

## Methods for determination of mercury in LP gas

1. Scope This standard specifies the methods for determination of gaseous mercury in vaporized liquid liquefied petroleum (LP) gas.

- Remarks 1 The concentration of mercury is expressed as the concentration of mercury in dry gas under standard conditions.
- 2 The sample gas is obtained from a sample vessel. The sample vessel is made of stainless steel and pressure-resistant glass. The gas is introduced to the sample vessel as specified in JIS K 2240.

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) gaseous mercury Generic term for mercury and mercury compounds present as a gas in LP gas.
- (2) gaseous metallic mercury Metallic mercury present as a gas in LP gas.

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

- (1) Wet absorption—atomic absorption spectrometry by reducing vaporization Gaseous mercury in the sample is absorbed in a sulfuric acid solution of potassium permanganate and captured, then the mercury in the absorption liquid reduced and the solution aerated to isolate the mercury, and the mercury determined by atomic absorption spectrometry. This method can be used for the determination of gaseous mercury in LP gas. The determination range is 1~1,000ng as mercury.
- (2) Gold-amalgam catching—atomic absorption spectrometry by heating vaporization The mercury in the sample gas is captured as gold amalgam and heated in a heating vaporization furnace to vaporize, and the mercury thus isolated determined by atomic absorption spectrometry. This method can be used for the determination of gaseous metallic mercury in LP gas. The determination range is 0.01~1,000ng as mercury mass.

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Date of establishment: October 1, 2002

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Reference standards:

JIS K 0095-1994 Methods for sampling of flue gas

JIS K 0211-1987 Technical terms for analytical chemistry (general part)

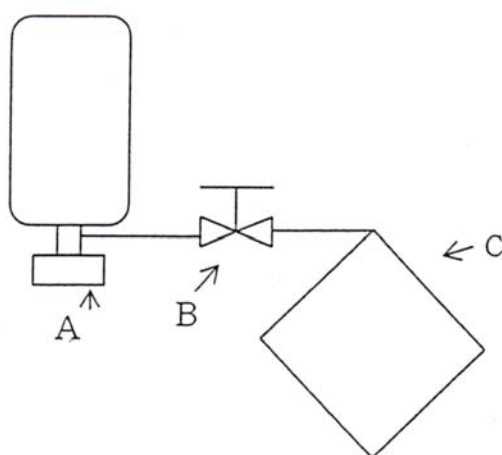
JIS K 0222-1997 Methods for determination of mercury in stack gas

JIS K 2240-1991 Liquefied petroleum gas (LP gas)

(3) Gold-amalgam catching—atomic fluorescent spectrometry by heating vaporization

The mercury in the sample gas is captured as gold amalgam and heated in a heating vaporization furnace to vaporize, and the mercury thus isolated determined by atomic fluorescent spectrometry. This method can be used for the determination of gaseous metallic mercury in LP gas. The determination range is 0.01~1,000ng as mercury mass.

4. Vaporization of gasification of sample [fluoride resin bag (Tedlar Bag, etc.)] Gas is extracted through a pressure reduction cock into a fluoride resin bag of at least 20l as shown in Fig. 1. The gas is extracted from the liquid phase of the sample vessel and extracted so that no vaporization occurs at the mouth of the vessel.



**Fig. 1 Gasification of sample (example)**

A: Sample vessel

B: Pressure reduction cock

C: Fluoride resin bag

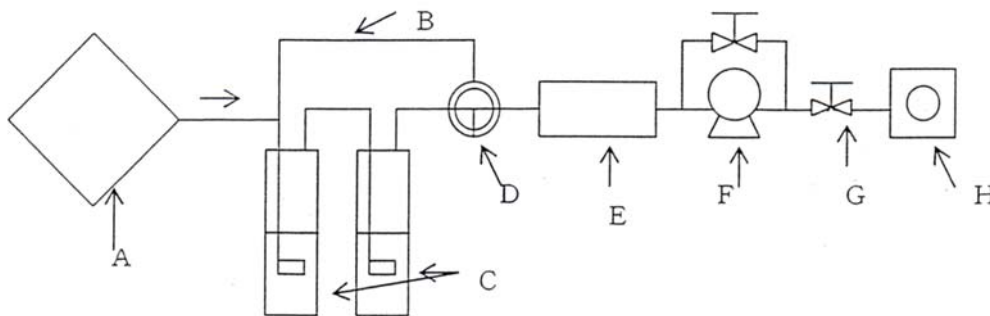
5. Wet absorption—atomic absorption spectrometry by reducing vaporization

5.1 Sampling of sample gas

The general matters on sampling method of sample gas are specified in JIS K 0095.

5.1.1 Sampling device for sample

Except where specified in 4. Sampling device for sample of JIS K 0095, the construction of the sampling device is as follows. An example of a sampling device for sample is shown in Fig. 2.



**Fig. 2 Construction of sampling device for sample (example)**

A: Fluoride resin bag	B: Bypass	C: Absorption bottle
D: Selector cock	E: Drying tube	F: Suction pump
G: Flow adjustment cock	H: Integrating gas meter	

- (1) Conduit: Made of tetrafluoroethylene resin. A conduit made of special vinyl chloride resin may also be used.
- (2) Absorption bottle: Two absorption bottles each of 250ml capacity are connected in series. The absorption bottles are washed with nitric acid (1+9) and water and dried in advance. 100ml of absorption liquid is added. The two absorption bottles are connected in series.
 

