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Methods for determination of pyridine
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 International Trade and Industry through deliberations at Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law:

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Methods for determination of pyridine in flue gas

1 Scope This Japanese Industrial Standard specifies the methods for determination of pyridine in flue gas.

Remarks 1 In this Standard, flue gas means ones which are generated by being accompanied with the manufacturing process of various kinds of chemicals, combustion, other chemical reaction, production process dealt with pyridines and exhausted out to flue, chimney or duct.

2 The normative references to this Standard are shown in Attached Table 1.

2 Common items The common items concerned with chemical analysis, sampling method of flue gas, molecular absorptiometric analysis and gas chromatograph shall be in accordance with JIS K 0050, JIS K 0095, JIS K 0115 and JIS K 0114 respectively.

3 Classification of analytical methods and their outlines Classification of analytical methods and their outline shall be as follows.

Table 1 Classification of analytical methods and their outlines

Classification of analytical methods	Outline of analytical method			Application requirement
	Summary	Sampling	Range of determination volppm	
Diaminostilbene-disulfonic acid absorptiometry	After absorbing pyridine in flue gas into diluted sulfuric acid, add 4,4'-diaminostilbene-2, 2'-disulfonic acid solution and cyanogen bromide solution to colour development. Measure its absorbance at 490 nm in wavelength and determine the pyridine.	Absorption bottle method Absorbent : 0.01 mol/l Sulfuric acid solution 20 ml×2	0.1 to [For 20 l of sample gas ⁽¹⁾]	According to 5.1.1
Gas chromatography	Absorb pyridine in flue gas which was collected in a collection bottle into sulfuric acid solution. After extracting it in carbon disulphide, determine the pyridine by the chromatogram obtained with gas chromatograph provided with a hydrogen flame ionization detector.	Vacuum flask method Absorbent : 0.01 mol/l Sulfuric acid solution 50 ml	0.6 to [For 1 l of sample gas ⁽²⁾]	

Notes ⁽¹⁾ It is the case of the sample solution in which 10 l to 20 l of sample gas is collected and the absorbent is diluted to 200 ml.

⁽²⁾ It is the case of the sample solution in which 1 l of sample gas is collect-

ed in a vacuum flask and extract into 1 ml of carbon disulfide.

4 Sampling method of gas Sampling method of gas shall be in accordance with methods of absorption bottle and vacuum flask. Sampling position of gas for analysis shall be selected the point where the typical gas is collected and , after collected at least two times in a same position within adjacent time , these gases shall be analyzed respectively .

4.1 Method with absorption bottle This method shall be applied for the absorptiometry.

4.1.1 Reagents and preparation of reagent solution

(1) Reagents

(a) **Water** A2 grade specified in JIS K 0557.

(b) **Sulfuric acid** Specified in JIS K 8951.

(2) Preparation of reagent solution (absorbent)

(a) **Absorbent : Sulfuric acid (Approx. 0.01 mol/l)** Dilute 1 g of sulfuric acid to 1 l with water.

