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Aluminium sulfate for water works

ICS 71.060.50

Descriptors : phosphoric acid, inorganic acids, water treatment, water supply,
aluminium inorganic compounds, sulphates, water softening

Reference number : JIS K 1450 : 1996 (E)

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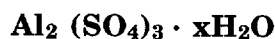
In the event of any doubts arising as to the contents,
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Aluminium sulfate for water works



1 Scope This Japanese Industrial Standard specifies aluminium sulfate for water works.

Remarks: The standards cited in this Standard are listed in Attached Table 1.

2 Classification Classification shall be as follows:

- (1) Solid
- (2) Liquid

3 Quality The quality, when being tested according to 5, shall conform to the requirements shown in Table 1.

Table 1 Quality

Item	Classification	
	Solid	Liquid
Appearance	—	Colorless to yellowish light brown transparent liquid
Aluminium oxide (Al_2O_3) wt%	15.0 min.	8.0 to 8.2
pH value	3.0 min.	3.0 min.
Insoluble matter wt%	0.1 max.	—
Ammoniacal nitrogen (N) wt ppm	300 max.	100 max.
Arsenic (As) wt ppm	4 max.	2.0 max.
Iron (Fe) wt ppm	600 max.	200 max.
Manganese (Mn) wt ppm	25 max.	15 max.
Cadmium (Cd) wt ppm	2.0 max.	1.0 max.
Lead (Pb) wt ppm	10 max.	5 max.
Mercury (Hg) wt ppm	0.2 max.	0.1 max.
Chromium (Cr) wt ppm	10 max.	5 max.

4 Sampling method Sampling shall be carried out so as to make the sample represent the whole, according to rational method based on the agreement between the parties concerned with delivery.

5 Test method

5.1 General matters General matters applicable commonly to tests shall follow **JIS K 0050**, those to absorptiometry follow **JIS K 0115**, those to atomic emission spectrometry follow **JIS K 0116**, and those to atomic absorption spectrometry follow **JIS K 0121**.

The water for tests shall be A2 specified in 3 of **JIS K 0557** or at least equivalent one, except when the test specifies the water to be used.

5.2 Aluminium oxide The determination of aluminium oxide shall follow titrimetry.

(1) **Summary** Add dihydrogen disodium ethylenediaminetetraacetate into sample liquid more excessively than the amount of aluminium in the sample, complete the formation of chelate, add xylenol orange indicator, carry out back titration with zinc solution, and obtain the content of aluminium oxide.

(2) **Reagents** Reagents shall be as follows:

- (a) **0.05 mol/l EDTA solution** Weigh 18.61 g of dihydrogen disodium ethylenediaminetetraacetate dihydrate specified in **JIS K 8107**, add 1000 ml of water to dissolve it, and store it in an air-tight polyethylene bottle.
- (b) **Sodium acetate buffer solution** Dissolve 272 g of sodium acetate trihydrate specified in **JIS K 8371** in water to make 1000 ml.
- (c) **Xylenol orange solution (1 g/l)** Dissolve 0.1 g of xylenol orange specified in **JIS K 9563** in water to make 100 ml.
- (d) **Aluminium standard solution (1 mgAl/ml)** Weigh 1.000 g of aluminium (purity: 99.99 % or more) specified in **JIS K 8069**, put it in a 100 ml beaker, cover its upper with a watch glass, add carefully a small quantity of nitric acid (1+1), and dissolve it by heating. After allowing it to cool, transfer whole liquid into a 1000 ml measuring flask, and add nitric acid (1+30) up to the marked line.
- (e) **0.02 mol/l zinc solution** Weigh 1.308 g of zinc specified in **JIS K 8005**, put it in a 100 ml beaker, add 6 ml to 7 ml of hydrochloric acid and small quantity of water, and dissolve it by heating. Evaporate water on a water bath nearly to dry up, add water to dissolve it, transfer it into a 1000 ml measuring flask, and add water up to the marked line.