Note 1 Unless there is a leak at a later stage, one absorption bottle is sufficient.
- (3) Suction pump: Air pump capable of controlling flow at between 0.5~1.0l.
- (4) Integrating gas meter: Meter made of anticorrosive material with a minimum scale of at least 0.01l (approximately 1l per rotation).
- (5) Absorption liquid: Equivolumes of potassium permanganate solution (3g/l) and sulfuric acid (1+15) are mixed, and the absorption liquid stored in a colored glass bottle.
- (6) Suction quantity: The flow rate is maintained at 0.5~1.0l/min during suction, and a quantity of approximately 20l sucked. However, suction must not be continued until the color of the potassium permanganate in the absorption liquid disappears.

### 5.1.2 Preparation of reagent for determination

- (1) Reagents: The reagents may if necessary be used for analysis for deleterious metals or for precision determination.
  - (a) Sulfuric acid (1+1): One unit of sulfuric acid specified in JIS K 8951 is added gradually to one unit of water in a beaker while cooling and stirring the water.
  - (b) Potassium permanganate solution (50g/l): 50g of potassium permanganate specified in JIS K 8247 is dissolved in water, filtrated through a glass filter (G4), and water added to make a total of 1l. The solution is stored in a colored glass bottle.
  - (c) Hydroxylammonium chloride solution (200g/l): 20g of hydroxylammonium chloride specified in JIS K 8201 is dissolved in water to make a total of 100ml.

- (d) Hydroxylammonium chloride solution (20g/l): Several drops of sulfuric acid (1+1) are added to 10ml of hydroxylammonium chloride solution (200g/l), and water added to make a total of 100ml.
- (2) Implements: The implements used are as follows:
- (a) Flask: 500ml glass flask capable of being equipped with a ground-in reflux condenser.
- (b) Reflux condenser: Approximately 30cm in length.
- (3) Procedure: The procedure followed is as follows:
- (a) The absorption liquid into which sample gas has passed is transferred into a flask. The absorption bottle is washed with a small quantity of hydroxylammonium chloride solution (20g/l) and water, and the washings added to the flask.<sup>2</sup>
- Note 2 If the sample gas does not contain organic gas, step (b) may be omitted. In this case, the absorption liquid at step (a) is transferred to a suitable beaker, and the procedure continued from step (c).
- (b) A reflux condenser is attached, and the solution gently heated avoiding bumping and boiled for 1 hour. If during the color of potassium permanganate disappears, the temperature is lowered to about 60 °C, 2ml of potassium permanganate solution (50g/l) added, and the solution boiled again. This procedure is repeated until the color of potassium permanganate lasts for about 10 minutes. The solution is then cooled to 40 °C or lower.
- Note 3 If there is manganese dioxide present despite the disappearance of the color of potassium permanganate, supplementation of the potassium permanganate solution is not performed.
- (c) Hydroxylammonium chloride solution (200g/l) is added drop by drop while shaking the solution to decompose the superfluous potassium permanganate.<sup>4</sup>
- Note 4 Do not add excessive hydroxylammonium chloride solution.
- (d) After cooling, the solution is transferred to a 500ml measuring flask and water added up to the marked line to make the sample solution.
- (e) The same quantity of absorption liquid as that used for sampling is taken and procedures (a) to (d) performed to make the solution for blank testing.

## 5.2 Determination method

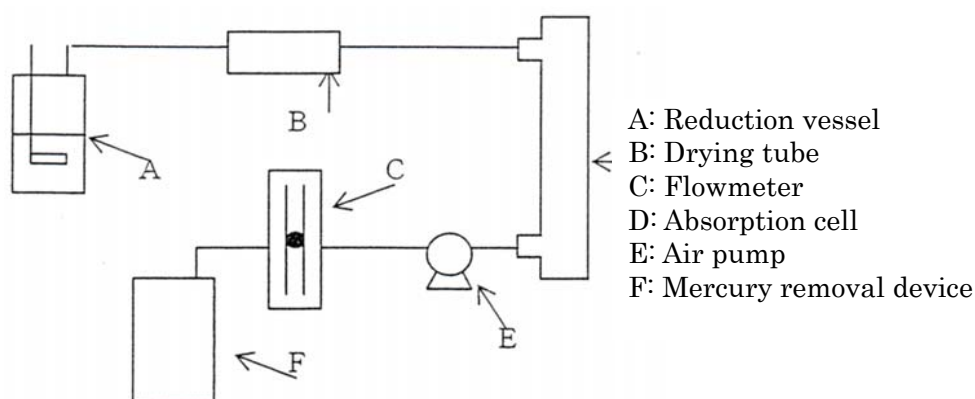
- (1) Reagents: If necessary, the reagents may be used for analysis for deleterious metals or for precision analysis.
- (a) Sulfuric acid (1+35)
- (b) Tin (II) chloride solution 60ml of sulfuric acid (1+20) is added to 10g of tin (II) chloride dihydrate specified in JIS K 8136, and heated to dissolve while stirring. After cooling, water is added to make a total of 100ml. This solution should be used within one week of preparation.
- (c) Solution for mercury dilution 10mg of L-Cysteine is put into a 1,000ml measuring flask, water added and the flask shaken to dissolve the L-Cysteine, 2ml of nitric acid specified in JIS K 8541 added, and water then added up to

the marked line.

- (d) Mercury reference solution (100mgHg/l) 67.7mg of mercury (II) chloride specified in JIS K 8139 is put into a 500ml measuring flask and dissolved in the solution for mercury dilution, and then the solution for mercury dilution added up to the marked line to make the stock solution. The stock solution is stored in a refrigerator. The reference solution is prepared by diluting this stock solution with the solution for mercury dilution.

- (2) Apparatus: The apparatus used includes an atomic absorption spectrometer, reduction vessel, absorption cell, air pump, flowmeter, drying tube and connecting tube.

Fig. 3 shows an example of an open air-supplying type apparatus. The details of the constituent parts are as follows.



