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Methods for determination of mercury
in stack gas

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Methods for determination of mercury in stack gas

1 Scope This Japanese Industrial Standard specifies the methods for determination of gaseous mercury in stack gas.

Remarks 1 The concentration of mercury is expressed by the concentration of mercury in dry gas under standard condition.

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

2 Definitions For the purposes of this Standard, the definitions given in **JIS K 0211** and **JIS K 0095**, and the following definitions apply.

- (1) **stack gas** The gas exhausted from such as a flue, chimney or duct (hereafter referred to as "duct") due to burning of fuels, roasting of ores, metal refining, and treatment of sludge or waste.
- (2) **gaseous mercury** Generic term of mercury and mercury compounds which are present in stack gas as the style of gas.

3 Matters in common The matters commonly used in this Standard follow **JIS K 0050**, **JIS K 0095**, **JIS K 0121**, **JIS K 8001**, and **JIS Z 8808**.

4 Classification of methods for determination The methods for determination are classified into the following three.

- (1) **Wet absorption—atomic absorption spectrometry by reducing vaporization** Make gaseous mercury in sample be absorbed in sulfuric acidic solution of potassium permanganate, catch it, reduce mercury in absorption liquid, aerate through this solution to isolate mercury, and determine the mercury by atomic absorption spectrometry.

This method can be used for the determination of gaseous mercury existing in every stack gas. Determination range shall be from 1 ng to 1 000 ng as mercury.

- (2) **Gold-amalgam catching—atomic absorption spectrometry by heating vaporization** After catching mercury in sample gas as gold amalgam, heat the caught mercury in a heating vaporization furnace to vaporize mercury, and determine isolated mercury by atomic absorption spectrometry.

This method is used for the analysis of stack gas after treatment, and applicable to the determination of metal mercury in stack gas. Determination range shall be from 0.01 ng to 1 000 ng as mercury mass.

- (3) **Continuous measuring method** Introduce directly sample gas into an absorption cell, vaporize it by reduction, and determine continuously it by atomic absorption spectrometry.

This method is applicable to the stack gas exhausted from city garbage burning plant. Determination range shall be from 1 $\mu\text{g}/\text{m}^3$ to 5 mg/m^3 as mercury concentration.

5 Wet absorption—atomic absorption spectrometry by reducing vaporization

5.1 Sampling of sample gas The general matters on sampling method of sample gas follow JIS K 0095.

5.1.1 Sampling place Choose the place where distribution of flow rate is uniform.

5.1.2 Sampling device for sample Except the prescription in 4 of JIS K 0095, it shall be as follows. An example of sampling device for sample is shown in Fig. 1.

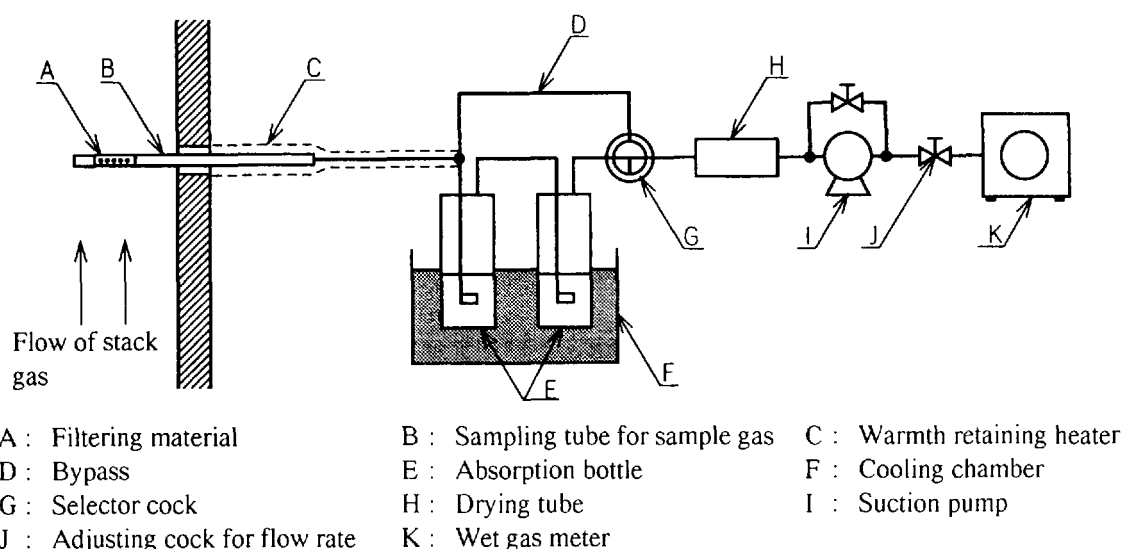


Fig. 1 Construction of sampling device for sample (example)

- (1) **Sampling tube** Made of borosilicate glass, quartz glass, titanium, or ceramics.
- (2) **Introducing tube** Made of tetrafluoroethylene resin. When heating is not needed, the introducing tube made of special vinyl chloride resin can be used.
- (3) **Collecting bottle** Follow 4.7 of JIS K 0095. Two absorption bottles each of which has 250 ml capacity are connected in series ⁽¹⁾.

Note ⁽¹⁾ Ordinarily, one bottle is enough.

