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**Methods for determination of
fluorine compounds 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 0105:1998** is replaced with this Standard.

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Methods for determination of fluorine compounds in flue gas

Introduction

This Japanese Industrial Standard has been prepared based on the first edition of **ISO 15713** published in 2006 with some modifications of the technical contents such that items unique to **JIS** not given in the corresponding International Standard are added but some specifications in the corresponding International Standard are not adopted as **JIS**.

The portions with continuous side lines or dotted underlines are the matters either modified from or not given in the corresponding International Standard. Annex JA is not given in the corresponding International Standard. A list of modifications with the explanations is given in Annex JB.

1 Scope

This Standard specifies the methods for determination of gaseous inorganic fluorine compounds in flue gas as fluoride ions and calculation of hydrogen fluoride concentration. In this Standard, the flue gas means the gas exhausted from flue, chimney, duct, etc. (hereafter referred to as “duct”) in combustion of wastes, smelting process of aluminium, manufacturing process of chemical fertilizer, manufacturing process of glass, etc. The gaseous inorganic fluorine compounds are those passing through the filter having not less than 99.5 % in collection efficiency of particles of 0.3 µm. The fluoride ion is the designation of F⁻.

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

ISO 15713:2006 *Stationary source emissions—Sampling and determination of gaseous fluoride content* (MOD)

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standard 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. 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 *General rules for chemical analysis*

JIS K 0095 *Methods for sampling of flue gas*

JIS K 0115 *General rules for molecular absorptiometric analysis*

JIS K 0122 *General rules for ion selective electrode method*

JIS K 0127	<i>General rules for ion chromatographic analysis</i>
JIS K 0557	<i>Water used for industrial water and wastewater analysis</i>
JIS K 1107	<i>Nitrogen</i>
JIS K 8005	<i>Reference materials for volumetric analysis</i>
JIS K 8032	<i>Acetonitrile (Reagent)</i>
JIS K 8034	<i>Acetone (Reagent)</i>
JIS K 8085	<i>Ammonia solution (Reagent)</i>
JIS K 8102	<i>Ethanol (95) (Reagent)</i>
JIS K 8121	<i>Potassium chloride (Reagent)</i>
JIS K 8150	<i>Sodium chloride (Reagent)</i>
JIS K 8180	<i>Hydrochloric acid (Reagent)</i>
JIS K 8223	<i>Perchloric acid (Reagent)</i>
JIS K 8288	<i>Trisodium citrate dihydrate (Reagent)</i>
JIS K 8295	<i>Glycerol (Reagent)</i>
JIS K 8355	<i>Acetic acid (Reagent)</i>
JIS K 8359	<i>Ammonium acetate (Reagent)</i>
JIS K 8371	<i>Sodium acetate trihydrate (Reagent)</i>
JIS K 8506	<i>Potassium bromide (Reagent)</i>
JIS K 8541	<i>Nitric acid (Reagent)</i>
JIS K 8548	<i>Potassium nitrate (Reagent)</i>
JIS K 8576	<i>Sodium hydroxide (Reagent)</i>
JIS K 8622	<i>Sodium hydrogen carbonate (Reagent)</i>
JIS K 8625	<i>Sodium carbonate (Reagent)</i>
JIS K 8799	<i>Phenolphthalein (Reagent)</i>
JIS K 8821	<i>Sodium fluoride (Reagent)</i>
JIS K 8863	<i>Boric acid (Reagent)</i>
JIS K 8866	<i>Sodium tetraborate decahydrate (Reagent)</i>
JIS K 8885	<i>Silicon dioxide (Reagent)</i>
JIS K 8951	<i>Sulfuric acid (Reagent)</i>
JIS K 9005	<i>Phosphoric acid (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) (Reagent)</i>
JIS R 3503	<i>Glass apparatus for chemical analysis</i>
JIS Z 8802	<i>Methods for determination of pH of aqueous solutions</i>
JIS Z 8808:1995	<i>Methods of measuring dust concentration in flue gas</i>

3 General matters

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 chromatographic method 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) General matters common to the ion selective electrode method shall be in accordance with **JIS K 0122**.
- f) 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.
- 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) 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.
- i) Apparatus and instruments to be used shall be provided with the specified functions.
- j) When disposing flue gas, or absorbent of the flue gas used for the analysis of hydrogen chloride, it shall be done with utmost care.