**Fig. 3 Construction of open air-supplying type apparatus (example)**

- (a) Atomic absorption spectrometer: Atomic absorption spectrometer or atomic absorption spectrometer for mercury.
- (b) Reduction vessel: Aeration tube with filter for bubbling. Capacity is as specified by the measuring apparatus.
- (c) Absorption cell: A tube measuring 100~300mm in length made of quartz glass, glass, or plastic (that does not adsorb mercury) with both ends covered with a quartz glass window.
- (d) Air pump: Diaphragm pump capable of pumping 0.2~3l/min of air, or an air pump of equivalent performance.
- (e) Flowmeter: Capable of measuring flow rate of 0.2~3l/min.<sup>5</sup>  
Note 5 The flow rate of the air pump is preadjusted to determine the optimum flow rate.
- (f) Drying tube: Thermoelectronic refrigeration type, or straight tube or U tube packed with granular-type desiccant.<sup>6</sup>  
Note 6 If the temperature in the absorption cell is designed to be about 10°C higher than ambient, the drying tube is unnecessary.
- (g) Connecting tube: Made of soft vinyl-chloride resin.
- (h) Mercury removal device: Gas washing bottle into which sulfuric acid (1+4)

containing potassium permanganate (50g/l) has been put, or active carbon for mercury removal.

(3) Procedure: The procedure used is as follows:

(a) A suitable quantity of sample solution<sup>7</sup> is taken in a reduction vessel, and sulfuric acid (1+35)<sup>8</sup> added to the sample solution.

Note 7 The quantity of sample solution to be taken is determined according to the measuring apparatus.

Note 8 The quantity of sulfuric acid to be added is determined according to the measuring apparatus.

Remarks A sample containing large quantities of chloride ions may give a positive error because, when preparing the sample solution, the chlorine absorbs light produced from chloride ions by means of oxidation resulting from the addition of potassium permanganate. In this case, an excess quantity of hydroxylammonium chloride solution is added to reduce the chlorine. The chlorine present in the reduction vessel is expelled in advance using nitrogen.

(b) A quantity of tin (II) chloride solution 1/20th that of the solution specified in (a) is added promptly, the air pump activated to cause air to flow at the predetermined optimum rate<sup>9</sup>, and the mercury generated introduced into the absorption cell.

Note 9 Because the optimum flow rate differs depending on the type of apparatus used, the optimum flow rate must be determined in advance.

(c) Absorption at a wavelength of 253.7nm is measured.<sup>10</sup>

Note 10 The rate of reaction can differ depending on the sample, and so the integrated value of the absorption peak should be measured.

(d) For the blank test solution, a quantity of solution of the same quantity as the sample solution is taken and steps (a) to (d) performed to obtain the indicated value of absorption and correct the indicated value obtained for the sample.

(e) Using a working curve, the mass of mercury in the sample is obtained and the mercury concentration in the sample gas calculated according to the following formula:

$$C = A \times \frac{v}{v_l} \times \frac{1}{V_s}$$

where

C: concentration of mercury ( $\mu\text{g}/\text{Nm}^3$ )

A: mass of mercury obtained on the working curve (ng)

v: volume of sample solution (mL)

$v_l$ : volume of aliquot taken from sample solution (mL)

$V_s$ : quantity of sample gas taken (NL)

$$V_s = V \times \frac{273.15}{273.15 + t} \times \frac{P_a + P_m - P_v}{101.32}$$

where

$V$ : volume of gas measured by multiplication gas meter (L)

$T$ : temperature in multiplication gas meter (degrees Celcius)

$P_a$ : atmospheric pressure (kPa)

$P_m^{11}$ : gauge pressure in multiplication gas meter (kPa)

$P_v$ : saturated steam pressure at  $t$  degrees Celcius (kPa)<sup>12</sup>

Note 11 Negligible in most cases.

Note 12 Refer to the attached table 1.1 Maximum vapour pressure of water, as per JIS Z 8806 (2001).

- (f) To prepare the working curve, take stepwise several quantities of the mercury reference solution into the reduction vessels,<sup>13</sup> add respectively water and sulfuric acid (1+35) of the same volumes as added to sample solution at (a), and then perform steps (a) to (d).

Carry out steps (a) to (d) on the water and sulfuric acid (1+35) used above to obtain a blank-test value and correct the indicated value. Plot the relation curve between the corrected value and mass of mercury to give the working curve. Prepare the working curve when the sample is measured.

Note 13 These quantities of mercury reference solution taken differ depending on the apparatus, but should result in  $1\mu\text{g}$  or less as mercury.

## 6. Gold-amalgam catching—atomic absorption spectrometry by heating vaporization

### 6.1 Sampling of sample gas

#### 6.1.1 Reagents

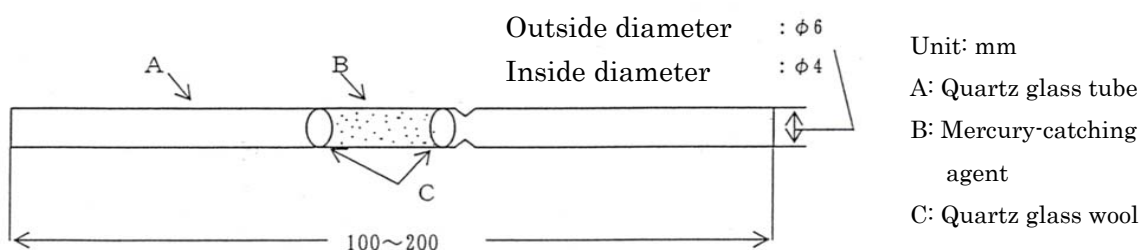
Catching agent of mercury: To 3g of diatomaceous earth with a grain size of  $420\sim 590\mu\text{m}$  is added the solution prepared by dissolving 1g of tetrachloroauric (III) acid specified in JIS K 8127 in  $20\sim 30\text{ml}$  of water, and the two mixed to make uniform. The product is then heated at about  $80^\circ\text{C}$  to dry, put in a tubular furnace, and heated at about  $800^\circ\text{C}$  for 30 min while being ventilated with air.

