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**Methods for determination of sulfur  
oxides 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 & 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 0103: 2005 is replaced with this Standard.

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Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public or utility model right. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public or the utility model right.

# Methods for determination of sulfur oxides in flue gas

## Introduction

This Japanese Industrial Standard has been prepared based on the first edition of ISO 7934 published in 1989 and the first edition of ISO 11632 published in 1998. For ISO 7934, the technical contents thereof have been specified in Annex A without any modifications. For ISO 11632, the specification items and specification contents not given in it have been added but part of it has not been adopted.

The portions given sidelines or dotted underlines are the matters in which the contents of the corresponding International Standards have been modified. A list of modifications with the explanations is given in Annex JE. Annex JA to Annex JD are the matters not contained in the corresponding International Standards.

## 1 Scope

This Standard specifies the methods for analysis of sulfur oxides in flue gas (hereafter referred to as "analysis methods").

In this Standard, the flue gas means the gas which is exhausted into flue, chimney, duct, etc. from such processes as burning of fuels, roasting of ores, refining of metals, manufacturing of sulfur, other chemical reactions, or desulfurization.

NOTE : The International Standards corresponding to this Standard and the symbol of degree of correspondence are as follows.

ISO 7934 : 1989 *Stationary source emissions — Determination of the mass concentration of sulfur dioxide — Hydrogen peroxide/barium perchlorate/Thorin method*

ISO 11632 : 1998 *Stationary source emissions — Determination of mass concentration of sulfur dioxide — Ion chromatography method* (Overall evaluation : MOD)

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standards and JIS are IDT (identical), MOD (modified), and NEQ (not equivalent) according to ISO/IEC Guide 21-1.

## 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 B 7981 *Automated measuring systems and analyzers for sulfur dioxide in flue gas*

NOTE : Corresponding International Standard : ISO 7935 : 1992 *Stationary source emissions — Determination of the mass concentration of sulfur dioxide — Performance characteristics of automated measuring methods* (MOD)

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*

NOTE : Corresponding International Standard : ISO 10396 : 1993 *Stationary source emissions — Sampling for the automated determination of gas concentrations* (MOD)

JIS K 0104 *Methods for determination of nitrogen oxides in flue gas*

JIS K 0107 *Methods for determination of hydrogen chloride in flue gas*

JIS K 0115 *General rules for molecular absorptiometric analysis*

JIS K 0127 *General rules for ion chromatographic analysis*

JIS K 0557 *Water used for industrial water and wastewater analysis*

JIS K 8005 *Reference materials for volumetric analysis*

JIS K 8032 *Acetonitrile (Reagent)*

JIS K 8102 *Ethanol (95) (Reagent)*

JIS K 8150 *Sodium chloride (Reagent)*

JIS K 8155 *Barium chloride dihydrate (Reagent)*

JIS K 8180 *Hydrochloric acid (Reagent)*

JIS K 8230 *Hydrogen peroxide (Reagent)*

JIS K 8295 *Glycerol (Reagent)*

JIS K 8355 *Acetic acid (Reagent)*

JIS K 8374 *Lead (II) acetate trihydrate (Reagent)*

JIS K 8376 *Barium acetate (Reagent)*

JIS K 8576 *Sodium hydroxide (Reagent)*

JIS K 8603 *Soda lime (Reagent)*

JIS K 8622 *Sodium hydrogen carbonate (Reagent)*

JIS K 8625 *Sodium carbonate (Reagent)*

JIS K 8839 *2-Propanol (Reagent)*

JIS K 8844 *Bromophenol blue (Reagent)*

JIS K 8863 *Boric acid (Reagent)*

JIS K 8866 *Sodium tetraborate decahydrate (Reagent)*

JIS K 8896 *Methyl red (Reagent)*

JIS K 8897 *Methylene blue (Reagent)*

JIS K 8951 *Sulfuric acid (Reagent)*

|            |                                                                                |
|------------|--------------------------------------------------------------------------------|
| JIS K 8962 | <i>Potassium sulfate (Reagent)</i>                                             |
| JIS K 8987 | <i>Sodium sulfate (Reagent)</i>                                                |
| JIS K 9524 | <i>Arsenazo III (Reagent)</i>                                                  |
| JIS K 9704 | <i>2-Amino-2-hydroxymethyl-1,3-propanediol (Reagent)</i>                       |
| JIS K 9808 | <i>2-Bis (2-hydroxyethyl) amino-2-hydroxymethyl-1,3-propanediol (Bis-Tris)</i> |
| JIS R 3503 | <i>Glass apparatus for chemical analysis</i>                                   |
| JIS Z 8808 | <i>Methods of measuring dust concentration in flue gas</i>                     |
| ISO 3696   | <i>Water for analytical laboratory use — Specification and test methods</i>    |

### 3 General matters

The general matters shall be as follows.

- a) General matters common to the chemical analysis shall be in accordance with **JIS K 0050**.
- b) General matters common to the sampling of flue gas shall be in accordance with **JIS K 0095**.
- c) General matters common to the ion chromatography shall be in accordance with **JIS K 0127**.
- d) General matters common to the molecular absorptiometric analysis shall be in accordance with **JIS K 0115**.
- e) Water to be used in analysis shall be of A2 or A3 specified in clause 4 of **JIS K 0557** or any water equivalent to these.
- f) 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.
- g) For the reference solution, the reference solution with traceability secured or that solution diluted to a definite concentration, and in addition, the solutions of which the preparation methods are specified for respective test items 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.

NOTE : For the reagents with traceability secured, there are the reagents labelled with JCSS mark specified in the national measurement standard (Article 134 of the Measurement Act).

#### 4 Classification and summary of analysis methods

The classification and summary of analysis methods shall be as follows.

- a) **Chemical analysis method (objective components : SO<sub>2</sub> + SO<sub>3</sub>)** The classification and summary of chemical analysis method shall be given in table 1.