Take 20 ml of 0.05 mol/l EDTA solution into a 200 ml beaker, add 2 ml of nitric acid (1+12), carry out the procedures shown in (c) and (d) in below (3), and the used quantity (ml) of 0.02 mol/l zinc solution shall be made a_1 . Take 20 ml of aluminium standard solution and 20 ml of 0.05 mol/l EDTA solution into another 200 ml beaker, add 2 ml of nitric acid (1+12), cover it with a watch glass, boil it for 1 min, allow it to cool, carry out the procedures shown in (c) and (d) in below (3), and the used quantity (ml) of 0.02 mol/l zinc solution shall be made a_2 .

Calculate amount of aluminium, equivalent to 1 ml of 0.02 mol/l zinc solution, according to the following formula:

$$f = \frac{0.001 \times 20}{a_1 - a_2}$$

where, f : amount of aluminium equivalent to 1 ml of 0.02 mol/l zinc solution (g)

(3) **Operations** Operations shall be carried out as follows:

- (a) Weigh sample by about 5 g in case of solid or about 10 g in case of liquid to the nearest 1 mg, put it in a 200 ml beaker, and add about 100 ml of water to dissolve it. Filtrate it if necessary, transfer it into a 500 ml measuring flask, and add water up to the marked line.
- (b) Take 20 ml of this solution into a 200 ml Erlenmeyer flask, add 20 ml of 0.05 mol/l EDTA solution, and after boiling for 1 min allow it to cool.
- (c) Add about 10 ml of sodium acetate buffer solution and 2 drops to 5 drops of xylenol orange solution (1 g/l).
- (d) Titrate it with 0.02 mol/l zinc solution, and make it an end point when color of the solution turns faint red.
- (e) Take 20 ml of 0.05 mol/l EDTA solution, transfer it into another 200 ml Erlenmeyer flask, add about 20 ml of water and 2 ml of nitric acid (1+12), and thereafter carry out procedures as shown in (c) and (d).

(4) **Calculation** Calculate the concentration (wt%) of aluminium oxide in the sample according to the following formula:

$$A = \frac{1.8895 \times (b' - b) \times f}{S \times \frac{20}{500}} \times 100 - (c \times 0.9129 \times 10^{-4})$$

where, A : concentration of aluminium oxide in sample (wt%)

1.8895 : conversion factor from 1 g of aluminium to aluminium oxide

f : amount of aluminium equivalent to 1 ml of 0.02 mol/l zinc solution (g)

b : volume of 0.02 mol/l zinc solution consumed for sample solution (ml)

b' : volume of 0.02 mol/l zinc solution consumed at (3) (e) (ml)

c : concentration of iron obtained at 5.7 (wt ppm)

0.9129 : conversion factor from iron to aluminium

S : mass of sample (g)

5.3 pH value

(1) **Summary** Measure pH value of sample solution by glass-electrode method.

(2) **Apparatus**

Glass-electrode pH meter Type II specified in JIS Z 8802.

(3) **Operations** Operations shall be carried out as follows:

- (a) Weigh the sample by 1.0 g in case of solid or 2.0 g in case of liquid, put it into a beaker, add about 50 ml of water to dissolve it, transfer it into a 100 ml measuring flask, and add water up to the marked line.
- (b) While keeping it at about 20 °C temperature, measure its pH value according to 7 of JIS Z 8802.

5.4 Insoluble matter

(1) **Summary** Dissolve sample in water, filtrate it through a glass filter, dry the residue, weigh its mass, and calculate insoluble matter.

(2) **Implements** Implements shall be as follows:

- (a) **Filter** Crucible-type glass filter 1G4.
- (b) **Thermostatic dryer**

(3) **Operations** Operations shall be carried out as follows:

- (a) Weigh 10 g of sample to the nearest 1 mg, put it into a 200 ml beaker, and add about 100 ml of water to dissolve it.
- (b) Filtrate whole this solution through a filter whose mass has been known, and wash the residue with water until the washing shows no reaction by sulfate ion.
- (c) Dry the filter in a thermostatic dryer kept at 105 °C to 110 °C for 1 h, allow it to cool in a desiccator, and measure its mass to the nearest 0.1 mg.