6.1.2 Sampling device for sample: An example of a sampling device for sample is shown in Fig. 5.

- (1) Conduit: The tube should be made of tetrafluoroethylene resin. A conduit made of special vinyl chloride resin may also be used.

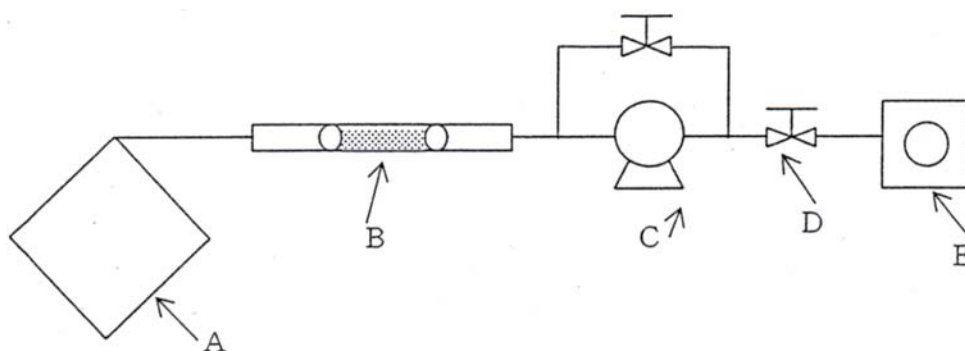
- (2) Mercury-catching tube: Fig. 4 shows an example of a mercury-catching tube. As shown in Fig. 5, an indented quartz glass tube is packed in order with quartz glass wool,  $80\sim 200\text{mg}$  of mercury-catching agent, and quartz glass wool. The mercury-catching tube is heated at  $600\sim 800^\circ\text{C}$  for 5 minutes through which carrier gas is caused to flow at a rate of  $0.2\sim 0.5\text{l/min}$ , and then placed in a glass test tube, which is closed hermetically with a stopper made of butyl rubber and stored. The storage limit is 6 months from preparation.

(3) Integrating gas meter: Meter made of anticorrosive material with a minimum scale of at least 0.01l (approximately 1l per rotation).



**Fig. 4 Mercury-catching tube (example)**

Gas in the fluoride resin pack is collected in the mercury-catching tube by the following apparatus:



A: Fluoride resin bag      B: Mercury-catching tube      C: Suction pump  
 D: Flow adjustment pump      E: Integrating gas meter

### 6.1.3 Sampling

Using the sampling device, the sample gas is passed through the mercury-catching tube at a rate of 0.5~1.0L/min for a specified duration<sup>14</sup>, and the sample collected. After sampling, the catching tube is swiftly put into a glass test tube, which is closed hermetically with a stopper made of butyl rubber. To measure the value for the blank test, a mercury-catching tube through which no sample gas has passed is prepared.

Note 14 The passing rate of the sample gas is adjusted to result in  $1\mu\text{g}$  or less as mercury.

## 6.2 Determination method

### 6.2.1 Reagents and apparatus

(1) Buffer solution: Neutral phosphate pH reference solution is used as specified in

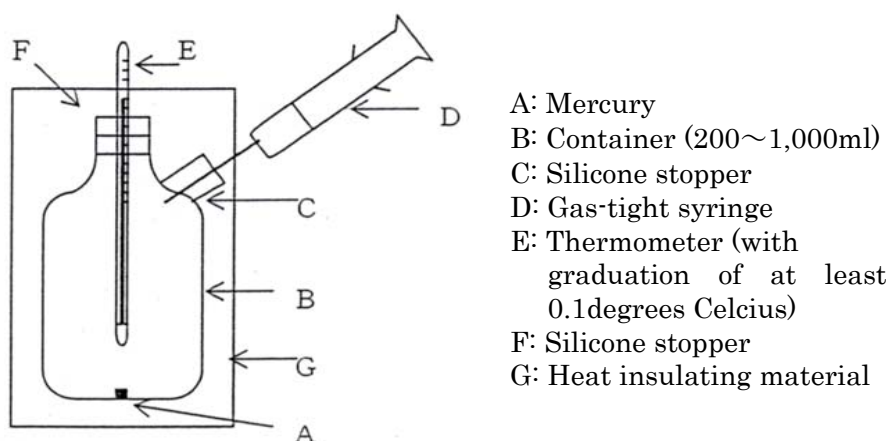


JIS Z 8802.

- (2) Carrier gas: Air or inactive gas from which mercury has been removed.
- (3) Mercury reference gas: Fig. 6 shows an example of the apparatus used to prepare the mercury reference gas.

A small amount of mercury is placed in a container and allowed to stand in a room at constant temperature for at least 1 hour. The temperature in the container is read with a thermometer, and the concentration of mercury in the container found using Table 1.

An aliquot of the gas is taken using a gas-tight syringe for use as the mercury reference gas.