5.1.3 Sampling Except the prescription in 5.1 of JIS K 0095, the sampling shall be as follows ⁽²⁾.

Note ⁽²⁾ In case of stack gas containing high concentration of sulfur dioxide as in ore roasting gas, wash the gas with absorption liquid prepared by potassium hydroxide.

- (1) **Absorption liquid** Mix in equivolume potassium permanganate solution (3 g/l) and sulfuric acid (1+15). Store it in a colored glass bottle.
- (2) **Sampling tube, introducing tube** In order to avoid the condensation of water in stack gas on sampling tube or introducing tube, if necessary, heat or keep its warmth.

- (3) **Absorption bottle** Absorption bottle shall be previously washed with nitric acid (1+9) and water, and be dried. Put 100 ml of absorption liquid, and cool it in a cooling chamber. Connect 2 absorption bottles in series.
- (4) **Sucked quantity** While sucking, the flow rate shall be 0.5 l/min to 1.0 l/min, and sucked quantity shall be about 20 l. However, don't suck in until the color of potassium permanganate of absorption liquid disappears.

5.1.4 Preparation of reagent for determination

- (1) **Reagents** If necessary, the reagents shall be for analysis for deleterious metal or for precision determination.
 - (a) **Sulfuric acid (1+1)** Take one unit of water in a beaker, and add gently one unit of sulfuric acid specified in **JIS K 8951** while cooling and stirring the water.
 - (b) **Potassium permanganate solution (50 g/l)** Dissolve 50 g of potassium permanganate specified in **JIS K 8247** in water, filtrate it through a glass filter (G4), and add water to make total 1 l. Store it in a colored glass bottle.
 - (c) **Hydroxylammonium chloride solution (200 g/l)** Dissolve 20 g of hydroxylammonium chloride specified in **JIS K 8201** in water to make total 100 ml.
 - (d) **Hydroxylammonium chloride solution (20 g/l)** Add several drops of sulfuric acid (1+1) into 10 ml of hydroxylammonium chloride solution (200 g/l), and add water to make total 100 ml.
- (2) **Implements** Implements shall be as follows.
 - (a) **Flask** Made of glass. 500 ml flask capable of being equipped with a ground-in reflux condenser.
 - (b) **Reflux condenser** With about 30 cm in length.
- (3) **Procedures** Procedures shall be as follows.
 - (a) Transfer the absorption liquid, in which sample gas has passed, into a flask. Wash the absorption bottle with a little quantity of hydroxylammonium chloride solution (20 g/l) and water, and put the washings into the flask ⁽³⁾.

Note ⁽³⁾ When sample gas does not contain organic gas, the procedure at (b) can be omitted. In this case, transfer the absorption liquid at (a) into a suitable beaker, and then carry out the procedures stated at (c) and thereafter.
 - (b) Attach a reflux condenser, heat gently it avoiding bumpings, and boil it for 1 h. During this heating, if color of potassium permanganate disappears⁽⁴⁾, lower the temperature down to about 60 °C, add 2 ml of potassium permanganate solution (50 g/l), boil it again, and repeat these procedures until the color of potassium permanganate lasts for about 10 min. Then cool the temperature of solution to 40 °C or lower.

Note ⁽⁴⁾ Even when the color of potassium permanganate disappeared, if manganese dioxide exists, the supplementation of potassium permanganate solution is not carried out.

- (c) While shaking the solution, add drop by drop hydroxylammonium chloride solution (200 g/l), and decompose the superfluous potassium permanganate (⁵).

Note (⁵) Avoid to add excessively hydroxylammonium chloride solution.

- (d) After cooling, transfer the solution into a 500 ml measuring flask, add water up to the marked line, and make it sample solution.
- (e) Take absorption liquid that is the same quantity as that used for sampling, carry out the procedures from (a) to (d), and make it the solution for blank test.

5.2 Method for determination

- (1) **Reagents** If necessary, the reagents shall be for analysis for deleterious metal or for precision analysis.

- (a) **Sulfuric acid (1+35)**

- (b) **Tin (II) chloride solution** Add 60 ml of sulfuric acid (1+20) onto 10 g of tin (II) chloride dihydrate specified in **JIS K 8136**, and heat it to dissolve while stirring them. After cooling, add water to make total 100 ml.

This solution shall be used up within one week from preparation.

- (c) **Solution for mercury dilution** Put 10 mg of *L*-cysteine into a 1 000 ml measuring flask, add water, shake to dissolve it, add 2 ml of nitric acid specified in **JIS K 8541**, and add water up to the marked line.
- (d) **Mercury reference solution (100 mgHg/l)** Put 67.7 mg of mercury (II) chloride specified in **JIS K 8139** into a 500 ml measuring flask, dissolve it in the solution for mercury dilution, then add the solution for mercury dilution up to the marked line, and make it stock solution. Store the stock solution in a refrigerator.

Reference solution shall be prepared by diluting this stock solution with the solution for mercury dilution.

- (2) **Apparatus** Apparatus is composed of an atomic absorption spectrometer, reduction vessel, absorption cell, air pump, flowmeter, drying tube, connecting tube, and others.

Fig. 2 shows an example of a closed circulation type, and Fig. 3 shows an example of open air-supplying type apparatus. The detail of each constructing part is as follows.