(4) **Calculation** Find the difference (g) between the mass of the filter before operation at (3) (b) and the mass at (3) (c), and calculate the insoluble matter (wt%) according to the following formula:

$$B = \frac{e}{S} \times 100$$

where, B : insoluble matter in sample (wt%)

e : difference between masses (g)

S : sample (g)

5.5 Ammoniacal nitrogen

(1) **Summary** Add sodium carbonate into sample to make precipitate of aluminium as hydroxide, add sodium hypochlorite and then 1-naphthol into its supernatant, measure the absorbance of generated indophenol-type coloring stuff (bluish green) with wavelength of 720 nm vicinity, and obtain the content of ammoniacal nitrogen.

(2) **Reagents** Reagents shall be as follows:

- (a) **Sodium carbonate solution (30 g/l)** Dissolve 3 g of sodium carbonate (anhydride) specified in JIS K 8625 in water to make 100 ml solution.
- (b) **Sodium hydroxide solution (10 g/l)** Dissolve 1 g of sodium hydroxide specified in JIS K 8576 in water to make 100 ml solution.

- (c) **Sodium hypochlorite solution (available chlorine 1 g/l)** Dissolve 100/C ml (C: concentration of available chlorine) of sodium hypochlorite (on the market) and 15 g of sodium hydroxide in water to make 1000 ml solution.
 - (d) **EDTA·sodium hydroxide mixed solution** Dissolve 0.93 g of disodium ethylenediaminetetraacetate dihydrate specified in **JIS K 8107** in sodium hydroxide solution (40 g/l) to make 250 ml solution.
 - (e) **1-naphthol solution** Dissolve 1.6 g of 1-naphthol specified in **JIS K 8698** in acetone-ethyl alcohol solution (15 : 85) to make 100 ml solution.
 - (f) **Ammoniacal-nitrogen standard stock solution (0.1 mgN/ml)** Allow ammonium chloride specified in **JIS K 8116** in a desiccator [keeping magnesium perchlorate specified in **JIS K 8228**] to stand for at least 16 h, weigh its 0.382 g, put it in a 100 ml beaker, dissolve it in a small quantity of water, transfer it into a 1000 ml measuring flask, and add water up to the marked line.
 - (g) **Ammoniacal-nitrogen standard solution (5 µgN/ml)** Take 50 ml of ammoniacal-nitrogen standard stock solution (0.1 mgN/ml), transfer it into a 1000 ml measuring flask, and add water up to the marked line. Prepare this solution when it is used.
- (3) **Apparatus Photoelectric spectrophotometer**
- (4) **Operations** Operations shall be carried out as follows:
- (a) Weigh the sample by about 5 g in case of solid or about 10 g in case of liquid to the nearest 1 mg, put it into a 200 ml beaker, add about 100 ml of water to dissolve it, transfer it into a 500 ml measuring flask, and add water up to the marked line.
 - (b) Take 25 ml of this solution, transfer it into a 100 ml measuring cylinder with a stopper, add 5 ml of sodium carbonate solution (30 g/l), then add water up to the marked line, and shake it gently.
 - (c) Allow it to stand quietly to make hydroxide precipitate, take 50 ml of its supernatant, and transfer it into a 100 ml color comparison tube with a ground stopper.
 - (d) Add sodium hydroxide solution (10 g/l) to control its pH, and make its pH about 11⁽¹⁾.
 - (e) Add 1 ml of sodium hypochlorite solution (available chlorine: 1 g/l), mix them, 2 min later and within 5 min add 1 ml of EDTA·odium hydroxide mixed solution, and mix them again. Then 1 min later and within 5 min⁽²⁾ add 5 ml of 1-naphthol solution, mix them, add water up to the marked line, and allow it to stand at 25 °C to 30 °C for 15 min.
 - (f) Take its portion into a 10 mm absorption cell, and measure its absorbance at wavelength of 720 nm vicinity.
 - (g) **Working curve** Take stepwise, 0, 1, up to 7 ml, of ammoniacal nitrogen standard solution (5 µgN/ml), transfer them into as many 100 ml measuring flasks, and add water to make them about 50 ml. Thereafter, carry out the procedures as in (e) and (f), and plot the working curve making use of the relation between the quantity of ammoniacal nitrogen and the absorbance.