NOTE 1 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).

NOTE 2 The units of vol ppm and mg/m³ indicated in this Standard are the volume fraction and the mass concentration under the standard condition [273.15 K (0 °C), 101.32 kPa].

4 Type and summary of analysis methods

The type and summary of analysis methods shall be as given in table 1.

Table 1 Type and summary of analysis methods

Type of analysis method	Summary of analysis method			Applicable condition
	Outline	Sampling	Determination range vol ppm (mg/m ³)	
Lanthanum-alizarin complexon absorptiometric method	After absorption of fluorine compounds in sample gas into absorbent, pH is adjusted by adding buffer solution, lanthanum solution, alizarin complexon solution and acetone are added for colouring, and absorbance is measured.	Absorbing bottle method Absorbent: sodium hydroxide solution (0.1 mol/L) Liquid amount: 50 ml × 2 bottles Standard sampling volume: 40 L	1.2 to 14.8 (1.0 to 13.2)	According to 7.1.1.
Ion selective electrode method	After absorption of fluorine compounds in sample gas into absorbent, ion intensity adjusting buffer solution is added; and measurement is carried out using fluoride ion electrode.	Absorbing bottle method Absorbent: sodium hydroxide solution (0.1 mol/L) Liquid amount: 50 ml × 2 bottles Standard sampling volume: 40 L	7.4 to 737 (6.6 to 658)	According to 7.2.1.
Ion chromatographic method	After absorption of fluorine compounds in sample gas into absorbent, cation exchange resin is added to a definite amount of absorbent and air is passed as pretreatment. This solution is introduced into ion chromatograph and chromatogram is recorded.	Absorbing bottle method Absorbent: sodium hydroxide solution (0.1 mol/L) Liquid amount: 25 ml ^{a)} × 2 bottles or 50 ml ^{b)} × 2 bottles Standard sampling volume: 40 L	0.3 to 14.8 ^{c)} (0.3 to 13.2) 3.0 to 148 ^{d)} (2.7 to 132)	According to 7.3.1.
Notes ^{a)} Liquid amount for absorption when using absorbing bottle (100 ml in capacity). ^{b)} Liquid amount for absorption when using absorbing bottle (250 ml in capacity). ^{c)} In the case of employing the sample solution for analysis prepared by diluting 50 ml of absorbent 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, the amount of sample solution for analysis and the optimum range of the working curve of the sample gas. When determining the concentration over the determination range, after the collection using the absorbent, the sample solution for analysis shall be diluted to be within the determination range. ^{d)} In the case of employing the sample solution for analysis prepared by diluting 100 ml of absorbent 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, the amount of sample solution for analysis and the amount of titre.				

In addition, other than methods in table 1, the simultaneous analytical method of fluorine compounds, chlorine compounds and bromine compounds by ion chromatographic method is specified in Annex JA.

5 Sampling method of sample gas

5.1 Sampling of sample gas

The sampling position of sample gas shall be as follows. The point where representative sample gas can be sampled shall be selected. 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 reagent solutions

5.2.1 Reagent

Sodium hydroxide specified in **JIS K 8576** shall be used.