**Table 1 Type and summary of chemical analysis method**

| Type of analysis method                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | Summary                                                                                                                                                                                                                      |                                                                                                                                                                                                                          |                                                                                                                                     | Applicable condition |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------|----------------------|
|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             | Outline                                                                                                                                                                                                                      | Sampling                                                                                                                                                                                                                 | Determination range<br>vol ppm<br>(mg/m <sup>3</sup> )                                                                              |                      |
| Ion chromatography                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          | After absorption of sulfur oxides in sample gas into hydrogen peroxide solution to make sulfuric acid, it is introduced into ion chromatograph and chromatogram thereof is recorded.                                         | Absorbing bottle method<br>Absorption solution : hydrogen peroxide solution (1 + 99) or (1 + 9)<br>Liquid amount : 25 ml <sup>a)</sup> × 2 bottles or 50 ml <sup>b)</sup> × 2 bottles<br>Standard sampling volume : 20 L | 0.5 to 58 <sup>c)</sup><br>(1.4 to 160) <sup>c)</sup><br>12 to 290 <sup>d)</sup><br>(34 to 830) <sup>d)</sup>                       | According to 7.1.1   |
| Precipitation titrimetry (arsenazo III method)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              | After absorption of sulfur oxides in sample gas into hydrogen peroxide solution to make sulfuric acid, add 2-propanol and acetic acid, and titrate it with barium acetate solution with adding arsenazo III as an indicator. | Absorbing bottle method<br>Absorption solution : hydrogen peroxide solution (1 + 9)<br>Liquid amount : 50 ml <sup>b)</sup> × 2 bottles<br>Standard sampling volume : 20 L                                                | 140 to 700 <sup>d)</sup><br>(400 to 2 000) <sup>d)</sup><br>For photometric titrimetry, lowest determination limit : 50 (about 140) | Not specified        |
| Notes <sup>a)</sup> Liquid amount for absorption when using absorbing bottle (100 ml in capacity).<br><sup>b)</sup> Liquid amount for absorption when using absorbing bottle (250 ml in capacity).<br><sup>c)</sup> In the case of employing the sample solution for analysis prepared by diluting 50 ml of absorption solution through which the sample gas has been passed to 100 ml. The determination range shown in this column is obtained according to the standard sampling volume of the sample gas, the amount of sample solution for analysis and the optimum range of the working curve. When determining the concentration in this determination range or under, the concentrating column shall be used.<br><sup>d)</sup> In the case of employing the sample solution for analysis prepared by diluting 100 ml of absorption solution through which the sample gas has been passed to 250 ml. The determination range shown in this column is obtained according to the standard sampling volume of the sample gas, the amount of sample solution for analysis and the optimum range of the working curve. For the concentration over the determination range, it shall be measured in such a manner that after collecting by using aqueous solution of hydrogen peroxide solution (1 + 9), the sample solution for analysis shall be diluted within the determination range. |                                                                                                                                                                                                                              |                                                                                                                                                                                                                          |                                                                                                                                     |                      |

**NOTE 1** The symbols, vol ppm and mg/m<sup>3</sup>, in this Standard express the volume fraction and the mass concentration, respectively under the standard condition [273.15 K (0 °C), 101.32 kPa].

**NOTE 2** For the simplified method for analysis for the concentration of sulfur dioxide in flue gas, a detecting tube method, a simplified type of automated measuring instrument, and so on are included. For the detecting tube, the sample gas of approximately 100 ml is sucked in the detecting tube, reacted with the reagent in the tube, and the concentration of sulfur dioxide is obtained from the coloured length of reagent. The measurement range of concentration is 5 vol ppm to 3 % by the suction of 100 ml, depending on the type of detecting tube and the suction amount of sample gas. The concentration of sulfur dioxide in flue gas can be approximated by means of this method.

Besides the methods in table 1, the precipitation titrimetry (Thorin method) of ISO 7934 is specified in Annex A (normative), the turbidimetry (light scattering method) is specified in Annex JA (normative), the neutralization titrimetry is specified in Annex JB (normative), the simultaneous analysis method of sulfur oxides and hydrogen chloride by ion chromatography is specified in Annex JC (normative) and the simultaneous analysis method of sulfur oxides, hydrogen chloride and nitrogen oxides by ion chromatography is specified in Annex JD (normative).

- b) **Automated measuring method (objective component : SO<sub>2</sub>)** The automated measuring method for sulfur dioxide in flue gas shall be in accordance with the method specified in JIS B 7981.

## 5 Sampling method of sample gas

### 5.1 General

The sampling method of sample gas used in the chemical analysis shall be as follows. The sampling position of sample gas used for analysis shall be selected from the point where representative sample gas can be sampled. The sample gas shall be taken at least twice without interval as far as possible, as a rule, at the same sampling position. Each of them shall be used for the analysis.

### 5.2 Reagents and preparation of absorption solution

#### 5.2.1 Reagents

**Hydrogen peroxide** That specified in JIS K 8230.

#### 5.2.2 Preparation of absorption solution

- a) **Hydrogen peroxide solution (1 + 9)** Take 100 ml of hydrogen peroxide and add 900 ml of water to it. Store it in dark and cold place.
- b) **Hydrogen peroxide solution (1 + 99)** Take 10 ml of hydrogen peroxide and add 990 ml of water to it. Store it in dark and cold place.

### 5.3 Implements and apparatus

#### a) Absorbing bottle

- 1) **Case of ion chromatography** Connect two absorbing bottles (100 ml or 250 ml capacity) as exemplified in figure 1.
- 2) **Case of precipitation titrimetry (arsenazo III method)** Connect two absorbing

bottles (250 ml capacity) as exemplified in figure 1.

b) **Sampling apparatus of sample gas** The construction is as exemplified in figure 2, and the following conditions shall be satisfied.

- 1) The sampling tube of sample gas (B) shall not be eroded by the corrosive gas in flue gas, and shall be made of the material such as the glass tube, quartz tube, tetrafluoro ethylene resin tube which does not absorb the corrosive gas.
- 2) For preventing the dust contamination into the sample gas, the end or other suitable place of sampling tube of sample gas (B) shall be filled with appropriate filtering medium <sup>1)</sup>.
- 3) For preventing the moisture condensation in the sample gas, the apparatus shall be so constructed that the part from the sampling tube to the three-way cock for flow path switch (P<sub>1</sub>) can be heated.

To connect these parts, the ground connecting pipes, silicone rubber pipes or polytetrafluoroethylene pipes shall be used. The piping shall be as short as possible, and if there is a possibility of moisture condensation, the part between the sampling tube and the cock (P<sub>1</sub>) shall be heated at about 160 °C.