4.1.2 Device and apparatus Device and apparatus shall be as follows:

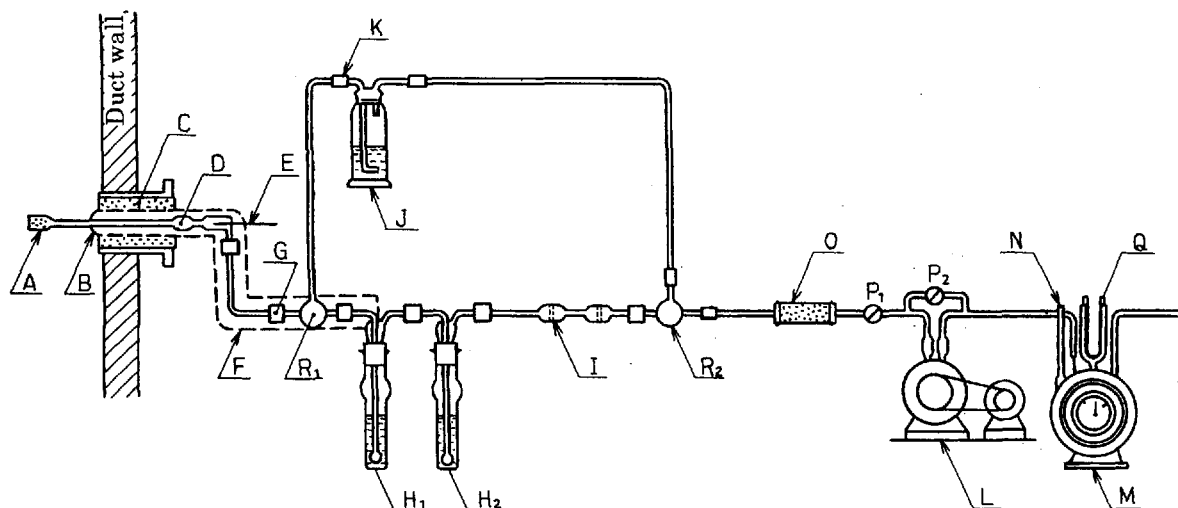
(1) **Absorption bottle** Connected two absorption bottles which have 100 ml in each volume as an example shown in Fig. 3 (a) (an example of absorption bottle) of JIS K 0095 shall be used.

(2) **Sampling apparatus** The apparatus shall be constructed as shown in Fig. 1 and provided with the following requirements.

(a) The sampling tube shall be made of such material which are not reacted chemically with pyridine, not adsorbed it in flue gas and have a greater heat-resistance for the temperature of flue gas. For example, glass tube, quartz tube, stainless steel tube and fluoride resin tube, etc. shall be used.

(b) In order to prevent against mixing-in of dust in flue gas, appropriate filter medium such as silica wool, etc. shall be put into the end part of gas sampling tube.

(c) For connecting part between the sampling tube and absorption bottle, glass grinding tube, silicon rubber tube shall be used.



A : Filter medium	H ₁ : No.1 absorption bottle	N : Thermometer
B : Sampling tube	H ₂ : No.2 absorption bottle	O : Desiccant (Silica gel)
C : Insulated material	I : Glass filter	P ₁ : Flow-rate adjusting cock
D : Fluorine-containing rubber tube	J : Gas washing bottle	P ₂ : Flow-rate adjusting cock
E : Thermometer	K : Silicone rubber tube	Q : Manometer
F : Heater	L : Suction pump	R ₁ : Passage change cock
G : Spherical ground joint	M : Gas meter	R ₂ : Passage change cock

Fig. 1 Sampling apparatus of gas with absorption bottle (an example)

4.1.3 Procedure of gas sampling The sampling of gas shall be carried out according to the following procedures. The symbols of the apparatus shown in the following sentences shall be in accordance with ones shown in Fig. 1.

- (1) Take 20 ml of the absorption solutions into each absorption bottle (H₁, H₂) separately.
- (2) After turning the passage change cock (R₁, R₂) to the side of bypass, start the suction pump (L) and substitute air in the piping (between B and R₁) with sample gas.
- (3) After stopping the suction pump (L), turn the passage change cock (R₁, R₂) to the side of absorption bottles (H₁, H₂) read off the indication (V₁) of gas meter (M) 0.01 l (liter) orders of magnitude.
- (4) Start the suction pump (L) and pass the sample gas into absorption bottles (H₁, H₂). Adjust the flow rate to approx. 0.1 l/min with the flow-rate adjusting cock (P₁, P₂) and take approx. 10 l of the sample gas.

Stop the suction pump (L), close the passage change cock (R₁, R₂) and read off the indication (V₂) of gas meter (M) 0.01 l orders of magnitude. At the same time, record indication value of thermometer (N), and manometer (Q). Atmospheric pressure shall be also measured.

- (5) According to necessity, measure the moisture in the sample gas according to 6 of JIS Z 8808.

- (6) **Calculation sampling volumes of the gas** A volume of dry gas (V_{SD}) or a volume of