absorbing bottle method Absorbing solution: in accordance with 6.3.2.3 b) Sampling amount of sample gas: 1 L to 20 L	0.01 vol ppm to 1 000 vol ppm	(Gas sampler) In the case of sampling amount of sample gas 1 L or over: two absorbing bottles as shown in figure 4 are used. In the case of sampling amount of sample gas less than 1 L: one absorbing bottle as shown in figure 6 is used.
Notes ^{a)} In addition to the methods in this table, there are the silver nitrate potentiometry (Annex A), sulfuric dioxide conversion ultraviolet ray fluorescent method (Annex B) and detecting tube method (Annex C). ^{b)} For the purpose of knowing the approximate concentration of hydrogen sulfide, a detecting tube type hydrogen sulfide measuring apparatus (Length of stain type) or detecting tube type and hydrogen sulfide densitometer may be used.				

6 Method of sampling gas

6.1 Sampling position

The sampling position of sample gas shall be as follows.

- a) For the point to sample representative gas, select a position with minimum ingress of air or sediment of dust, where the flow of flue gas is generally uniformly rectified, and avoid any curves in the flow path or positions where sectional shape suddenly changes.
- b) Select a place where the sampling work can be safely and easily performed and if necessary, provide a scaffold of appropriate area and height around the sampling position.
- c) At the sampling position, provide a sampling opening into which a gas sampling tube can be inserted approximately at right angles to the flow direction of the flue gas.
- d) The sampling opening shall be of a material and construction such that it has enough strength to hold the inserted gas sampling tube and thermal resistance to endure heat up to about 120 °C.
- e) The sampling opening shall be provided with a cover so that whenever the gas sampling tube is not being inserted, incidents such as spouting of flue gas (positive pressure) or ingress of air (negative pressure) can be avoided. Further, when opening the cover of the sampling opening, sufficient care shall be taken for the burn and the danger due to spouting of flue gas.

6.2 Sampling gas apparatus and instrument

The sampling apparatus and instrument for sample gas shall be provided with the following function and condition.

- a) The tube used for the gas sampling tube and tubings connecting between instruments shall be made of a material which is resistant to corrosion by hydrogen sulfide in flue gas, e.g. borosilicate tube, silica glass tube or fluoroethylene resin tube.
- b) In order to prevent dust from mixing into sample gas, plug the end of the sampling tube or other suitable place with filter medium (e.g. silica wool, sintered glass filter, etc.)
- c) Tubings shall be as short as possible in order to avoid condensation of moisture, and shall be heated to 120 °C. However, where there is no possibility of condensation of moisture, heating may be omitted.
- d) The washing bottle (E) as shown in figure 1 shall be used.
- e) Set up the gas sampling apparatus and instruments at a site free from direct sunlight.

6.3 Constitution of gas sampling apparatus and sampling procedure

The constitution of gas sampling apparatus and sampling procedure for respective analysis methods shall be as follows.

6.3.1 Gas chromatography

6.3.1.1 Gas sampling apparatus and instruments

An example of the gas sampling apparatus to be used in gas chromatography is shown in figure 1.