5.2.2 Preparation of reagent solution

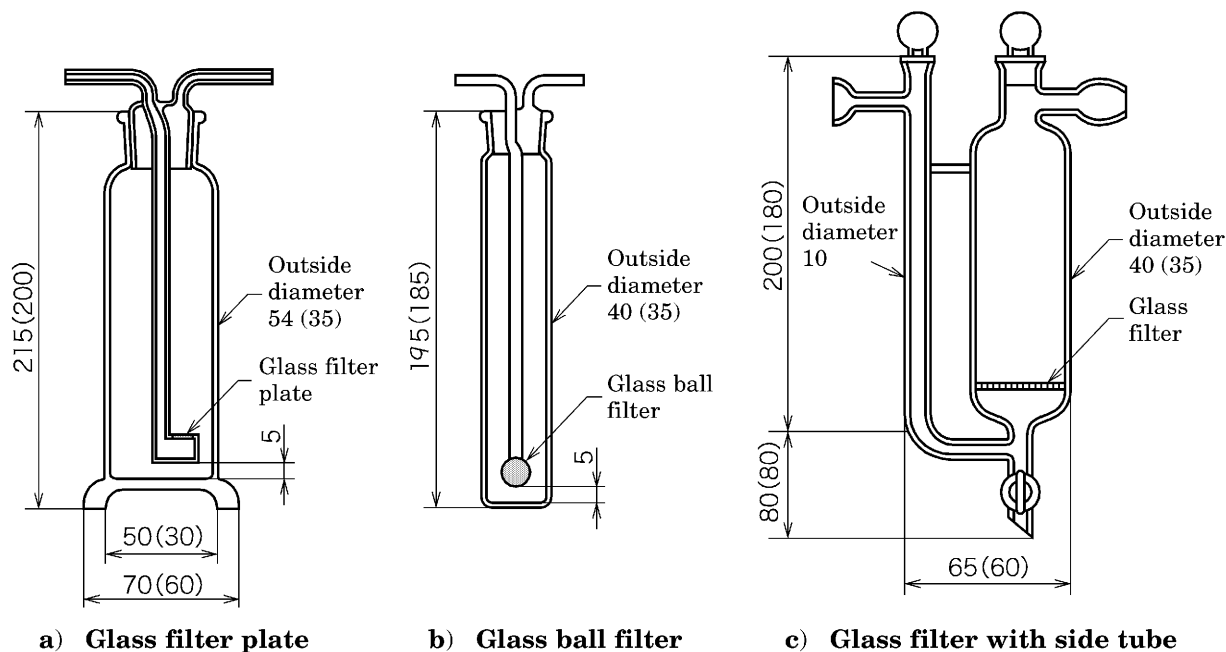
Sodium hydroxide solution (0.1 mol/L) shall be prepared in such a manner that 4.0 g of sodium hydroxide is taken and water is added to make the solution 1 L.

5.3 Instruments and apparatus

5.3.1 Absorbing bottle

- a) **For lanthanum-alizarin complexon absorptiometric method and ion selective electrode method** Two absorbing bottles (250 ml in capacity, see example in figure 1) shall be connected and used.
- b) **For ion chromatographic method** Two absorbing bottles (100 ml or 250 ml in capacity, see example in figure 1) shall be connected and used.

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



a) Glass filter plate

b) Glass ball filter

c) Glass filter with side tube

For the pore size of glass filter plate, glass ball filter and glass filter, the pore size symbol 1 or 2 specified in **JIS R 3503** shall be used.

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

5.3.2 Sampling apparatus of sample gas

The following conditions shall be satisfied as exemplified in figure 2.

- a) The sampling tube of sample gas (B) shall not be eroded by the corrosive gas in flue gas such as the stainless steel tube, borosilicate glass tube, tetrafluoro ethylene resin tube.
- b) For preventing the contamination of dust and the like into the sample gas, the end or other suitable place of sampling tube of sample gas (B) shall be filled with filtering medium. The filtering medium by which the chemical reaction with the component in flue gas is not produced in material, such as fibre and filter made of tetrafluoro ethylene resin shall be used. The filter made of cellulose material reacting with chemical components in flue gas, the glass fibre containing alkali content and the like shall not be used.
- c) For preventing the moisture condensation in the sample gas, the apparatus shall be so constructed that the part from the sampling tube of sample gas (B) to the absorbing bottle (F_1) can be heated. The piping shall be as short as possible, and if there is a possibility of moisture condensation, the part between the sampling tube (B) and the absorbing bottle (F_1) shall be heated at about 120 °C.
- d) For the connection part heated from the sampling tube of sample gas (B) to the three-way cock for flow path switch (P_1), a stainless steel tube, borosilicate glass tube, tetrafluoro ethylene resin tube, fluoro rubber tube, silicone rubber tube, etc. shall be used.
- e) The connection of each part in apparatus shall be assembled so as not to leak the gas. The gas leak shall be confirmed in accordance with 7.1.5 of JIS K 0095.

