

JAPANESE
INDUSTRIAL
STANDARD

Translated and Published by
Japanese Standards Association

JIS K 0106 : 2010

(JEMCA)

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

ICS 13.040.40;71.040.40

Reference number : JIS K 0106 : 2010 (E)

PROTECTED BY COPYRIGHT

K 0106 : 2010

Date of Establishment: 1968-09-01

Data of Revision: 2010-05-20

Date of Public Notice in Official Gazette: 2010-05-20

Investigated by: Japanese Industrial Standards Committee
Standards Board
Technical Committee on Environment and
Recycling Policy

JIS K 0106 : 2010, First English edition published in 2011-10

Translated and published by: Japanese Standards Association
4-1-24, Akasaka, Minato-ku, Tokyo, 107-8440 JAPAN

In the event of any doubts arising as to the contents,
the original JIS is to be the final authority.

© JSA 2011

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

Printed in Japan

NH/SW

PROTECTED BY COPYRIGHT

Contents

	Page
1 Scope	1
2 Normative references	1
3 Terms and definitions, and general matters	2
4 Types and summaries of analysis methods	2
5 Sampling method of sample gas	3
5.1 General	3
5.2 Reagents and preparation of reagent solutions	4
5.3 Implement and apparatus	4
5.4 Sampling procedure	6
5.5 Sampling amount of sample gas	7
6 Preparation of sample solution for analysis	8
7 Determination method	8
7.1 2, 2'-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) absorptiometry (ABTS absorptiometry)	8
7.2 4-pyridinecarboxylic acid-pyrazolone absorptiometry (PCP absorptiometry)	12
7.3 Ion chromatography (IC method)	14
8 Record of analysis result	18
8.1 Items to be recorded	18
8.2 Calculation of analysis value of flue gas	18
Annex A (normative) Absorptiometry by 3, 3'-dimethylbenzidinium dichloride (Absorptiometry by <i>o</i> -tolidine)	21
Annex B (informative) Detecting tube method	25

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) 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 0106 : 1995** is replaced with this Standard.

This **JIS** document is protected by the Copyright Law.

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, utility model right or application for registration of utility model after opening to the public which have technical properties. 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, utility model right or application for registration of utility model after opening to the public which have the said technical properties.

Methods for determination of chlorine in flue gas

1 Scope

This Japanese Industrial Standard specifies the methods for determination of chlorine in flue gas (hereafter referred to as "analysis method").

The methods specified in this Standard are influenced by the coexistence of the oxidizing gases such as bromine, iodine, ozone, chlorine dioxide or the reducing gases such as hydrogen sulfide, sulfur dioxide in sample gas. Therefore, the methods in this Standard shall only be applied if their influence is negligible, or can be removed. For the influence of nitrogen dioxide, the applicable condition given for each method shall be referred to.

In addition, in this Standard, the flue gas means the gas generated by combustion or chemical reaction or the like, which is exhausted into 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) indicated below 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 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 8180 *Hydrochloric acid (Reagent)*
- JIS K 8355 *Acetic acid (Reagent)*
- JIS K 8443 *Potassium cyanide (Reagent)*
- JIS K 8500 *N,N-Dimethylformamide (Reagent)*
- JIS K 8574 *Potassium hydroxide (Reagent)*
- JIS K 8622 *Sodium hydrogen carbonate (Reagent)*
- JIS K 8625 *Sodium carbonate (Reagent)*
- JIS K 8637 *Sodium thiosulfate pentahydrate (Reagent)*
- JIS K 8659 *Starch, soluble (Reagent)*
- JIS K 8913 *Potassium iodide (Reagent)*

JIS K 8951 *Sulfuric acid (Reagent)*

JIS K 9007 *Potassium dihydrogen phosphate (Reagent)*

JIS K 9020 *Disodium hydrogenphosphate (Reagent)*

JIS K 9548 *3-Methyl-1-phenyl-5-pyrazolone (Reagent)*

JIS Z 8808 *Methods of measuring dust concentration in flue gas*

3 Terms and definitions, and general matters

The main terms and definitions, and general matters of chemical analysis, sampling method for flue gas, absorptiometry and ion chromatography shall be in accordance with JIS K 0050, JIS K 0095, JIS K 0115 and JIS K 0127.