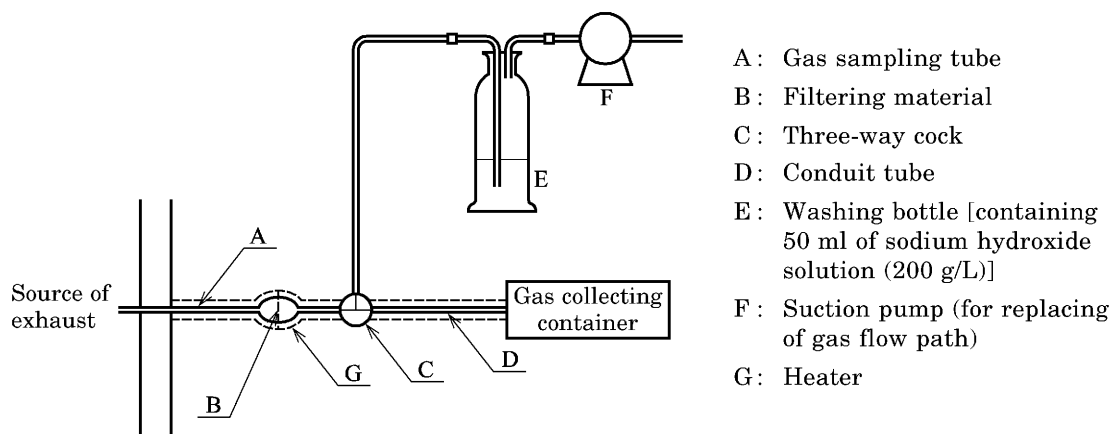


Figure 1 Example of gas sampling apparatus

6.3.1.2 Reagents for washing solution

Reagents used for the washing solution of the gas sampling apparatus shall be as follows.

- Sodium hydroxide**, specified in **JIS K 8576**.
- Phosphoric acid**, specified in **JIS K 9005**.

6.3.1.3 Preparation of washing solution

The preparation of washing solution shall be as follows.

- For the washing solution which is to be poured in washing bottle (E), sodium hydroxide solution shall be prepared by dissolving 200 g of sodium hydroxide in water and adding water to make total 1 L.
- For the washing solution which is to be used for a gas collecting container, phosphoric acid solution shall be prepared by adding water to 5.5 g of phosphoric acid to make total 1 L.

6.3.1.4 Sample gas collecting container

In gas chromatography, the sample gas collecting container as indicated in figure 1 can be either of the following examples.

- In the case where the sample gas is injected into gas chromatograph immediately after sampling**
 - Gas sampling syringe**, 1 ml to 5 ml in capacity and made of glass, the inside surface of which is expected to be with minimum adsorption. It shall be cleaned by using the phosphoric acid prepared in **6.3.1.3 b)** beforehand, and washed with water and dried.

b) **In the case where the sample gas (sample in laboratory) carried to a laboratory is injected into gas chromatograph**

- 1) **Gas sampling bag**, which is a bag for sampling gas 1 L or over in capacity, made of polyester resin, fluoroethylene resin, etc. unlikely to adsorb or absorb gas components. This method is not suitable when the sample gas has to be stored for a long time.
- 2) **Gas collecting bottle**, which is a glass bottle of about 1 L capacity exemplified in **b) 2)** of figure 4 in **JIS K 0095**. It shall be cleaned by using the phosphoric acid prepared in **6.3.1.3 b)** beforehand and then washed with water and dried.
- 3) **Gas collecting can**, of stainless steel, 1 L or over in capacity, of which the inside surface is electrolytically polished and inactivated by silica-coating and provided with a shut-out cock with the inside surface of gas flow path inactivated.

6.3.1.5 Gas sampling procedure

The gas sampling procedure in the case of gas chromatography shall be as follows.

The suction pump (F) shall be operated beforehand and the gas flow path of the gas sampling tube (A), filtering material (B), etc. shall be sufficiently replaced by sample gas beforehand.

- a) **In the case of using gas sampling syringe** Connect a gas sampling syringe to the conduit tube (D) of the gas sampling apparatus in figure 1. Open the three-way cock (C) and after repeating the pumping of syringe to replace sufficiently the gas in flow path, perform suction of the sample gas. Sample the gas, close the three-way cock (C), detach the gas sampling syringe and take it as the sample gas for analysis.
- b) **In the case of using gas sampling bag** An example of the gas sampling apparatus using gas sampling bag is as shown in figure 2. Connect the gas sampling bag (H) placed in the acrylic resin airtight container (I) as shown in figure 2 to the conduit tube (D) of the gas sampling apparatus in figure 1 and open the three-way cock (C). Operate the suction pump (L) in figure 2 and suction the sample gas by opening the shut-out cocks (J) and (K). Take the sample gas, then close the shut-out cock (J) and the preceding three-way cock (C), stop the suction pump (L), take out the gas sampling bag (H) from the acrylic resin airtight container (I), seal tightly the gas sampling bag and take it as the sample gas for analysis.