- 4) The whole apparatus shall be set up so as to get rid of any leakage at each connection.

Note <sup>1)</sup> The material, such as silica wool, non-alkaline wool which causes no chemical reaction with the component in flue gas shall be used.

Unit : mm    Inside (    ) for 100 ml and  
outside (    ) for 250 ml.

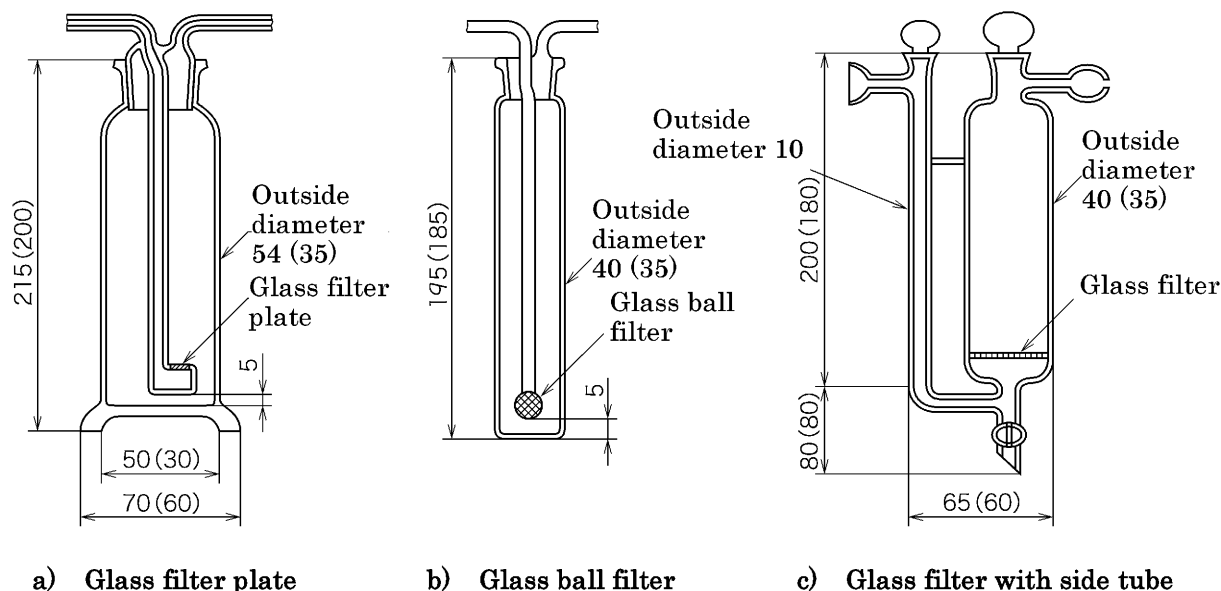
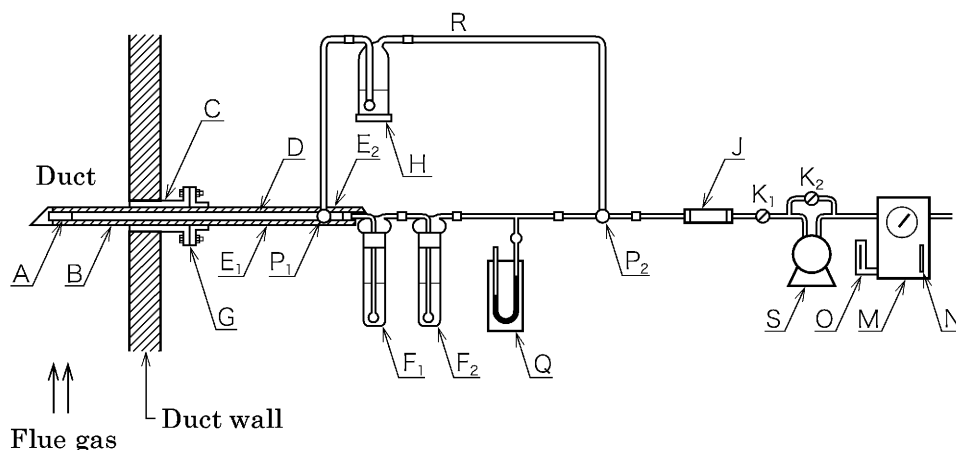


**Figure 1** Example of absorbing bottle (100 ml and 250 ml)

For the pore size of glass filter plate, glass ball filter and glass filter, the pore size symbol G1 or G2 specified in **JIS R 3503** shall be used.



- |                                                                                   |                                                                       |
|-----------------------------------------------------------------------------------|-----------------------------------------------------------------------|
| A : filtering medium                                                              | K <sub>1</sub> , K <sub>2</sub> : flow rate control cock              |
| B : sampling tube of sample gas                                                   | M : wet gas meter or dry gas meter                                    |
| C : inlet                                                                         | N : thermometer                                                       |
| D : thermometer                                                                   | O : manometer                                                         |
| E <sub>1</sub> , E <sub>2</sub> : heater, ribbon heater                           | P <sub>1</sub> , P <sub>2</sub> : three-way cock for flow path switch |
| F <sub>1</sub> , F <sub>2</sub> : absorbing bottle (250 ml or 100 ml in capacity) | Q : mercury manometer                                                 |
| G : flange                                                                        | R : bypass                                                            |
| H : washing bottle (put 50 ml of absorption solution)                             | S : suction pump                                                      |
| J : drying tube                                                                   |                                                                       |

Figure 2 Example of sampling apparatus of sample gas

#### 5.4 Sampling procedure

The sampling procedure shall be as follows.

The symbols of apparatus adopted here shall be as given in figure 2.