**Fig. 6 Apparatus for preparation of mercury reference gas (example)**

### 6.2.2 Apparatus <sup>15</sup>

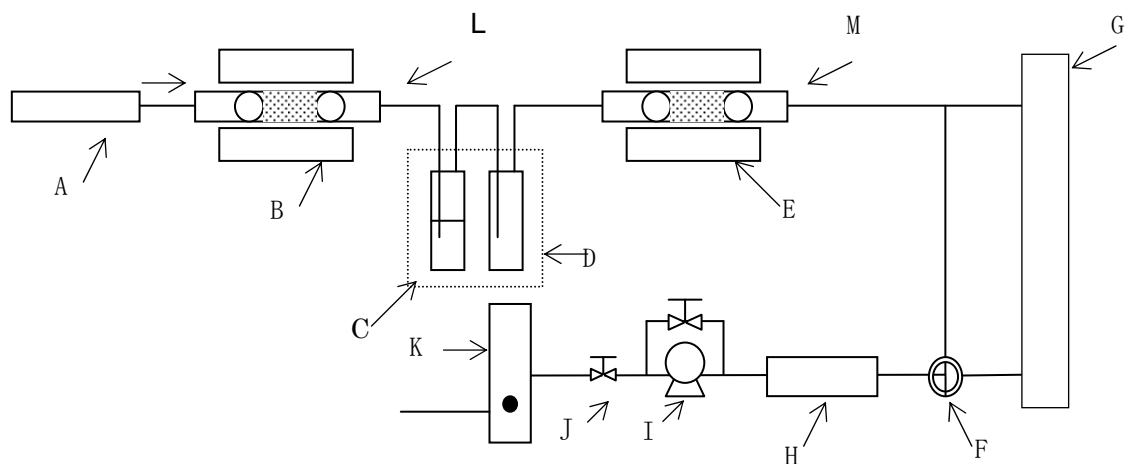
The apparatus consists of equipment including a mercury-removal filter, vaporization heating furnace, dehumidifying bottle, gas washing bottle, mercury re-catching furnace, selector cock, absorption cell, suction pump, flowmeter, mercury removal device, and mercury re-catching tube.

The construction of the apparatus is exemplified in Fig. 7.

Note 15 An automated version of this apparatus capable of continuous measurement may be used.

- (1) Mercury-removal filter: Mercury-catching tube or quartz glass tube packed with approximately 840 $\mu$ m of active carbon.
- (2) Vaporization heating furnace: A tubular furnace to heat the mercury catching tube. It should be capable of raising the temperature to 800<sup>o</sup> C within 3 minutes, and be cooled by air fan after heating has finished.
- (3) Gas washing bottle: Gas washing bottle containing 20~100ml of buffer solution.
- (4) Dehumidifying bottle: Empty gas washing bottle. Cooled together with (3).
- (5) Mercury-removal device: As specified in 5.2.(2)(h).
- (6) Mercury re-catching furnace: Electric tubular furnace capable of heating

- mercury-catching tube from 150 °C to 500~800 °C in 1 minute.
- (7) Mercury re-catching tube: As specified in 6.1.2(2).
- (8) Absorption cell: As specified in 5.2.(2)(c).



**Fig. 7 Construction of vaporization heating apparatus (example)**

- |                             |                                 |                          |
|-----------------------------|---------------------------------|--------------------------|
| A: Mercury-removal filter   | B: Vaporization heating furnace | C: Gas-washing bottle    |
| D: Dehumidifying bottle     | E: Mercury re-catching furnace  | F: Selector cock         |
| G: Absorption cell          | H: Mercury-removal device       | I: Suction pump          |
| J: Flow adjustment cock     | K: Flowmeter                    | L: Mercury-catching tube |
| M: Mercury re-catching tube |                                 |                          |

6.2.3 Procedure: The procedure used is as follows:

- (1) The mercury-catching tube<sup>16</sup> is placed inside the vaporization heating furnace, heated at 600~800 °C for about 3 minutes while carrier gas is flowing at a flow rate of 0.2~0.5L/min, and the mercury generated conducted into the re-catching tube<sup>17</sup>.

Note 16 The speed with which mercury is desorbed differs depending on the packing method, and so a re-catching tube with constant performance should be used for catching.

17 Some organic compounds indicate absorption at the wavelength of mercury measurement. To eliminate these organic compounds, the re-catching tube should be heated at about 150 °C.

- (2) The selector cock is switched to introduce carrier gas into the absorption cell.
- (3) The re-catching tube is heated at a constant temperature of 500~800 °C, and the mercury generated introduced into the absorption cell.
- (4) The peak height or peak area given by absorption at a wavelength of 253.7nm is measured.
- (5) Using the mercury-catching tube for blank-test value measurement, steps (1) to (4) are performed to obtain the value for the blank test.

- (6) The mass of mercury is found on the working curve, and the concentration of mercury in the sample gas calculated according to the following formula. The concentration is calculated to four significant figures and round to three significant figures according to JIS Z 8401.

$$C = (A - A_0) \times \frac{1}{V_s}$$

where

$C$ : concentration of mercury ( $\mu\text{g}/\text{m}^3$ )

$A$ : mass of mercury obtained on working curve (ng)

$A_0$ : value of blank test (ng)

$V_s$ : volume of sample gas taken (l)

$$V_s = V \times \frac{273.15}{273.15 + t} \times \frac{P_a + P_m - P_v}{101.32}$$

where

$V$ : volume of gas measured by multiplication gas meter (L)

$T$ : temperature in multiplication gas meter (degrees Celcius)

$P_a$ : atmospheric pressure (kPa)

$P_m^{18}$ : gauge pressure in multiplication gas meter (kPa)

$P_v$ : saturated steam pressure at  $t$  degrees Celcius (kPa)<sup>19</sup>

Note 18 Negligible in most cases.

Note 19 Refer to the attached table 1.1 Maximum vapour pressure of water, as per JIS Z 8806 (2001).