4 Types and summaries of analysis methods

The types and summaries of analysis methods shall be as shown 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 ^{a)} mg/m ³ (vol ppm)	
2, 2'-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) absorptiometry (ABTS methods)	Chlorine in sample gas is allowed to be absorbed into 2, 2'-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) absorbing solution, and then coloured. Its absorbance (400 nm) is measured, and the density of the sample gas is obtained.	Absorbing-bottle method Absorbing solution: ABTS solution (0.1 g/L) Quantity of absorbing solution: 20 ml × 2 Standard sampling volume: 20 L	0.10 to 2.0 (0.03 to 0.63)	Follow 7.1.1
4-pyridinecarboxylic acid-pyrazolone absorptiometry (PCP methods)	The sample solution for analysis is prepared by letting the chlorine in the sample gas be absorbed in <i>p</i> -toluene sulfonamide absorbing solution and changing to chloramine T. This solution is made to be cyanogen chloride by adding a small amount of potassium cyanite to it, then a colour is developed by 4-pyridinecarboxylic acid-pyrazolone solution, the absorbance (638 nm) is measured and the concentration of sample gas is obtained.	Absorbing-bottle method Absorbing solution: <i>p</i> -toluenesulfonamide solution (1.0 g/L) Quantity of absorbing solution: 20 ml × 2 Standard sampling volume: 20 L	0.25 to 5.0 (0.08 to 1.6)	Follow 7.2.1
Ion chromatography (IC method)	The sample solution for analysis is prepared by letting the chlorine in the sample gas be absorbed in <i>p</i> -toluene sulfonamide absorbing solution and changing to chloramine T. This solution is made to be cyanate ion by adding a small amount of potassium cyanate solution and potassium hydroxide solution, then it is measured by ion chromatography and the concentration of the sample gas is obtained.	Absorbing-bottle method Absorbing solution : <i>p</i> -toluenesulfonamide solution (1.0 g/L) Quantity of absorption: 20 ml × 2 Standard sampling volume: 20 L	1.3 to 25 (0.40 to 7.9)	Follow 7.3.1
NOTE 1 The units of mg/m³ and vol ppm indicated in this table mean the mass concentration and volume concentration under the standard condition [273.15 K (0 °C), 101.32 kPa]. NOTE 2 There are the 3,3'- dimethylbenzidine dichloride absorptiometry (<i>o</i>-tolidine absorptiometry) (see Annex A) and detecting tube method (see Annex B) in addition to the methods in this table. Note ^{a)} The ranges are set for a sample solution for analysis which has been prepared by diluting the absorbing solution (40 ml) with sample gas passed to 50 ml. The ranges of determination shown here are obtained from the standard sampling amount of sample gas, the sample solution for analysis and the optimum range of working curve. When the concentration exceeding the range of determination is measured, the sample solution for analysis should be diluted so as to be included in the range of determination.				

5 Sampling method of sample gas

5.1 General

When sampling the sample gas for analysis, choose the spot where representative

sample gas can be taken, sample the gas generally at least twice in a short time at the same sampling spot, and use each of them for analysis.

5.2 Reagents and preparation of reagent solutions

5.2.1 Reagents

The reagents shall be as follows.

5.2.1.1 **Water**, of A3 specified in JIS K 0557.

5.2.1.2 **Sulfuric acid**, specified in JIS K 8951.

5.2.1.3 **Potassium dihydrogenphosphate**, specified in JIS K 9007.

5.2.1.4 **Disodium dihydrogenphosphate**, specified in JIS K 9020.

5.2.1.5 **2,2'-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) diammonium (ABTS diammonium)**

5.2.1.6 ***p*-toluenesulfonamide**

5.2.2 Preparation of reagent solutions

The preparation of reagent solutions shall be as follows.

5.2.2.1 **Sulfuric acid (0.02 mol/L)** Add 1.2 ml of sulfuric acid into 1 L of water.

5.2.2.2 **Buffer solution (pH7)** Mix, in equal volume, the potassium dihydrogen phosphate of which 4.5 g is made to 1 L by dissolving with water (1/30 mol/L) and the disodium hydrogenphosphate of which 4.7 g is made to 1 L by dissolving with water (1/30 mol/L), then adjust it to pH7 with sodium hydroxide solution (2 g/L).