- a) The absorbing bottle and the liquid amount for absorption shall be either of the following.
  - 1) **Case of ion chromatography** Put 25 ml of absorption solution of 5.2.2 b) into each of the absorbing bottles (100 ml in capacity) (F<sub>1</sub> and F<sub>2</sub>). When the concentration of sulfur oxides is expected to exceed 58 vol ppm, put 50 ml of absorption solution of 5.2.2 a) into each of the absorbing bottles (250 ml in capacity) (F<sub>1</sub> and F<sub>2</sub>).
  - 2) **Case of precipitation titrimetry (arsenazo III method)** Put 50 ml of absorption solution of 5.2.2 a) into each of the absorbing bottles (250 ml in capacity) (F<sub>1</sub> and F<sub>2</sub>).
- b) Turn the three-way cocks for flow path switch (P<sub>1</sub> and P<sub>2</sub>) to bypass (R) side, adjust the flow rate 1 L/min to 2 L/min, start the suction pump (S), and replace inside air between the sampling tube of sample gas (B) and the three-way cock for flow path switch (P<sub>1</sub>) by the sample gas.

NOTE 1 The bypass (R) between P<sub>1</sub> and P<sub>2</sub> in figure 2 may be omitted, provided that the distance between the sampling tube of sample gas (B) and the absorbing bottle is short and the flow rate can be adjusted to a definite amount.

NOTE 2 When it can be confirmed that the leak does not occur in the sampling apparatus by other technique, a mercury manometer (Q) in figure 2 may be omitted.

- c) After stopping the suction pump (S), close the three-way cocks for flow path switch (P<sub>1</sub> and P<sub>2</sub>) toward the absorbing bottles' (F<sub>1</sub> and F<sub>2</sub>) side. Then, read the indication (V<sub>1</sub>) of a gas meter (M) to the nearest 0.01 L.
- d) Start the suction pump (S) and pass the sample gas through the absorbing bottles (F<sub>1</sub> and F<sub>2</sub>). At this time, adjust a flow rate to about 1 L/min by adjusting the flow rate control cocks (K<sub>1</sub> and K<sub>2</sub>).

If there is a possibility that the absorption solution is warmed, place the absorbing bottles in a cooling tank. In this case, the absorbing bottle shaped as figure 1 a) should be used. When the adsorbing bottle of 250 ml in capacity is used in ion chromatography and when it has been known that sulfur oxides will be completely absorbed in the absorption solution in precipitation titrimetry, the flow rate may be increased up to 2 L/min.

- e) Measure the temperature and the gauge pressure of the gas meter with the thermometer (N) and the manometer (O) of the gas meter (M). Also, measure the atmospheric pressure.
- f) After sampling about 20 L of sample gas, stop the suction pump (S), close the three-way cocks for flow path switch (P<sub>1</sub> and P<sub>2</sub>), and read the indication (V<sub>2</sub>) of a gas meter (M) to the nearest 0.01 L.

This amount may be suitably increased/decreased according to the sulfur oxides concentration.

- g) If necessary, measure the moisture in the sample gas in accordance with clause 6 of JIS Z 8808.

## 5.5 Sampling volume of sample gas

According to the following formulae, the sampling volume of sample gas under the standard conditions [273.15 K (0 °C), 101.32 kPa] shall be converted into the dry-base gas volume (V<sub>SD</sub>) or the wet-base gas volume (V<sub>SW</sub>).

### 5.5.1 When obtaining by dry-base gas amount

- a) When using a wet-base gas meter,

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

- b) When using a dry-base gas meter,

$$V_{SD} = V \times \frac{273.15}{273.15 + t} \times \frac{P_a + P_m}{101.32} + 22.41(a + b) \dots\dots\dots(2)$$

### 5.5.2 When obtaining by wet-base gas amount

#### a) When using a wet-base gas meter,

$$V_{\text{SW}} = V \times \frac{273.15}{273.15 + t} \times \frac{P_a + P_m - P_v}{101.32} + 22.41(a + b + c) \dots\dots\dots (3)$$

#### b) When using a dry-base gas meter,

$$V_{\text{SW}} = V \times \frac{273.15}{273.15 + t} \times \frac{P_a + P_m}{101.32} + 22.41(a + b + c) \dots\dots\dots (4)$$

- where,
- $V_{\text{SD}}$  : dry-base gas volume (L)
  - $V_{\text{SW}}$  : wet-base gas volume (L)
  - $V$  : gas volume measured by gas meter (L)  
[equals to  $V_2 - V_1$  at procedures of 5.4 c) and f)]
  - $t$  : temperature at gas meter (°C)
  - $P_a$  : atmospheric pressure (kPa)
  - $P_m$  <sup>2)</sup> : gauge pressure at gas meter (kPa)
  - $P_v$  : saturated water vapour pressure at  $t$  °C (kPa)
  - $a$  <sup>2)</sup> : analyte gas collected in absorption solution (mol)
  - $b$  <sup>2)</sup> : gases other than analyte gas collected in absorption solution (mol)
  - $c$  <sup>2)</sup> : moisture amount obtained in clause 6 of JIS Z 8808 (mol)
  - 22.41 : volume of 1 mol gas under standard condition (L)

Note <sup>2)</sup> In most cases, it can be neglected.

$P_v$  shall be as given in table 2.