Notes (1) The blank for working curve and standard solution have a low buffer action, so pH adjustment is not needed.

(2) The time control needed when reagents are added shall be carried out accurately, including working curve preparation.

- (5) **Calculation** On the working curve prepared at (4) (g), find the amount (μg) of ammoniacal nitrogen which is equivalent to absorbance measured at (4) (f), and calculate the concentration (wt ppm) of ammoniacal nitrogen in the sample according to the following formula:

$$C = \frac{g}{S \times \frac{25}{500} \times \frac{50}{100}}$$

where, C : concentration of ammoniacal nitrogen in sample (wt ppm)

g : amount of ammoniacal nitrogen (μg)

S : mass of sample (g)

5.6 Arsenic The determination of arsenic shall follow either atomic absorption spectrometry or absorptiometry.

5.6.1 Atomic absorption spectrometry

- (1) **Summary** Add reducing agent into sample, react arsenic and nascent hydrogen to generate gaseous arsenic hydride, introduce the gas into a heated absorption cell to atomize the arsenic, measure the absorbance at wavelength of 193.7 nm, and obtain the content of the arsenic.
- (2) **Reagents** Reagents shall be as follows:
- (a) **Hydrochloric acid (1+1)** The one prepared using hydrochloric acid, for arsenic analysis, specified in **JIS K 8180**.
 - (b) **Potassium iodide solution (200 g/l)** Dissolve 20 g of potassium iodide specified in **JIS K 8913** in water to make 100 ml solution. Prepare this solution when it is used.
 - (c) **Sodium boron hydride solution (10 g/l)** Dissolve 1 g of sodium hydroxide specified in **JIS K 8576** and 10 g of sodium boron hydride in water to make 1000 ml solution. Prepare this solution when it is used.
 - (d) **Arsenic standard stock solution (0.1 mgAs/ml)** Heat diarsenic trioxide specified in **JIS K 8005** at 105 °C for about 2 h, allow it to cool in a desiccator, weigh its 0.132 g, put it in a 100 ml beaker, cover its upper with a watch glass, add 250 ml of sodium hydroxide solution (4 g/l), and heat it to dissolve the diarsenic trioxide. After making it cool, transfer the solution into a 1000 ml measuring flask. Wash the beaker with hydrochloric acid (1+50) several times, transfer also the washings into the measuring flask, and add hydrochloric acid (1+50) up to the marked line. Alternatively, employ arsenic standard solution, As 100, specified in **JIS K 0026**.