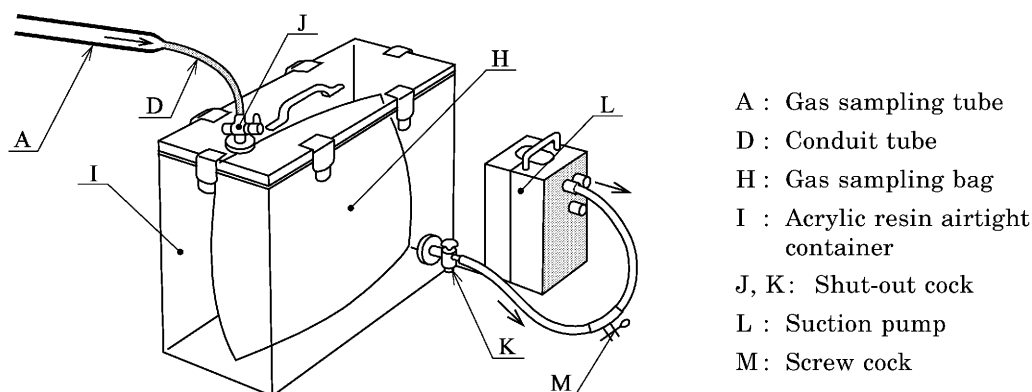


Figure 2 Example of gas sampling apparatus using gas sampling bag

c) In the case of gas collecting bottle

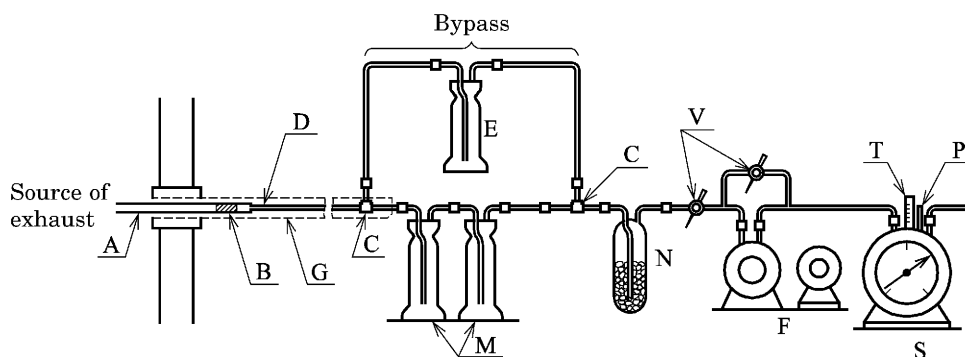
- 1) **Method of making vacuum** Connect the gas collecting bottle which has been made vacuum according to **7.4.2** and **7.4.5 a)** of **JIS K 0095** as the gas collecting container to the conduit tube (D) of gas sampling apparatus in figure 1. Open the three-way cock (C) and the shut-out cock of gas collecting bottle and suction the sample gas. After the sample gas is taken, close the three-way cock (C) and the shut-out cock of the gas collecting bottle, then detach the gas collecting bottle and take it as the sample gas for analysis.
 - 2) **Method of replacing with sample gas** Connect a gas collecting bottle between the three-way cock (C) and the washing bottle (E) of the gas sampling apparatus in figure 1. Open the three-way cock (C) and the shut-out cock of gas collecting bottle, and absorb the sample gas in the collecting bottle by operating the suction pump (F). In this case, allow the sample gas of a volume 10 times or over the capacity of the gas sampling tube, tubings and gas collecting bottle to flow. After replacing the air in the bottle with sample gas, close the shut-out cock of gas collecting bottle and the three-way cock (C), stop the suction pump (F), then detach the gas collecting bottle and take it as the sample gas for analysis.
- d) **In the case of using gas collecting can** Connect the gas collecting can which has been made vacuum beforehand by using a pump to the conduit tube (D) of the gas sampling apparatus in figure 1. Open the three-way cock (C) and the switching valve of gas collecting can and suction the sample gas. After suctioning the sample gas, close the three-way cock (C) and the shut-out cock of collecting can, then detach the gas collecting can and take it as the sample gas for analysis.