5.2.2.3 Absorbing solution

a) ABTS absorptiometry

ABTS solution (0.1g/L): Dissolve 0.1 g of 2, 2'-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) ammonium (ABTS ammonium) in 100 ml of buffer solution (pH7), and add sulfuric acid (0.02 mol/L) to make total 1 L. Storage period of this solution is 2 days in cold and dark place.

b) PCP absorptiometry and ion chromatography

***p*-toluenesulfonamide solution (1.0 g/L):** Add 0.1 g of *p*-toluenesulfonamide to 100 ml of water, heat it to 50 °C to 60 °C on a water bath, and after shaking sufficiently to dissolve, allow it to cool to room temperature.

5.3 Implement and apparatus

The implement and apparatus shall be as follows.

5.3.1 **Absorbing bottle**, as shown in figure 1 (capacity 50 ml), two of which are connected when used ^{1) 2)}.

5.3.2 **Sampling apparatus for sample gas**, constructed as shown in figure 2, and equipped with the following conditions:

a) For the sampling pipe of sample gas (B), a pipe made of a material which is resis-

tant to corrosive gas in flue gas such as chlorine shall be used, e.g. borosilicate pipe, silica glass pipe or polytetrafluoroethylene pipe.

- b) In order to prevent dust from mixing into sample gas, plug the end of the sampling pipe or other suitable place with filter medium ³⁾.
- c) In order to keep the moisture contained in sample gas from being condensed, the construction of the apparatus shall be such that the part from the gas sampling pipe to the cock (P₁) can be heated ⁴⁾.

To connect these parts, use commonly ground connecting pipe, silicone rubber pipe, or polytetrafluoroethylene pipe.

- d) Set up the whole apparatus so that it is free from leakage at any connection.

Notes ¹⁾ When it is confirmed that chlorine is completely captured, one absorbing bottle will be used.

²⁾ The presence of clogging, damage, etc. in the glass ball filter of absorbing bottle will influence the absorption of chlorine. Therefore, the absorbing bottle should be checked and confirmed for absence of clogging, damage, etc. before use. For checking of this purpose, 20 ml of water is poured in the absorbing bottle and aerated, and it is confirmed that the air is dispersed into small uniform bubbles, which indicates that there is no clogging or damage.

³⁾ Use a material which would not chemically react with the components in flue gas, such as silica wool, or alkali-free glass wool.

⁴⁾ Piping should be as short as possible, and where possibility of condensation of water is feared, the part from the sample gas pipe to the cock (P₁) shall be heated to about 120 °C.