- (e) **Arsenic standard solution (0.05 µgAs/ml)** Take 10 ml of arsenic standard stock solution (0.1 mgAs/ml), transfer it into a 100 ml measuring flask, and add water up to the marked line. Then, take 5 ml of this solution, transfer it into a 1000 ml measuring flask, and add water up to the marked line.
- (3) **Apparatus** Apparatus shall be as follows:
- (a) **Continuous-type arsenic hydride generating apparatus**
 - (b) **Atomic absorption spectrometer**
 - (c) **Hollow cathode lamp for arsenic**
 - (d) **Carrier gas** Either argon class 2, specified in **JIS K 1105**, or nitrogen gas (99.99 vol% or more).
- (4) **Operations** Operations shall be carried out as follows:
- (a) Weigh the sample by about 5 g in case of solid or about 10 g in case of liquid to the nearest 1 mg, put it in a 100 ml beaker, and add about 30 ml of water to dissolve it. Transfer it into a 100 ml measuring flask, and add water up to the marked line.
 - (b) Take 5 ml of this solution into a 100 ml beaker, add water to make total about 50 ml, add 10 ml of hydrochloric acid (1+1) and 5 ml of potassium iodide solution (200 g/l), and heat this solution to make concentration for about 30 min moderately enough not to boil. After making it cool, transfer it into a 50 ml measuring flask, and add water up to the marked line.
 - (c) Make react the sample treated as in (b) together with hydrochloric acid (1+1) and sodium boron hydride solution (10 g/l) while they are let flow into the hydride generating apparatus, introduce continuously the generated arsenic hydride, with carrier gas, into a heated absorption cell, and measure the absorbance at wavelength of 193.7 nm.
 - (d) **Working curve** Take stepwise, 0, 1, 2, up to 10 ml, of arsenic standard solution (0.05 µgAs/ml) into as many 100 ml beakers, and add water to make them about 50 ml. Thereafter, carry out the procedures as in (b) and (c), and plot the working curve making use of the relation between the amount (µg) of arsenic and the absorbance.
- (5) **Calculation** On the working curve prepared at (4) (d), find the amount (µg) of arsenic which is equivalent to the absorbance measured at (4) (c), and calculate the concentration (wt ppm) of arsenic in the sample according to the following formula:

$$D = \frac{h}{S \times \frac{5}{100}}$$

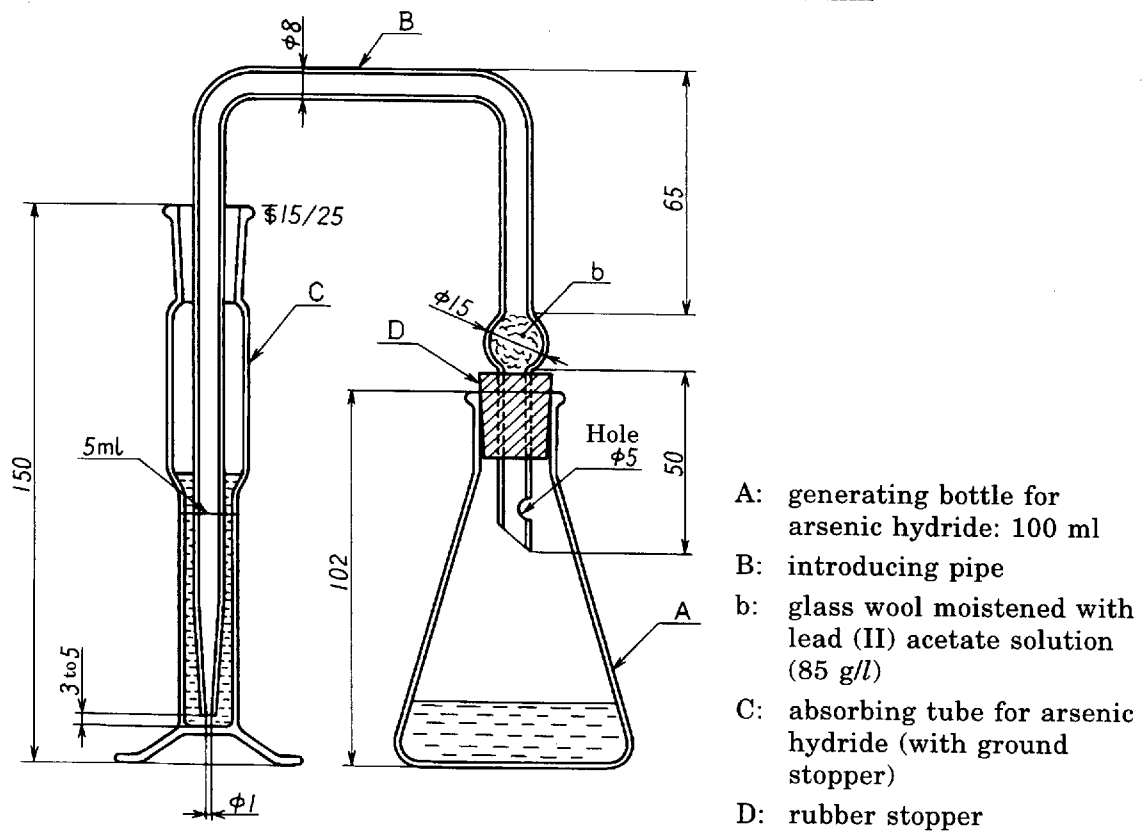
where, D : concentration of arsenic in sample (wt ppm)
 h : amount of arsenic (µg)
 S : mass of sample (g)