- (7) The working curve is prepared as follows:
- The mercury-removal filter is detached, and 0.1~10ml of mercury reference gas taken stepwise using a gas-tight syringe and introduced into mercury-catching tubes while a suction pump is operated.
  - Steps (1) to (5) are performed
  - After introducing the reference gas, the mercury-removal filter is attached
  - The relation curve between the mass of mercury and measured value is plotted to give the working curve. The working curve is prepared when the sample is determined.

## 7. Gold-amalgam catching—atomic absorption fluorescent spectrometry by heating vaporization

### 7.1 Sampling of sample gas

#### 7.1.1 Reagents

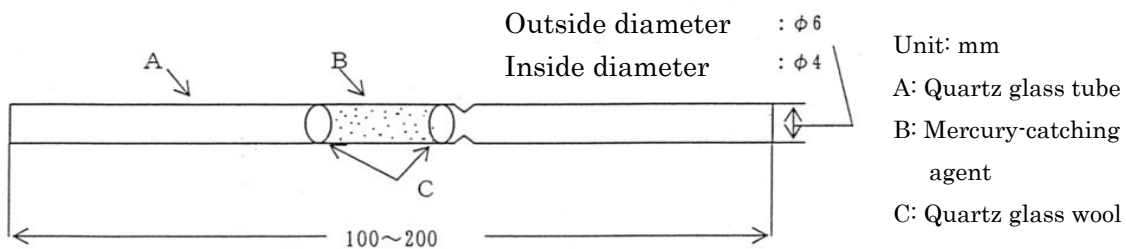
Catching agent of mercury: To 3g of diatomaceous earth with a grain size of 420~590 $\mu\text{m}$  is added the solution prepared by dissolving 1g of tetrachloroauric (III) acid specified in JIS K 8127 in 20~30ml of water, and the two mixed to make uniform. The product is then heated at about 80 $^{\circ}\text{C}$  to dry, put in a tubular furnace, and heated at about 800 $^{\circ}\text{C}$  for 30 min while being ventilated with air.

7.1.2 Sampling device for sample: An example of a sampling device for sample is shown in Fig. 9.

(1) Conduit: The tube should be made of tetrafluoroethylene resin. A conduit made of special vinyl chloride resin may also be used.

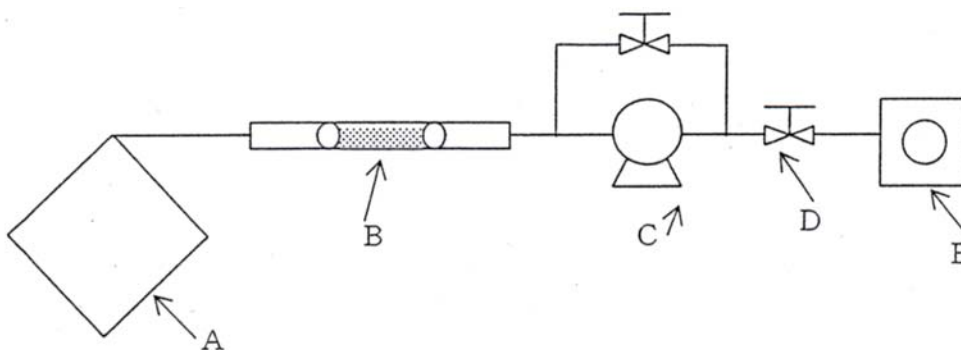
(2) Mercury-catching tube: Fig. 8 shows an example of a mercury-catching tube. As shown in Fig. 8, an indented quartz glass tube is packed in order with quartz glass wool, 80~200mg of mercury-catching agent, and quartz glass wool. The mercury-catching tube is heated at 600~800 C for 5 minutes through which carrier gas is caused to flow at a rate of 0.2~0.5l/min, and then placed in a glass test tube, which is closed hermetically with a stopper made of butyl rubber and stored. The storage limit is 6 months from preparation.

(3) Integrating gas meter: Meter made of anticorrosive material with a minimum scale of at least 0.01L (approximately 1l per rotation).



**Fig. 8 Mercury-catching tube (example)**

Gas in the fluoride resin bag is collected in the mercury-catching tube by the following apparatus:



**Fig. 9 Construction of sampling device for sample (example)**

A: Fluoride resin bag      B: Mercury-catching tube      C: Suction pump  
D: Flow adjustment pump      E: Integrating gas meter

### 7.1.3 Sampling

Using the sampling device, the sample gas is passed through the mercury-catching tube at a rate of 0.5~1.0l/min for a specified duration,<sup>20</sup> and the sample collected. After sampling, the catching tube is swiftly put into a glass test tube, which is closed hermetically with a stopper made of butyl rubber. To measure the value for the blank test, a mercury-catching tube through which no sample gas has passed is prepared.

Note 20 The passing rate of the sample gas is adjusted to result in 1 $\mu$ g or less as mercury.

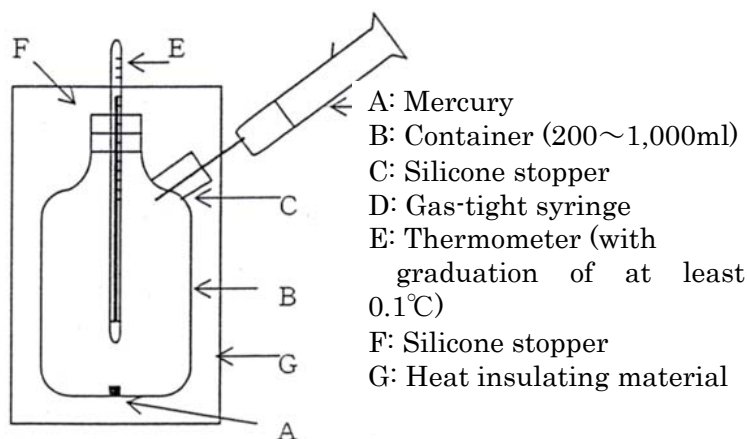
## 7.2 Determination method

### 7.2.1 Reagents and apparatus

- (1) Buffer solution: Neutral phosphate pH reference solution is used as specified in JIS Z 8802.
- (2) Carrier gas: Air and argon gas (purity more than 99.99%) from which mercury has been removed.
- (3) Mercury reference gas: Fig. 10 shows an example of the apparatus used to prepare the mercury reference gas.