**Table 2 Saturated vapour pressure of water**

Unit : Pa

| $t/^{\circ}\text{C}$ | 0.0     | 0.1     | 0.2     | 0.3     | 0.4     | 0.5     | 0.6     | 0.7     | 0.8     | 0.9     |
|----------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| 0.                   | 611.21  | 615.67  | 620.15  | 624.67  | 629.21  | 633.78  | 638.38  | 643.01  | 647.67  | 652.36  |
| 1.                   | 657.08  | 661.83  | 666.61  | 671.42  | 676.26  | 681.14  | 686.04  | 690.98  | 695.94  | 700.94  |
| 2.                   | 705.97  | 711.03  | 716.13  | 721.26  | 726.41  | 731.61  | 736.83  | 742.09  | 747.38  | 752.70  |
| 3.                   | 758.06  | 763.45  | 768.88  | 774.34  | 779.83  | 785.36  | 790.92  | 796.52  | 802.15  | 807.82  |
| 4.                   | 813.52  | 819.26  | 825.03  | 830.84  | 836.69  | 842.57  | 848.49  | 854.45  | 860.44  | 866.47  |
| 5.                   | 872.54  | 878.64  | 884.79  | 890.97  | 897.19  | 903.44  | 909.74  | 916.07  | 922.45  | 928.86  |
| 6.                   | 935.31  | 941.80  | 948.34  | 954.91  | 961.52  | 968.17  | 974.86  | 981.60  | 988.37  | 995.19  |
| 7.                   | 1 002.0 | 1 008.9 | 1 015.9 | 1 022.9 | 1 029.9 | 1 037.0 | 1 044.1 | 1 051.2 | 1 058.4 | 1 065.7 |
| 8.                   | 1 072.9 | 1 080.3 | 1 087.6 | 1 095.1 | 1 102.5 | 1 110.0 | 1 117.6 | 1 125.2 | 1 132.8 | 1 140.5 |
| 9.                   | 1 148.2 | 1 156.0 | 1 163.8 | 1 171.7 | 1 179.6 | 1 187.6 | 1 195.6 | 1 203.7 | 1 211.8 | 1 219.9 |
| 10.                  | 1 228.1 | 1 236.4 | 1 244.7 | 1 253.0 | 1 261.4 | 1 269.9 | 1 278.4 | 1 286.9 | 1 295.5 | 1 304.2 |
| 11.                  | 1 312.9 | 1 321.7 | 1 330.5 | 1 339.3 | 1 348.2 | 1 357.2 | 1 366.2 | 1 375.3 | 1 384.4 | 1 393.5 |
| 12.                  | 1 402.8 | 1 412.1 | 1 421.4 | 1 430.8 | 1 440.2 | 1 449.7 | 1 459.3 | 1 468.9 | 1 478.5 | 1 488.2 |
| 13.                  | 1 498.0 | 1 507.8 | 1 517.7 | 1 527.7 | 1 537.7 | 1 547.7 | 1 557.9 | 1 568.0 | 1 578.3 | 1 588.6 |
| 14.                  | 1 598.9 | 1 609.3 | 1 619.8 | 1 630.3 | 1 640.9 | 1 651.6 | 1 662.3 | 1 673.0 | 1 683.9 | 1 694.8 |
| 15.                  | 1 705.7 | 1 716.7 | 1 727.8 | 1 739.0 | 1 750.2 | 1 761.4 | 1 772.8 | 1 784.2 | 1 795.6 | 1 807.1 |
| 16.                  | 1 818.7 | 1 830.4 | 1 842.1 | 1 853.9 | 1 865.8 | 1 877.7 | 1 889.7 | 1 901.7 | 1 913.8 | 1 926.0 |
| 17.                  | 1 938.3 | 1 950.6 | 1 963.0 | 1 975.5 | 1 988.0 | 2 000.6 | 2 013.3 | 2 026.0 | 2 038.8 | 2 051.7 |
| 18.                  | 2 064.7 | 2 077.7 | 2 090.8 | 2 104.0 | 2 117.2 | 2 130.5 | 2 143.9 | 2 157.4 | 2 170.9 | 2 184.5 |
| 19.                  | 2 198.2 | 2 212.0 | 2 225.8 | 2 239.7 | 2 253.7 | 2 267.8 | 2 281.9 | 2 296.1 | 2 310.4 | 2 324.8 |
| 20.                  | 2 339.2 | 2 353.8 | 2 368.4 | 2 383.1 | 2 397.8 | 2 412.7 | 2 427.6 | 2 442.6 | 2 457.7 | 2 472.9 |
| 21.                  | 2 488.2 | 2 503.5 | 2 518.9 | 2 534.4 | 2 550.0 | 2 565.7 | 2 581.4 | 2 597.3 | 2 613.2 | 2 629.2 |
| 22.                  | 2 645.3 | 2 661.5 | 2 677.7 | 2 694.1 | 2 710.5 | 2 727.1 | 2 743.7 | 2 760.4 | 2 777.2 | 2 794.1 |
| 23.                  | 2 811.0 | 2 828.1 | 2 845.2 | 2 862.5 | 2 879.8 | 2 897.2 | 2 914.8 | 2 932.4 | 2 950.1 | 2 967.9 |
| 24.                  | 2 985.8 | 3 003.7 | 3 021.8 | 3 040.0 | 3 058.3 | 3 076.6 | 3 095.1 | 3 113.6 | 3 132.3 | 3 151.1 |
| 25.                  | 3 169.9 | 3 188.9 | 3 207.9 | 3 227.0 | 3 246.3 | 3 265.6 | 3 285.1 | 3 304.6 | 3 324.3 | 3 344.0 |
| 26.                  | 3 363.9 | 3 383.8 | 3 403.9 | 3 424.0 | 3 444.3 | 3 464.7 | 3 485.2 | 3 505.7 | 3 526.4 | 3 547.2 |
| 27.                  | 3 568.1 | 3 589.1 | 3 610.2 | 3 631.5 | 3 652.8 | 3 674.2 | 3 695.8 | 3 717.4 | 3 739.2 | 3 761.1 |
| 28.                  | 3 783.1 | 3 805.2 | 3 827.4 | 3 849.7 | 3 872.2 | 3 894.7 | 3 917.4 | 3 940.2 | 3 963.1 | 3 986.1 |
| 29.                  | 4 009.2 | 4 032.5 | 4 055.8 | 4 079.3 | 4 102.9 | 4 126.6 | 4 150.5 | 4 174.4 | 4 198.5 | 4 222.7 |
| 30.                  | 4 247.0 | 4 271.5 | 4 296.0 | 4 320.7 | 4 345.5 | 4 370.5 | 4 395.5 | 4 420.7 | 4 446.0 | 4 471.5 |
| 31.                  | 4 497.0 | 4 522.7 | 4 548.5 | 4 574.5 | 4 600.5 | 4 626.7 | 4 653.1 | 4 679.5 | 4 706.1 | 4 732.8 |
| 32.                  | 4 759.7 | 4 786.7 | 4 813.8 | 4 841.0 | 4 868.4 | 4 895.9 | 4 923.6 | 4 951.4 | 4 979.3 | 5 007.4 |
| 33.                  | 5 035.6 | 5 063.9 | 5 092.4 | 5 121.0 | 5 149.7 | 5 178.6 | 5 207.7 | 5 236.8 | 5 266.2 | 5 295.6 |
| 34.                  | 5 325.2 | 5 355.0 | 5 384.8 | 5 414.9 | 5 445.1 | 5 475.4 | 5 505.9 | 5 536.5 | 5 567.2 | 5 598.1 |
| 35.                  | 5 629.2 | 5 660.4 | 5 691.8 | 5 723.3 | 5 754.9 | 5 786.8 | 5 818.7 | 5 850.8 | 5 883.1 | 5 915.5 |
| 36.                  | 5 948.1 | 5 980.8 | 6 013.7 | 6 046.8 | 6 080.0 | 6 113.3 | 6 146.9 | 6 180.5 | 6 214.4 | 6 248.4 |
| 37.                  | 6 282.5 | 6 316.9 | 6 351.3 | 6 386.0 | 6 420.8 | 6 455.8 | 6 490.9 | 6 526.2 | 6 561.7 | 6 597.3 |
| 38.                  | 6 633.1 | 6 669.1 | 6 705.2 | 6 741.5 | 6 778.0 | 6 814.7 | 6 851.5 | 6 888.5 | 6 925.6 | 6 963.0 |
| 39.                  | 7 000.5 | 7 038.2 | 7 076.0 | 7 114.1 | 7 152.3 | 7 190.7 | 7 229.2 | 7 268.0 | 7 306.9 | 7 346.0 |
| 40.                  | 7 385.3 | 7 424.8 | 7 464.4 | 7 504.2 | 7 544.3 | 7 584.5 | 7 624.8 | 7 665.4 | 7 706.2 | 7 747.1 |