Remarks: There is another arsenic hydride generating apparatus which is noncontinuous type using zinc powder, and this method is also available.

5.6.2 Absorptiometry

- (1) **Summary** Add reducing agent into sample, react arsenic and nascent hydrogen to generate gaseous arsenic hydride, introduce the gas into silver diethyldithiocarbamate in pyridine solution to make absorbing and coloring, measure the absorbance at wavelength of 530 nm vicinity, and obtain the content of the arsenic.
- (2) **Reagents** Reagents shall be as follows:
 - (a) **Hydrochloric acid** For arsenic analysis, specified in **JIS K 8180**.
 - (b) **Potassium iodide solution (150 g/l)** Dissolve 15 g of potassium iodide specified in **JIS K 8913** in water to make 100 ml solution. Prepare this solution when it is used.
 - (c) **Tin (II) chloride solution** Dissolve 40 g of tin (II) chloride dihydrate specified in **JIS K 8136** in 100 ml of hydrochloric acid. Prepare this solution when it is used.
 - (d) **Lead acetate solution (85 g/l)** Add 1 drop of acetic acid (1+2) specified in **JIS K 8355** onto 10 g of lead (II) acetate trihydrate specified in **JIS K 8374**, and dissolve it in water to make 100 ml solution. Store it with close stoppering.
 - (e) **Granular zinc** Granular zinc (arsenic-free) which has passed through a standard sieve with 1410 μm mesh specified in **JIS Z 8801** and stayed on 1000 μm mesh.
 - (f) **DDTC silver pyridine solution (5 g/l)** Dissolve 1 g of silver N,N-diethyldithiocarbamate specified in **JIS K 9512** in 200 ml of pyridine. If stored in a brown bottle under cold and dark place, it can be kept stable for about 3 months.
 - (g) **Arsenic standard stock solution (0.1 mgAs/ml)** Follow 5.6.1 (2) (d).
 - (h) **Arsenic standard solution (1 $\mu\text{gAs/ml}$)** Take 10 ml of arsenic standard stock solution (0.1 mgAs/ml), transfer it into a 1000 ml measuring flask, and add water up to the marked line.
- (3) **Apparatus** Apparatus shall be as follows:
 - (a) **Photoelectric spectrophotometer**
 - (b) **Arsenic testing apparatus** Figs. 1 and 2 show its example.