A small amount of mercury is placed in a container and allowed to stand in a room at constant temperature for at least 1 hour. The temperature in the container is read with a thermometer, and the concentration of mercury in the container found using Table 1.

An aliquot of the gas is taken using a gas-tight syringe for use as the mercury reference gas.



**Fig. 10 Apparatus for preparation of mercury reference gas (example)**

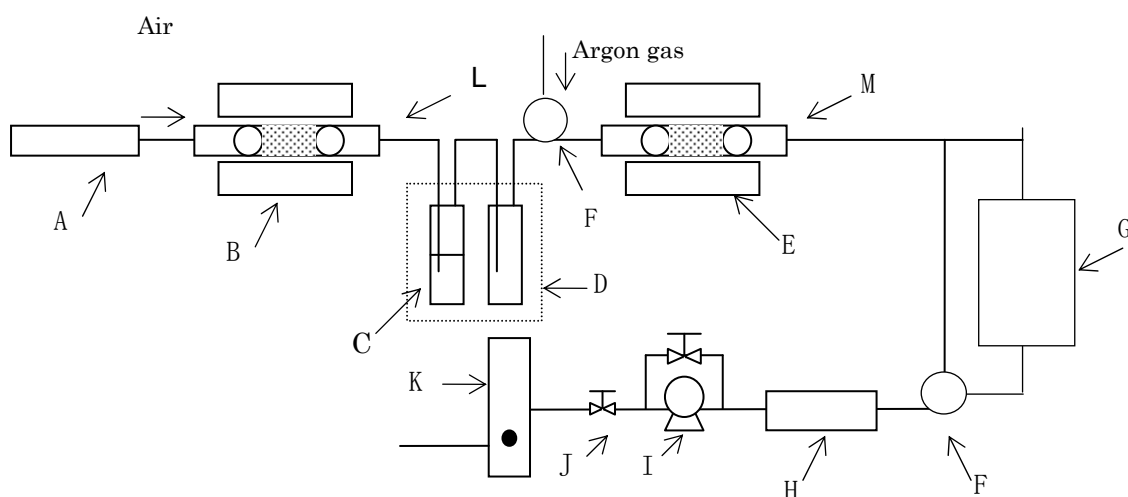
### 7.2.2 Apparatus<sup>21</sup>

The apparatus consists of equipment including a mercury-removal filter, vaporization heating furnace, dehumidifying bottle, gas washing bottle, mercury re-catching furnace, selector cock, absorption cell, suction pump, flowmeter, mercury removal device, and mercury re-catching tube.

The construction of the apparatus is exemplified in Fig. 11.

Note 21 An automated version of this apparatus capable of continuous measurement may be used.

- (1) Mercury-removal filter: Mercury-catching tube or quartz glass tube packed with approximately  $840\mu\text{m}$  of active carbon.
- (2) Vaporization heating furnace: A tubular furnace to heat the mercury catching tube. It should be capable of raising the temperature to  $600^{\circ}\text{C}$  within 3 minutes, and be cooled by air fan after heating has finished.
- (3) Gas washing bottle: Gas washing bottle containing 20~100ml of buffer solution.
- (4) Dehumidifying bottle: Empty gas washing bottle. Cooled together with (3).
- (5) Mercury-removal device: As specified in 5.2.(2)(h).
- (6) Mercury re-catching furnace: Electric tubular furnace capable of heating mercury-catching tube from  $150^{\circ}\text{C}$  to  $500\sim 800^{\circ}\text{C}$  in 1 minute.
- (7) Mercury re-catching tube: As specified in 6.1.2(2).
- (8) Fluorescent cell: Made from stray light blocked quartz, glass or plastic (that does not absorb mercury). Quartz glass window is attached light source side and photometry side that is right-angled to light source.



**Fig. 7 Construction of vaporization heating apparatus (example)**

- |                             |                                 |                          |
|-----------------------------|---------------------------------|--------------------------|
| A: Mercury-removal filter   | B: Vaporization heating furnace | C: Gas-washing bottle    |
| D: Dehumidifying bottle     | E: Mercury re-catching furnace  | F: Selector cock         |
| G: Fluorescent cell         | H: Mercury-removal device       | I: Suction pump          |
| J: Flow adjustment cock     | K: Flowmeter                    | L: Mercury-catching tube |
| M: Mercury re-catching tube |                                 |                          |

7.2.3 Procedure: The procedure used is as follows:

- (1) The mercury-catching tube<sup>22</sup> is placed inside the vaporization heating furnace, heated at  $600\sim 800^{\circ}\text{C}$  for about 3 minutes while carrier gas is flowing at a flow rate of  $0.2\sim 0.5\text{L}/\text{min}$ , and the mercury generated conducted into the re-catching tube.<sup>23</sup>

Note 22 The speed with which mercury is desorbed differs depending on the packing method, and so a re-catching tube with constant performance should be used for catching.

23 Some organic compounds indicate absorption at the wavelength of mercury measurement. To eliminate these organic compounds, the re-catching tube should be heated at about 150 °C.