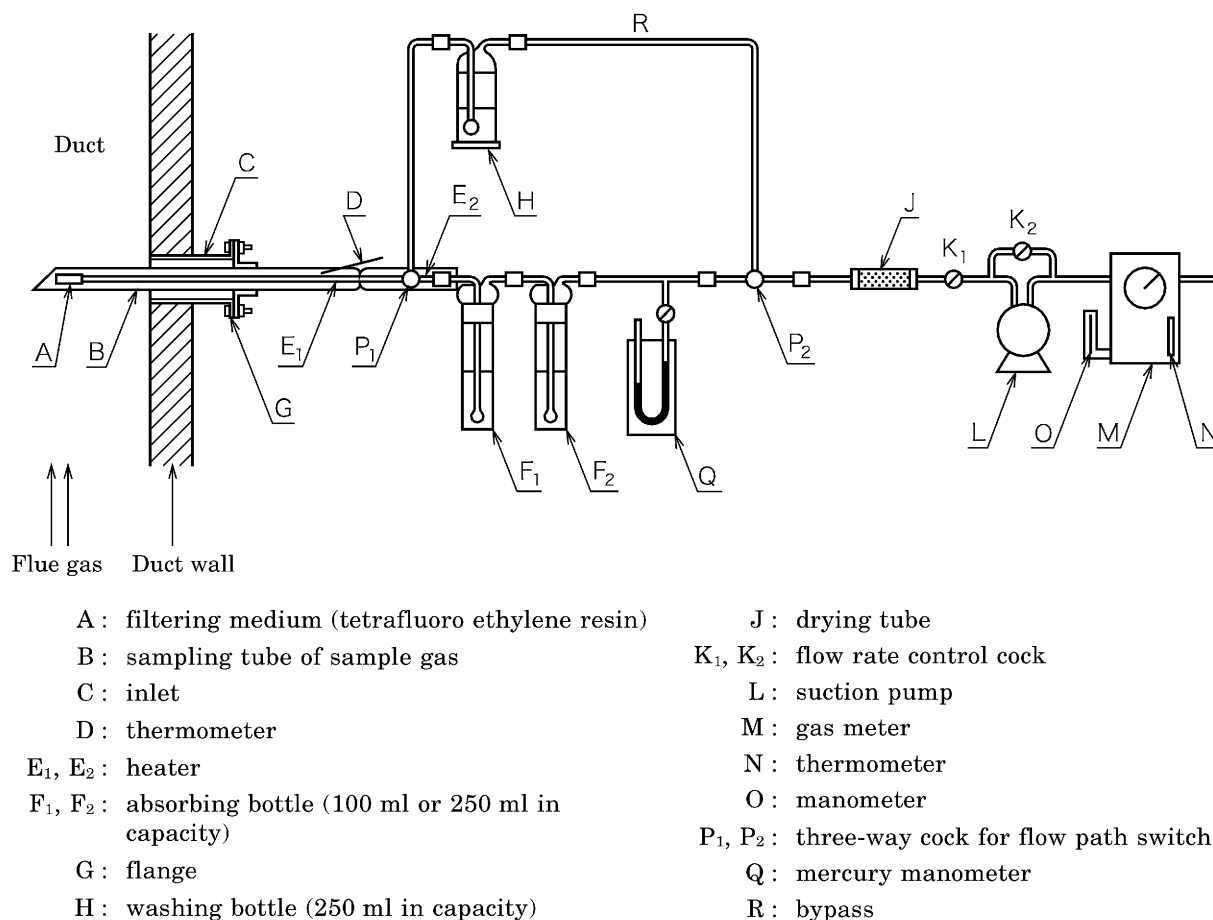


Figure 2 Example of sampling apparatus of sample gas

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) **For lanthanum-alizarin complexon absorptiometric method and ion selective electrode method** Put 50 ml of absorbent of 5.2.2 into each absorbing bottle (250 ml in capacity) (F₁ and F₂).
 - 2) **For ion chromatographic method** Put 25 ml of absorbent of 5.2.2 into each absorbing bottle (100 ml in capacity) (F₁ and F₂). In the case of using the absorbing bottles of 250 ml in capacity, put 50 ml of absorbent into each bottle.
- b) Turn the three-way cocks for flow path switch (P₁ and P₂) to bypass side, start the suction pump (L), and replace inside air between the sampling tube of sample gas (B) and the three-way cocks for flow path switch (P₁ and P₂) by the sample gas.

NOTE 1 The bypass (R) between P₁ and P₂ in figure 2 may be omitted, provided that the distance between the sampling tube of sample gas (B) and the absorption 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 (L), close the three-way cocks for flow path switch (P₁ and P₂) to stop the flow of gas. Then, read the indication (V₁) of a gas meter (M) to the nearest 0.01 L.
- d) Start the suction pump (L), turn the three-way cocks for flow path switch (P₁ and P₂) and pass the sample gas through the absorbing bottles (F₁ and F₂)¹⁾. At this time, adjust a flow rate to about 1 L/min by adjusting the flow rate control cocks (K₁ and K₂)²⁾. After sampling about 40 L of the sample gas³⁾, close the three-way cocks for flow path switch (P₁ and P₂), stop the suction pump (L), and read the indication (V₂) of a gas meter (M) to the nearest 0.01 L. At the same time, measure the temperature and the gauge pressure of the gas with the thermometer (N) and the manometer (O) of the gas meter (M). Also, measure the atmospheric pressure.
- e) If necessary, measure the moisture in the sample gas in accordance with clause 6 of JIS Z 8808:1995.

Notes ¹⁾ If there is a possibility that the absorbent is warmed, place the absorbing bottles in a cooling tank.

²⁾ When it has been known that hydrogen chloride will be completely absorbed in the absorbent (250 ml in capacity), the flow rate may be increased up to 2 L/min.

