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JIS K 0092 : 1998

**Methods for determination of  
mercaptan 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 mercaptan in flue gas

**1 Scope** This Japanese Industrial Standard specifies methods for determination of mercaptan ( thiol ) in flue gas.

**Remarks 1** In this Standard, flue gas means ones which are generated by being accompanied with chemical reaction, etc. 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 chromatographic analysis 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 outlines 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 vol ppm                                                        |                           |
| Dimethyl-phenylene diamine absorptiometry | After absorbing mercaptan in sample gas into the absorption solution, add mixture of N,N-dimethyl- <i>p</i> -phenylenediamine and iron (III) chloride, develop the colour and measure the absorbance (505 nm). | Absorption bottle method<br>Absorption solution: mercury (II) acetate solution<br>Volume : 20 ml × 2 | For 10 l of sampling volume : 0.5 to 10<br>For 100 l of sampling volume : 0.05 to 0.5 | In accordance with 5.1.1. |
| Gas chromatography                        | Collect the sample gas, introduce the gas into the gas chromatograph.<br>Determine mercaptan with chromatogram obtained.                                                                                       | Vacuum collecting bottle method or collecting bag method                                             | Not less than 0.05 (flame photometric detector)                                       |                           |

**4 Gas sampling method** Gas sampling method shall be as follows :

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.

Gas sampling method shall be according to absorption bottle method, vacuum collecting bottle method or collecting bag method.

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

#### 4.1.1 Reagents and preparation of reagent solution

##### (1) Reagents

- (a) **Water** A2 grade specified in JIS K 0557.
  - (b) **Acetic acid** Specified in JIS K 8355.
  - (c) **Mercury (II) acetate** Specified in JIS K 8369.
- (2) **Preparation of reagent solution (absorption solution)** Dissolve 50 g of mercury (II) acetate in 400 ml of water, add 25 ml of acetic acid and make up the volume to 1 l with water.

#### 4.1.2 Device and apparatus

- (1) **Absorption bottle** Absorption bottle of 50 ml as shown in Fig. 3 (a) of JIS K 0095 shall be used.
- (2) **Gas sampling apparatus** The gas sampling apparatus shall be in accordance with Fig. 2 (a) of JIS K 0095.

#### 4.1.3 Sampling of gas and preparation of sample solution

##### (1) Sampling of gas

- (a) Prepare two absorption bottles and put 20 ml of absorption solutions into them separately.
- (b) The suction flow rate of sample gas shall be approx. 1 l/min to 2 l/min. At the same time as the measurement of sample gas volume, measure the temperature and pressure of gas in gas meter.

Remarks : Sampling volume of gas shall be as shown in Table 2 according to the concentration of mercaptan in flue gas.

**Table 2 Sampling amount of gas**

| Concentration of mercaptan vol ppm | Sampling volume of gas l |
|------------------------------------|--------------------------|
| 0.05 to 0.5                        | 100                      |
| Over 0.5 to and excl. 10           | 10                       |

## (2) Preparation of sample solution

- (a) After sucking sample gas, transfer the solution <sup>(1)</sup> in all absorption bottles into a volumetric flask of 50 ml, wash the absorption bottle with absorption solution, combine the whole washings, add absorption solution up to the marked line and take it as the sample solution for determination.

Note <sup>(1)</sup> If hydrogen sulfide is coexisted, the solution turns white turbid.

**4.2 Vacuum collecting bottle method and collecting bag method** These methods shall be applied for gas chromatograph method.

### 4.2.1 Device and apparatus

- (1) **Vacuum collecting bottle** The glass bottle <sup>(2)</sup> of 1 l in inner volume shown in Fig. 4 (b-2) of JIS K 0095.

Note <sup>(2)</sup> The inner part shall be washed previously by acetone containing approx. 2 g/l of phosphoric acid and dried.

- (2) **Collection Bag** The bag of about 5 l in inner volume which is made of fluoro-resin <sup>(3)</sup> shown in Fig. 4 (c) of JIS K 0095. It shall be used by being installed with the sampling container as shown in Fig. 1.

Note <sup>(3)</sup> The bag made of the film of approx. 50  $\mu\text{m}$  in thickness shall be used.

- (3) **Gas sampling apparatus** The gas sampling apparatus shall be in accordance with Figs. 2 (c) and (e) of JIS K 0095.

**4.2.2 Sampling of gas** Sampling of gas shall be in accordance with 5.4 and 5.6 of JIS K 0095.

- (1) **For vacuum collecting bottle** Before sampling of gas, evacuate air in the collecting bottle previously through a dehumidifying tube, and take by sucking <sup>(5)</sup> the sample gas by passing through the introduction tube <sup>(4)</sup>.

Notes <sup>(4)</sup> For materials of sampling tube, introduction tube, etc., hard glass or fluoride resin, etc. shall be used.

<sup>(5)</sup> Because mercaptan is adsorbed in collecting bottle and is subject to air oxidation, it shall be rapidly determined after sampling.

- (2) **For collecting bag** Set the collecting bag in the airtight container as shown in Fig. 1 and connect with cock (F). Next, connect the introduction tube (A) with suction pump (E). After evacuating air contained in the collection bag, close the cock (F).

Exchange the suction pump for the connection of position as shown in Fig. 1 and put the introduction tube into the measuring place. Open the cocks (F, G), operate the suction pump, reduce pressure in the airtight container to take sample