Table 2 (continued)

Unit : Pa

| t/°C | 0.0     | 0.1     | 0.2     | 0.3     | 0.4     | 0.5     | 0.6     | 0.7     | 0.8     | 0.9     |
|------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| 41.  | 7 788.2 | 7 829.6 | 7 871.1 | 7 912.8 | 7 954.6 | 7 996.7 | 8 039.0 | 8 081.5 | 8 124.1 | 8 167.0 |
| 42.  | 8 210.0 | 8 253.2 | 8 296.7 | 8 340.3 | 8 384.1 | 8 428.2 | 8 472.4 | 8 516.8 | 8 561.5 | 8 606.3 |
| 43.  | 8 651.3 | 8 696.5 | 8 742.0 | 8 787.6 | 8 833.5 | 8 879.5 | 8 925.8 | 8 972.3 | 9 018.9 | 9 065.8 |
| 44.  | 9 112.9 | 9 160.2 | 9 207.7 | 9 255.5 | 9 303.4 | 9 351.6 | 9 399.9 | 9 448.5 | 9 497.3 | 9 546.3 |
| 45.  | 9 595.6 | 9 645.0 | 9 694.7 | 9 744.6 | 9 794.7 | 9 845.0 | 9 895.6 | 9 946.4 | 9 997.4 | 10 049. |
| 46.  | 10 100. | 10 152. | 10 204. | 10 256. | 10 308. | 10 361. | 10 414. | 10 467. | 10 520. | 10 573. |
| 47.  | 10 627. | 10 681. | 10 735. | 10 790. | 10 845. | 10 899. | 10 955. | 11 010. | 11 066. | 11 122. |
| 48.  | 11 178. | 11 234. | 11 291. | 11 348. | 11 405. | 11 462. | 11 520. | 11 578. | 11 636. | 11 694. |
| 49.  | 11 753. | 11 812. | 11 871. | 11 930. | 11 990. | 12 049. | 12 110. | 12 170. | 12 231. | 12 292. |
| 50.  | 12 353. | 12 414. | 12 476. | 12 538. | 12 600. | 12 663. | 12 725. | 12 788. | 12 852. | 12 915. |
| 51.  | 12 979. | 13 043. | 13 107. | 13 172. | 13 237. | 13 302. | 13 368. | 13 433. | 13 499. | 13 566. |
| 52.  | 13 632. | 13 699. | 13 766. | 13 833. | 13 901. | 13 969. | 14 037. | 14 106. | 14 175. | 14 244. |
| 53.  | 14 313. | 14 383. | 14 453. | 14 523. | 14 594. | 14 665. | 14 736. | 14 807. | 14 879. | 14 951. |
| 54.  | 15 023. | 15 096. | 15 169. | 15 242. | 15 316. | 15 389. | 15 464. | 15 538. | 15 613. | 15 688. |
| 55.  | 15 763. | 15 839. | 15 915. | 15 991. | 16 068. | 16 145. | 16 222. | 16 299. | 16 377. | 16 455. |
| 56.  | 16 534. | 16 613. | 16 692. | 16 771. | 16 851. | 16 931. | 17 012. | 17 093. | 17 174. | 17 255. |
| 57.  | 17 337. | 17 419. | 17 501. | 17 584. | 17 667. | 17 750. | 17 834. | 17 918. | 18 003. | 18 087. |
| 58.  | 18 173. | 18 258. | 18 344. | 18 430. | 18 516. | 18 603. | 18 690. | 18 778. | 18 866. | 18 954. |
| 59.  | 19 043. | 19 131. | 19 221. | 19 310. | 19 400. | 19 491. | 19 581. | 19 672. | 19 764. | 19 856. |
| 60.  | 19 948. | 20 040. | 20 133. | 20 226. | 20 320. | 20 414. | 20 508. | 20 603. | 20 698. | 20 793. |
| 61.  | 20 889. | 20 985. | 21 082. | 21 179. | 21 276. | 21 374. | 21 472. | 21 571. | 21 669. | 21 769. |
| 62.  | 21 868. | 21 968. | 22 069. | 22 170. | 22 271. | 22 372. | 22 474. | 22 577. | 22 679. | 22 783. |
| 63.  | 22 886. | 22 990. | 23 094. | 23 199. | 23 304. | 23 410. | 23 516. | 23 622. | 23 729. | 23 836. |
| 64.  | 23 944. | 24 052. | 24 160. | 24 269. | 24 379. | 24 488. | 24 598. | 24 709. | 24 820. | 24 931. |
| 65.  | 25 043. | 25 155. | 25 268. | 25 381. | 25 494. | 25 608. | 25 723. | 25 837. | 25 953. | 26 068. |
| 66.  | 26 184. | 26 301. | 26 418. | 26 535. | 26 653. | 26 772. | 26 890. | 27 010. | 27 129. | 27 249. |
| 67.  | 27 370. | 27 491. | 27 612. | 27 734. | 27 857. | 27 979. | 28 103. | 28 226. | 28 351. | 28 475. |
| 68.  | 28 600. | 28 726. | 28 852. | 28 979. | 29 106. | 29 233. | 29 361. | 29 489. | 29 618. | 29 748. |
| 69.  | 29 877. | 30 008. | 30 138. | 30 270. | 30 402. | 30 534. | 30 667. | 30 800. | 30 933. | 31 068. |
| 70.  | 31 202. | 31 338. | 31 473. | 31 609. | 31 746. | 31 883. | 32 021. | 32 159. | 32 298. | 32 437. |
| 71.  | 32 577. | 32 717. | 32 858. | 32 999. | 33 140. | 33 283. | 33 425. | 33 569. | 33 713. | 33 857. |
| 72.  | 34 002. | 34 147. | 34 293. | 34 439. | 34 586. | 34 734. | 34 882. | 35 030. | 35 179. | 35 329. |
| 73.  | 35 479. | 35 630. | 35 781. | 35 933. | 36 085. | 36 238. | 36 391. | 36 545. | 36 700. | 36 855. |
| 74.  | 37 010. | 37 166. | 37 323. | 37 480. | 37 638. | 37 796. | 37 955. | 38 115. | 38 275. | 38 436. |
| 75.  | 38 597. | 38 758. | 38 921. | 39 084. | 39 247. | 39 411. | 39 576. | 39 741. | 39 907. | 40 073. |
| 76.  | 40 240. | 40 408. | 40 576. | 40 744. | 40 914. | 41 084. | 41 254. | 41 425. | 41 597. | 41 769. |
| 77.  | 41 942. | 42 116. | 42 290. | 42 464. | 42 640. | 42 815. | 42 992. | 43 169. | 43 347. | 43 525. |
| 78.  | 43 704. | 43 884. | 44 064. | 44 245. | 44 426. | 44 608. | 44 791. | 44 974. | 45 158. | 45 343. |
| 79.  | 45 528. | 45 714. | 45 900. | 46 088. | 46 275. | 46 464. | 46 653. | 46 843. | 47 033. | 47 224. |
| 80.  | 47 416. | 47 608. | 47 801. | 47 994. | 48 189. | 48 384. | 48 579. | 48 776. | 48 972. | 49 170. |