6.3.2 In the case of methylene blue absorptiometry and ion selective electrode method

6.3.2.1 Apparatus and instruments for sampling gas

For the gas sampling apparatus and absorbing bottle in the case of methylene blue absorptiometry and ion selective electrode method, the following shall be used depending on the sampling amount of sample gas.

- a) **In the case of sampling amount 1 L or over** An example of gas sampling apparatus is as shown in figure 3 and an example of absorbing bottle to be used is as shown in figure 4.



- | | |
|--|---------------------------------------|
| A: Gas sampling tube | M: Absorbing bottle |
| B: Filtering material | N: Trap (packed with glass wool) |
| C: Three-way cock | S: Wet gas meter |
| D: Conduit tube | T: Thermometer |
| E: Washing bottle (containing 50 ml of washing solution) | P: Manometer |
| F: Suction pump (1 L/min to 5 L/min) | V: Flow rate controlling valve (cock) |
| G: Heater | |

Figure 3 Example of gas sampling apparatus

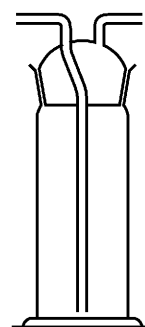
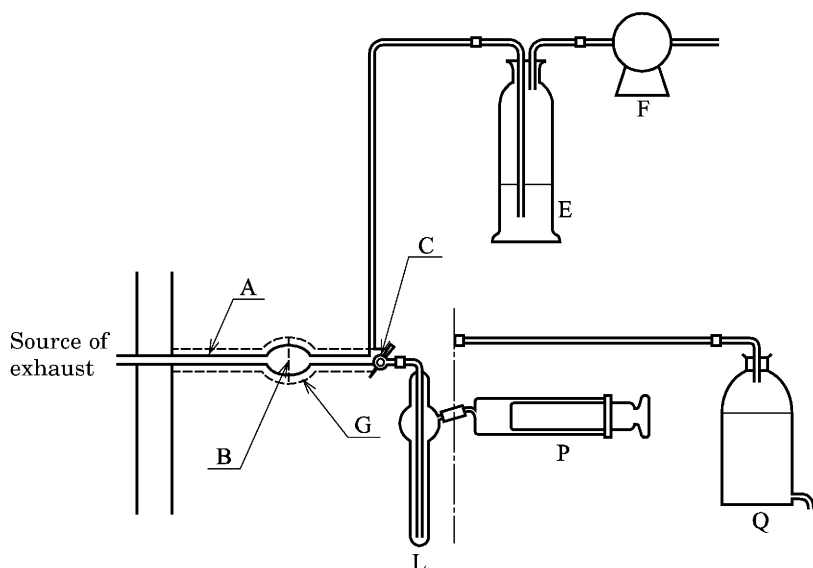


**Figure 4
Example of
absorbing
bottle**

- b) **In the case of sampling amount under 1 L** An example of the gas sampling apparatus is shown in figure 5 and an example of the absorbing bottle to be used is shown in figure 6.



- | | |
|--|------------------------------|
| A: Gas sampling tube | G: Heater |
| B: Filtering material | L: Absorbing bottle |
| C: Three-way cock | P: Injector syringe (100 ml) |
| E: Washing bottle (containing 50 ml of washing solution) | Q: Absorbing bottle (1 L) |
| F: Suction pump | |

NOTE : L and Q are connected when using an absorbing bottle.