- (2) The selector cock is switched to introduce carrier gas (argon gas) into the absorption cell.
- (3) The re-catching tube is heated at a constant temperature of 500~800 °C, and the mercury generated introduced by argon gas into the fluorescent cell.
- (4) The peak height or peak area given by absorption at a wavelength of 253.7nm is measured.
- (5) Using the mercury-catching tube for blank-test value measurement, steps (1) to (4) are performed to obtain the value for the blank test.
- (6) The mass of mercury is found on the working curve, and the concentration of mercury in the sample gas calculated according to the following formula.

$$C = (A - A_0) \times \frac{1}{V_s}$$

where

C: concentration of mercury ( $\mu\text{g}/\text{m}^3$ )

A: mass of mercury obtained on working curve (ng)

A<sub>0</sub>: value of blank test (ng)

V<sub>s</sub>: volume of sample gas taken (l)

$$V_s = V \times \frac{273.15}{273.15 + t} \times \frac{P_a + P_m - P_v}{101.32}$$

where

V: volume of gas measured by multiplication gas meter (L)

T: temperature in multiplication gas meter (degrees Celcius)

P<sub>a</sub>: atmospheric pressure (kPa)

P<sub>m</sub><sup>11</sup>: gauge pressure in multiplication gas meter (kPa)

P<sub>v</sub>: saturated steam pressure at t degrees Celcius (kPa)<sup>25</sup>

Note 24 Negligible in most cases.

Note 25 Refer to the attached table 1.1 Maximum vapour pressure of water, as per JIS Z 8806 (2001).

- (7) The working curve is prepared as follows:
  - (a) The mercury-removal filter is detached, and 0.1~10ml of mercury reference gas taken stepwise using a gas-tight syringe and introduced into mercury-catching tubes while a suction pump is operated.
  - (b) Steps (1) to (5) are performed
  - (c) After introducing the reference gas, the mercury-removal filter is attached
  - (d) The relation curve between the mass of mercury and measured value is plotted to give the working curve. The working curve is prepared when the sample is determined.

**Table 1 Concentration of saturated mercury vapor**

Temperature °C	Mercury concentration Ng/ml	Temperature °C	Mercury concentration ng/ml	Temperature °C	Mercury concentration ng/ml	Temperature °C	Mercury concentration ng/ml	Temperature °C	Mercury concentration ng/ml
0.0	2.179	0.2	2.225	0.4	2.271	0.6	2.319	0.8	2.368
1.0	2.417	1.2	2.465	1.4	2.514	1.6	2.564	1.8	2.614
2.0	2.666	2.2	2.716	2.4	2.766	2.6	2.818	2.8	2.871
3.0	2.924	3.2	2.978	3.4	3.033	3.6	3.089	3.8	3.146
4.0	3.204	4.2	3.264	4.4	3.325	4.6	3.388	4.8	3.451
5.0	3.516	5.2	3.583	5.4	3.650	5.6	3.719	5.8	3.789
6.0	3.861	6.2	3.933	6.4	4.007	6.6	4.083	6.8	4.159
7.0	4.237	7.2	4.316	7.4	4.396	7.6	4.478	7.8	4.561
8.0	4.645	8.2	4.731	8.4	4.817	8.6	4.905	8.8	8.994
9.0	5.085	9.2	5.178	9.4	5.273	9.6	5.369	9.8	5.467
10.0	5.567	10.2	5.666	10.4	5.767	10.6	5.870	10.8	5.974
11.0	6.079	11.2	6.187	11.4	6.296	11.6	6.407	11.8	6.519
12.0	6.633	12.2	6.751	12.4	6.870	12.6	6.992	12.8	7.115
13.0	7.240	13.2	7.369	13.4	7.501	13.6	7.635	13.8	7.771
14.0	7.909	14.2	8.049	14.4	8.191	14.6	8.339	14.8	8.481
15.0	8.630	15.2	8.781	15.4	8.935	15.6	9.092	15.8	9.251
16.0	9.412	16.2	9.575	16.4	9.742	16.6	9.910	16.8	10.081
17.0	10.255	17.2	10.429	17.4	10.604	17.6	10.783	17.8	10.964
18.0	11.148	18.2	11.337	18.4	11.529	18.6	11.724	18.8	11.922
19.0	12.123	19.2	12.328	19.4	12.536	19.6	12.747	19.8	12.961
20.0	13.179	20.2	13.400	20.4	13.623	20.6	13.851	20.8	14.081
21.0	14.315	21.2	14.553	21.4	14.795	21.6	15.040	21.8	15.289
22.0	15.542	22.2	15.800	22.4	16.061	22.6	16.326	22.8	16.569
23.0	16.869	23.2	17.148	23.4	17.431	23.6	17.718	23.8	18.010
24.0	18.306	24.2	18.606	24.4	18.911	24.6	19.220	24.8	19.534
25.0	19.852	25.2	20.174	25.4	20.500	25.6	20.830	25.8	21.166
26.0	21.506	26.2	21.853	26.4	22.204	26.6	22.560	26.8	22.922
27.0	23.289	27.2	23.660	27.4	24.036	27.6	24.418	27.8	24.805
28.0	25.198	28.2	25.598	28.4	26.003	28.6	26.415	28.8	26.832
29.0	27.255	29.2	27.685	29.4	28.121	29.6	28.564	29.8	29.012
30.0	29.467	30.2	29.928	30.4	30.395	30.6	30.868	30.8	31.348
31.0	31.835	31.2	32.329	31.4	32.830	31.6	33.339	31.8	33.854
32.0	34.376	32.2	34.908	32.4	35.448	32.6	35.995	32.8	36.549
33.0	37.111	33.2	37.681	33.4	38.258	33.6	38.843	33.8	39.437
34.0	40.038	34.2	40.647	34.4	41.264	34.6	31.889	34.8	42.523
35.0	43.165	35.2	43.819	35.4	44.481	35.6	45.152	35.8	45.832