**Table 2 (concluded)**

Unit : Pa

| $t/^{\circ}\text{C}$ | 0.0      | 0.1      | 0.2      | 0.3      | 0.4      | 0.5      | 0.6      | 0.7      | 0.8      | 0.9      |
|----------------------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|
| 81.                  | 49 368.  | 49 567.  | 49 767.  | 49 967.  | 50 168.  | 50 370.  | 50 572.  | 50 775.  | 50 979.  | 51 183.  |
| 82.                  | 51 388.  | 51 594.  | 51 800.  | 52 007.  | 52 215.  | 52 424.  | 52 633.  | 52 843.  | 53 053.  | 53 265.  |
| 83.                  | 53 477.  | 53 689.  | 53 903.  | 54 117.  | 54 332.  | 54 547.  | 54 764.  | 54 981.  | 55 198.  | 55 417.  |
| 84.                  | 55 636.  | 55 856.  | 56 076.  | 56 298.  | 56 520.  | 56 743.  | 56 966.  | 57 190.  | 57 415.  | 57 641.  |
| 85.                  | 57 868.  | 58 095.  | 58 323.  | 58 552.  | 58 781.  | 59 011.  | 59 242.  | 59 474.  | 59 707.  | 59 940.  |
| 86.                  | 60 174.  | 60 409.  | 60 644.  | 60 881.  | 61 118.  | 61 356.  | 61 594.  | 61 834.  | 62 074.  | 62 315.  |
| 87.                  | 62 557.  | 62 799.  | 63 042.  | 63 286.  | 63 531.  | 63 777.  | 64 024.  | 64 271.  | 64 519.  | 64 768.  |
| 88.                  | 65 017.  | 65 268.  | 65 519.  | 65 771.  | 66 024.  | 66 278.  | 66 532.  | 66 788.  | 67 044.  | 67 301.  |
| 89.                  | 67 559.  | 67 817.  | 68 077.  | 68 337.  | 68 598.  | 68 860.  | 69 123.  | 69 386.  | 69 651.  | 69 916.  |
| 90.                  | 70 182.  | 70 449.  | 70 717.  | 70 986.  | 71 255.  | 71 526.  | 71 797.  | 72 069.  | 72 342.  | 72 616.  |
| 91.                  | 72 890.  | 73 166.  | 73 442.  | 73 719.  | 73 998.  | 74 277.  | 74 556.  | 74 837.  | 75 119.  | 75 401.  |
| 92.                  | 75 685.  | 75 969.  | 76 254.  | 76 540.  | 76 827.  | 77 115.  | 77 404.  | 77 693.  | 77 984.  | 78 276.  |
| 93.                  | 78 568.  | 78 861.  | 79 155.  | 79 450.  | 79 746.  | 80 043.  | 80 341.  | 80 640.  | 80 940.  | 81 240.  |
| 94.                  | 81 542.  | 81 844.  | 82 148.  | 82 452.  | 82 757.  | 83 064.  | 83 371.  | 83 679.  | 83 988.  | 84 298.  |
| 95.                  | 84 609.  | 84 921.  | 85 234.  | 85 547.  | 85 862.  | 86 178.  | 86 495.  | 86 812.  | 87 131.  | 87 451.  |
| 96.                  | 87 771.  | 88 093.  | 88 415.  | 88 739.  | 89 063.  | 89 389.  | 89 715.  | 90 043.  | 90 371.  | 90 701.  |
| 97.                  | 91 031.  | 91 362.  | 91 695.  | 92 028.  | 92 363.  | 92 698.  | 93 035.  | 93 372.  | 93 711.  | 94 050.  |
| 98.                  | 94 391.  | 94 732.  | 95 075.  | 95 418.  | 95 763.  | 96 109.  | 96 455.  | 96 803.  | 97 152.  | 97 502.  |
| 99.                  | 97 853.  | 98 204.  | 98 557.  | 98 911.  | 99 266.  | 99 623.  | 99 980.  | 100 338. | 100 697. | 101 058. |
| 100.                 | 101 419. | 101 782. | 102 145. | 102 510. | 102 875. | 103 242. | 103 610. | 103 979. | 104 349. | 104 720. |

NOTE : It is based on SONNTAG (1990). A temperature scale is ITS-90.