³⁾ This amount may be suitably increased/decreased according to the fluorine compounds concentration.

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_{SD}) or the wet-base gas volume (V_{SW}).

a) For dry-base gas volume

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

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

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

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

b) For wet-base gas volume

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

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

2) 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_{SD} : dry-base gas volume (L)

V_{sw} : wet-base gas volume (L)

V : gas volume measured by gas meter (L)

t : temperature at gas meter (°C)

P_a : atmospheric pressure (kPa)

P_m : gauge pressure at gas meter (kPa)⁴⁾

P_v : saturated water vapour pressure at t °C (kPa)

For P_v , the unit is converted from Pa to kPa based on table 2.

a : target gas collected in absorbent (mol)⁴⁾

b : gases other than target gas collected in absorbent (mol)⁴⁾

c : moisture amount obtained in clause 6 of **JIS Z 8808:1995** (mol)⁴⁾

273.15: absolute temperature equivalent to 0 °C (K)

101.32: pressure corresponding to 1 atmospheric pressure (kPa)

22.41: volume of 1 mol gas under standard condition (L)

Note ⁴⁾ In most cases, it can be neglected.

6 Preparation of sample solution for analysis

6.1 Reagents

The reagents shall be as follows.

- a) **Hydrochloric acid** That specified in **JIS K 8180**.
- b) **Nitric acid** That specified in **JIS K 8541**.
- c) **Phosphoric acid** That specified in **JIS K 9005**.
- d) **Perchloric acid** That specified in **JIS K 8223**.
- e) **Sodium hydroxide** That specified in **JIS K 8576**.
- f) **Silicon dioxide** That specified in **JIS K 8885** of which the particle size is 100 μm to 150 μm.
- g) **Ethanol (95)** That specified in **JIS K 8102**.
- h) **Phenolphthalein** That specified in **JIS K 8799**.

6.2 Preparation of reagent solutions

The preparation of reagent solutions shall be as follows.