Figure 5 Example of gas sampling apparatus

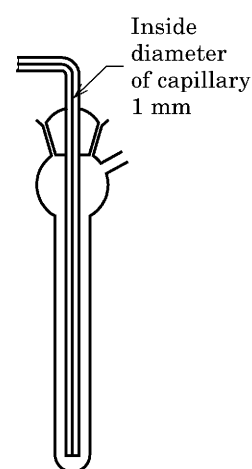


Figure 6 Example of absorbing bottle

6.3.2.2 Reagents for absorbing solution and washing solution

In the case of methylene blue absorptiometry and ion selective electrode method, the reagents to be used for the absorbing solution poured in the absorbing bottle of (M) in figure 3 or (L) in figure 5 and for the washing solution poured in the washing bottle (E) shall be as follows.

- Zinc sulfate heptahydrate**, specified in **JIS K 8953**.
- Sodium hydroxide**, specified in **JIS K 8576**.
- Ammonium sulfate**, specified in **JIS K 8960**.
- Glycerin**, specified in **JIS K 8925**.
- Disodium dihydrogen ethylenediamine tetraacetate dihydrate**, specified in **JIS K 8107**.
- L(+)-Ascorbic acid**, specified in **JIS K 9502**.

6.3.2.3 Method for preparation of absorbing solution and washing solution

In the case of methylene blue absorptiometry and ion selective electrode method, the absorbing solution and washing solution shall be prepared as follows.

- a) **Absorbing solution for methylene blue absorptiometry** Dissolve 5 g of zinc sulfate pentahydrate in about 500 ml of water and add the solution in which 6 g of sodium hydroxide is dissolved in about 300 ml of water. Further, add 70 g of ammonium sulfate while stirring, and after the precipitate of zinc hydroxide is dissolved, add water to make total 1 L.
- b) **Absorbing solution for ion selective electrode method** After dissolving 4 g of sodium hydroxide, 200 ml of glycerin, 4 g of disodium dihydrogen ethylenediamine tetraacetate dihydrate and 10 g of L(+)-ascorbic acid in water, add water to make total 1 L.
- c) **Washing solution for gas sampling apparatus** The method for preparation of the gas washing solution for methylene blue absorptiometry and ion selective electrode method shall be in accordance with 6.3.1.3 a).

6.3.2.4 Sample gas sampling procedure

The sample gas sampling procedure for methylene blue absorptiometry and ion selective electrode method shall be as follows.

- a) In the case of the sampling amount 1 L or over, prepare two absorbing bottles shown in figure 4 and pour 50 ml of absorbing solution in each of them. In the case of sampling amount under 1 L, prepare one absorbing bottle shown in figure 6 and pour 10 ml of absorbing solution.
- b) In figure 3, before the sample gas is introduced, replace sufficiently the inside of the conduit tube with sample gas by utilizing bypass, etc. When the conduit tube for bypass can not be provided because of the restriction of space due to the circumstances of sampling site, the conduit tube for bypass may be omitted, provided that the conduit tube (D) from the gas sampling tube (A) to the three-way cock (C) is sufficiently replaced by sample gas.
- c) The sampling amount and suction rate of sample gas shall be in accordance with the values specified for each method.
- d) Measure the temperature and pressure beforehand, at the same time as the measurement of sample gas amount.

6.3.2.5 Sampling amount of sample gas

The sampling amount of sample gas under the standard condition [273.15 K (0 °C), 101.32 kPa] shall be calculated as the dry-base gas amount (V_{SD}) or the wet-base gas amount (V_{SW}) according to formula (1) to formula (4) indicated as follows.

a) Calculation of dry-base gas amount

1) In the case of using wet gas meter

$$V_{SD} = V \times \frac{273.15}{273.15 + t} \times \frac{P_a + P_m - P_v}{101.32} + 22.41 \times (a + b) \dots\dots\dots (1)$$