## 6 Preparation of sample solution for analysis

Preparation shall be carried out according to any one of the following.

### 6.1 Case of ion chromatography

- After the completion of the procedure of 5.4, transfer the content solution in the absorbing bottles ( $F_1$  and  $F_2$ ) into a beaker of 300 ml. Besides, wash the absorbing bottles, etc. with water and transfer the washings into the beaker.
- When an absorbing bottle of 100 ml in capacity is used, transfer the content solution in the beaker into a 100 ml volumetric flask by washing with water and add water up to the marked line. Take this as the sample solution for analysis.
- When an absorbing bottle of 250 ml in capacity is used, transfer the contents solution in the beaker into a 250 ml volumetric flask by washing with water and add water up to the marked line. Take this as the sample solution for analysis. In this case, to lessen the influence on a separation column, it shall be diluted by 10 times or more.
- If solid precipitate is found in the sample solutions for analysis of b) and c), eliminate it by filtering through the filter with 0.45  $\mu\text{m}$  pore diameter or less.

## 6.2 Case of precipitation titrimetry (arsenazo III method)

- a) After the completion of the procedure of 5.4, carry out the procedure of 6.1 a).
- b) Transfer the content solution in the beaker into a 250 ml volumetric flask by washing with water and add water up to the marked line. Take this as the sample solution for analysis.

The measurement of this solution may be carried out by the ion chromatography. But in this case, to lessen the influence on a separation column, it shall be diluted by 10 times or more.

## 7 Determination by chemical analysis

### 7.1 Ion chromatography

#### 7.1.1 Applicable condition

This method is affected by the high concentrated coexistence of reductive gas such as sulfides in the sample gas, therefore this can be applicable when such influence can be neglected or eliminated.

#### 7.1.2 Reagents and preparation of reagent solutions

- a) **Reagents** Reagents shall be as follows.

- 1) **Sulfuric acid** That specified in JIS K 8951.
- 2) **Boric acid** That specified in JIS K 8863.
- 3) **Sodium hydrogen carbonate** That specified in JIS K 8622.
- 4) **Sodium carbonate** That specified in JIS K 8625.
- 5) **Sodium tetraborate decahydrate** That specified in JIS K 8866.
- 6) **Sodium sulfate** That specified in JIS K 8987.
- 7) **Potassium sulfate** That specified in JIS K 8962.
- 8) **Potassium gluconate**
- 9) **p-hydroxybenzoic acid**
- 10) **2-Bis(2-hydroxyethyl)amino-2-hydroxymethyl-1,3-propanediol(Bis-Tris)** That specified in JIS K 9808.
- 11) **Phthalic acid**
- 12) **2-amino-2-hydroxymethyl-1, 3-propanediol [tris (hydroxymethyl) aminomethane]** That specified in JIS K 9704.
- 13) **Acetonitrile** That specified in JIS K 8032.
- 14) **Glycerol** That specified in JIS K 8295.
- 15) **Sulfate ion reference solution (SO<sub>4</sub><sup>2-</sup> : 1 000 mg/L)**

- b) **Preparation of reagent solutions**

- 1) **Eluant** The eluant differs depending on the type of apparatus and the type of separation column to be used, therefore that capable of separating the target ion by not less than 1.3 in resolution ( $R$ ) respectively shall be used<sup>3)</sup>. The confirmation of resolution shall be in accordance with clause 10 of JIS K 0127.

NOTE : Examples of eluant are as follows.

Moreover, if it is confirmed that sulfate ions can be measured quantitatively, other eluant than that indicated herein may be used according to the characteristic of separation column.

- **Hydrogen carbonate-carbonate solution (1)** Dissolve 0.025 g (0.3 mmol) of sodium hydrogen carbonate and 0.286 g (2.7 mmol) of sodium carbonate in water, and transfer it into a 1 000 ml volumetric flask by washing with water and add water up to the marked line.
  - **Hydrogen carbonate-carbonate solution (2)** Dissolve 0.143 g (1.7 mmol) of sodium hydrogen carbonate and 0.191 g (1.8 mmol) of sodium carbonate in water, and transfer it into a 1 000 ml volumetric flask by washing with water and add water up to the marked line.
  - **Gluconate-tetra borate-boric acid solution** Dissolve 0.305 g (1.3 mmol) of potassium gluconate, 0.496 g (1.3 mmol) of sodium tetra borate decahydrate, 1.855 g (30 mmol) of boric acid, 100 ml of acetonitrile and 5 ml of glycerine in water, and transfer it into a 1 000 ml volumetric flask by washing with water and add water up to the marked line.
  - ***p*-hydroxybenzoic acid-2-[bis(2-hydroxyethyl)amino]-2-hydroxymethyl-1,3-propanediol solution** Dissolve 1.105 g (8.0 mmol) of *p*-hydroxybenzoic acid and 0.669 g (3.2 mmol) of 2-bis(2-hydroxyethyl) amino-2-hydroxymethyl-1,3-propanediol in water, and transfer it into a 1 000 ml volumetric flask by washing with water and add water up to the marked line.
  - **Phthalic acid-2-amino-2-hydroxymethyl-1,3-propanediol solution** Dissolve 0.415 g (2.5 mmol) of phthalic acid and 0.290 g (2.4 mmol) of 2-amino-2-hydroxymethyl-1,3-propanediol, or 0.382 g (2.3 mmol) of phthalic acid and 0.303 g (2.5 mmol) of 2-amino-2-hydroxymethyl-1,3-propanediol in water, and transfer it into a 1 000 ml volumetric flask by washing with water and add water up to the marked line.
- 2) **Regeneration solution (removal solution)** This solution is used for regeneration or maintaining continuously the function of a suppressor, and used for regeneration with electrical or chemical methods. The optimum solution shall be used for the type of apparatus and suppressor.

NOTE : Examples of regeneration solution and regenerating material are as follows.

- **Water** That in 3 e) is decomposed by an electric current and used for the suppressor in the system producing regeneration