Unit: mm

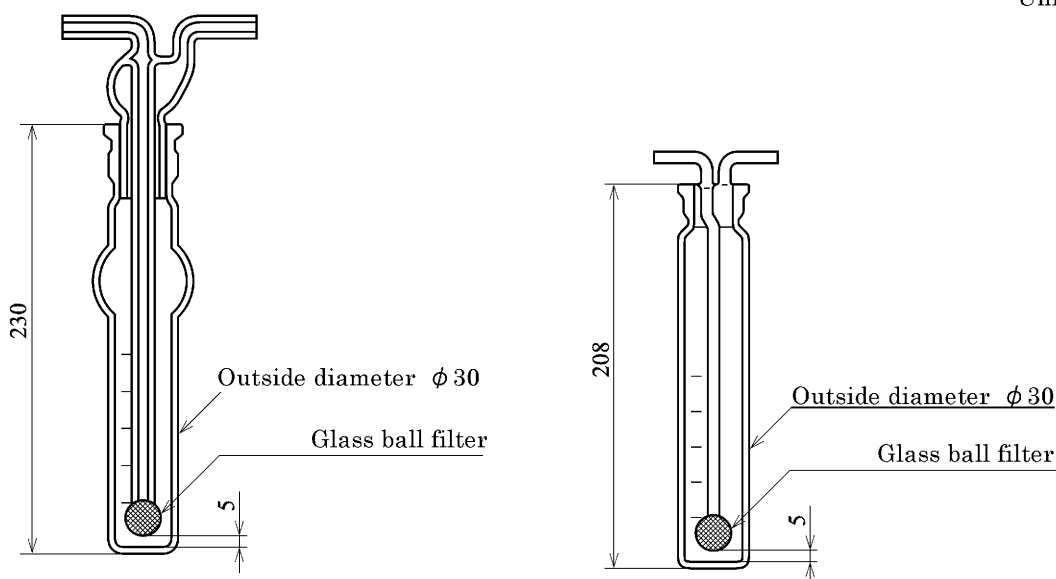


Figure 1 Example of absorbing bottle

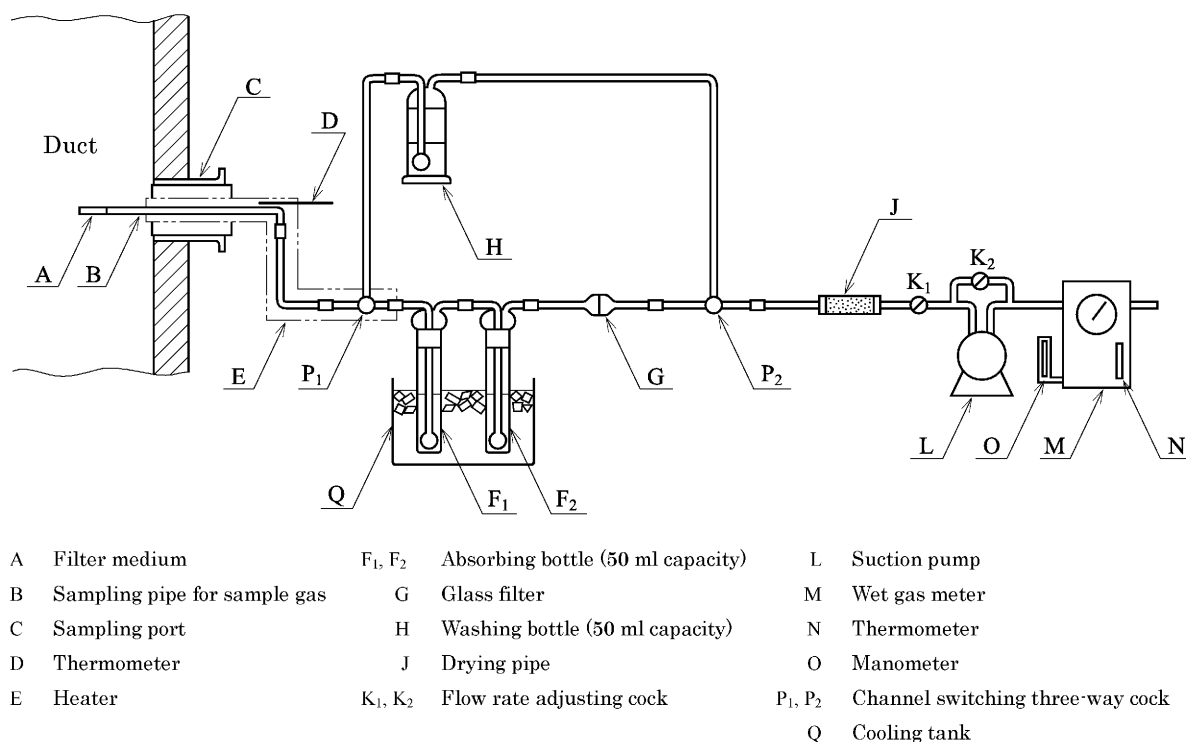


Figure 2 Example of sampling apparatus of sample gas

5.4 Sampling procedure

The sampling procedure shall be as follows. The symbols used below are in accordance with figure 2.

- Put 20 ml of either solution of **a)** or **b)** in 5.2.2.3, which is specified for each analysis method, into each of absorbing bottles (F₁, F₂)¹⁾.
- Set the absorbing bottles (F₁, F₂) in the cooling tank (Q) and connect to the gas sampling pipe ⁵⁾.
- Turn the channel switching three-way cock (P₁, P₂) to the bypass side, then operate a suction pump (L) to replace the inside of the part from the gas sampling pipe (B) to the three-way cock (P₁) with sample gas. Where the shortage of space due to such as narrowness of sampling site makes setting up of bypass introduction pipe impossible, the bypass may be omitted, provided that the conduit pipe between the sampling pipe and the three-way cock (P₁) is thoroughly replaced with sample gas.
- After stopping suction pump (L), turn the channel switching three-way cock (P₁, P₂) to the absorbing bottles (F₁, F₂) side. Then, read the indication (V₁) on the wet gas meter (M) to the nearest 0.01 L.
- Operate the suction pump (L) to pass the sample gas through the absorbing bottles (F₁, F₂). At this time, adjust the flow rate controlling cocks (K₁, K₂) to make the flow rate about 1 L/min [0.5 L/min ⁶⁾ in the case of 3, 3'-dimethylbenzidinium dichloride absorptiometry]. Take about 20 L (2.5 L in the case of 3, 3'-dimethylbenzidinium dichloride absorptiometry) ⁷⁾ of sample gas, then stop the suction pump (L) and shut the