Unit: mm

**Fig. 1** Example of arsenic testing apparatus

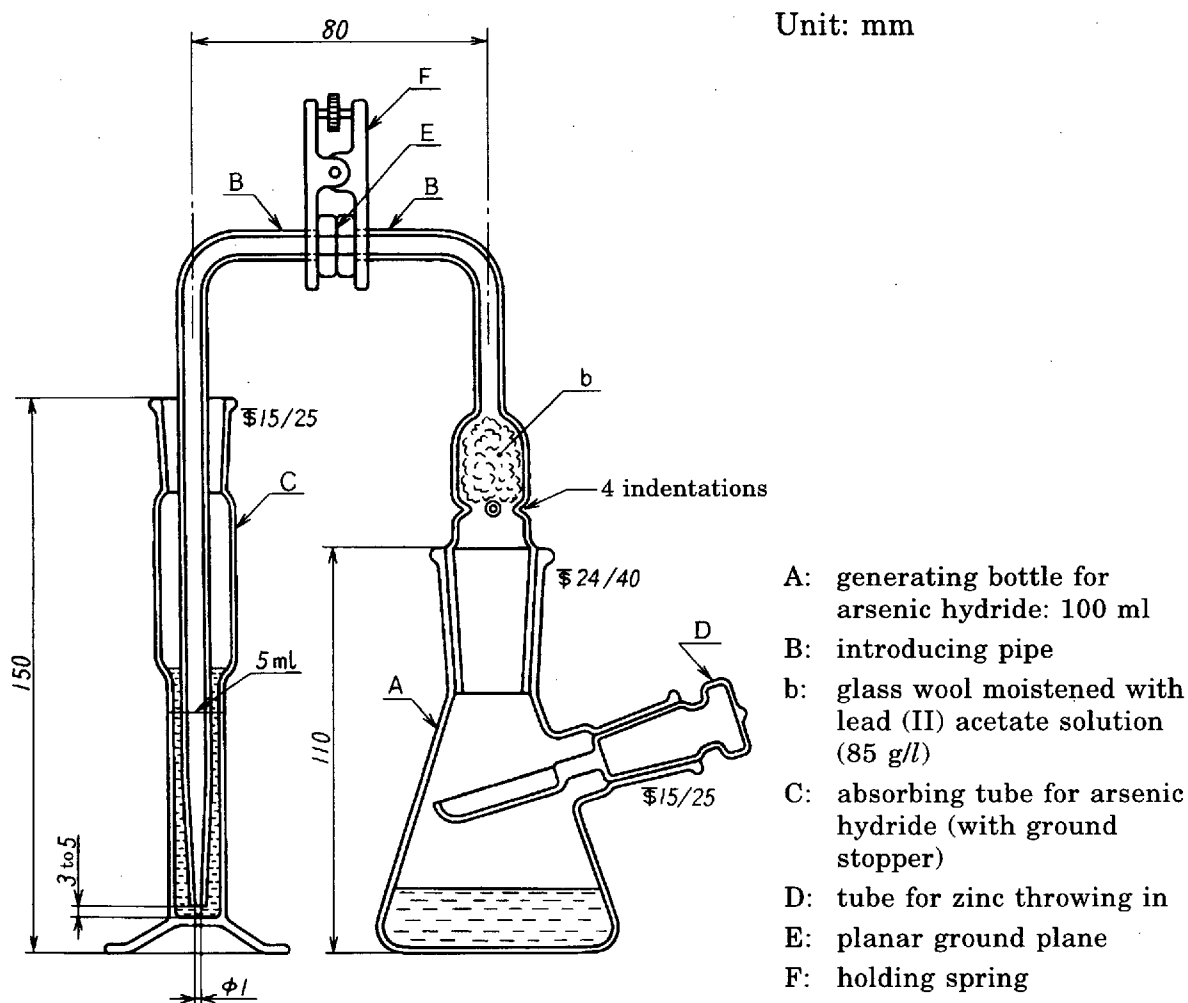


Fig. 2 Example of arsenic testing apparatus

(4) Operations Operations shall be carried out as follows:

- (a) Weigh the sample by about 1 g in case of solid or about 2 g in case of liquid to the nearest 1 mg, put it into a generating bottle of an arsenic testing apparatus, and add 5 ml of hydrochloric acid and water to make total about 40 ml.
- (b) Add 2 ml of potassium iodide solution (150 g/l), allow it to stand for 2 min to 3 min, add 1 ml of tin (II) chloride solution, mix them, and stand it quietly for about 15 min.
- (c) Place 5 ml of DDTC silver pyridine solution (5 g/l) in an absorbing tube, add 3 g of granular zinc in the generating bottle, then promptly connect the generating bottle, an introducing pipe, and the absorbing tube, and absorb generated arsenic hydride by standing it at ordinary temperature for about 1 h.
- (d) Put a portion of the solution kept in the absorbing tube into a 10 mm absorption cell, and measure its absorbance at wavelength of 530 nm vicinity with setting DDTC silver pyridine solution (5 g/l) as a contrast solution.