

JIS

JAPANESE INDUSTRIAL STANDARD

**Testing methods for
paint components**

JIS K 5407—1990

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In the event of any doubt arising,
the original Standard in Japanese is to be final authority.

Testing Methods for Paint Components

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JAPANESE INDUSTRIAL STANDARD
Testing Methods for Paint Components

J I S
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1. Scope

This Japanese Industrial Standard specifies the testing methods for paint components.

Remark: The units and numerical values given in { } in this Standard are based on the traditional units and are currently the criteria in force.

2. Common Items

The common items shall be as follows:

(1) General Items

(1.1) General Rules In the tests for paint components, unless otherwise specified, general items common to chemical analyses shall be in accordance with JIS K 0050, general items common to preparation of titrants, indicators, and buffer solutions, with JIS K 8001 and JIS K 8005, general items common to potentiometric titration methods, with JIS K 0113, general items common to gas chromatography, with JIS K 0114, general items common to absorptiometries, with JIS K 0115, and general items common to infrared spectroscopic methods, with JIS K 0117.

(1.2) Filter Paper The filter paper specified in JIS P 3801 shall be used.

(1.3) Apparatus For glass apparatus used for chemical analysis those specified in JIS R 3503, for volumetric glassware those specified in JIS R 3505, and for porcelain apparatus those specified in JIS R 1301 shall be used. However, when special apparatus are used, examples thereof are shown in a figure or described in each item.

Further, for glassware in the case of training heating operation, hard grade 1 specified in JIS R 3503 shall be used.

Applicable Standards and Corresponding International Standards: See pages 117 to 120.

(1.4) Reagents The reagents shall be as follows:

- (a) For reagents, those marked with JIS mark shall be used. For reagents not specified as allowable products marked with JIS mark, reagents certificated based on reagents marking certification system of the Ministry of International Trade and Industry shall be used.

Further, when there are neither reagents marked with JIS mark nor those certificated based on the reagents marking certification system, those without hindrance to a test shall be used.

For standardization of titrants, the reagents specified in JIS K 8005 shall be used.

- (b) The concentration indicated in () after the name of solution in preparation of reagents means approximate concentration.

For instance, potassium hydroxide - ethanol solution (0.5 mol/l) indicates potassium hydroxide ethanol solution of about 0.5 mol/l in concentration.

Further, the concentration given before the name of solution means accurate concentration. However, it is generally indicated by a numerical value having no fraction, and a factor has been independently previously obtained.

- (c) Though water used for preparation of reagents shall be the water of (1.5), in the case of chelatometric titration method, ion exchange water shall be used and when specified in each item, the operation shall follow the said specification.
- (d) When standard solution is prepared by diluting standard stock solution, 10 ml or over shall be taken with a transfer pipet.
- (1.5) Water The water shall be either distilled water or ion exchange water (water from which dissolved ion is removed by using ion exchange resin), and be preserved by using polyethylene, hard glass, and other material containers capable of being sealed according to the purpose of a test.
- (a) Distilled Water Distilled water of which electric conductivity is $2 \mu\text{S}/\text{cm}$ (25°C) or under and of which color retention time ⁽¹⁾ by potassium permanganate is 10 min or over.
- (b) Ion Exchange Water Ion exchange water which is filtered by a membrane filter of $0.45 \mu\text{m}$ ⁽²⁾ in pore size and of which electric conductivity is $1 \mu\text{S}/\text{cm}$ (25°C) or under and color retention time ⁽¹⁾ by potassium permanganate is 10 min or over.

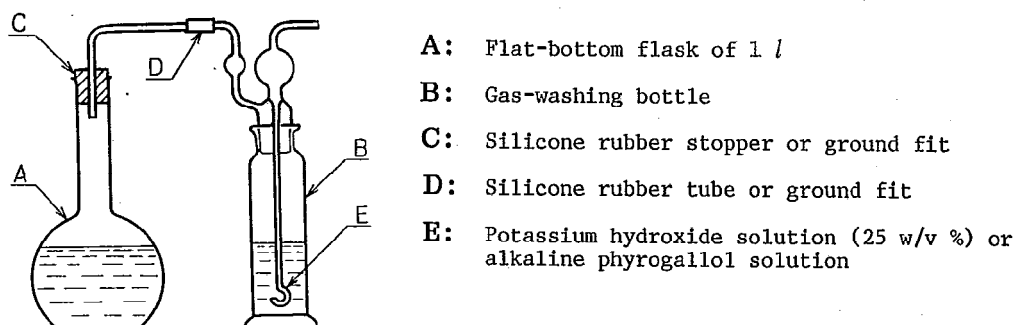
Notes ⁽¹⁾ The color retention time herein means a time until reddish color disappears when 0.2 ml of 0.002 mol/l (0.01 N) potassium permanganate solution and 2 ml of sulfuric acid (1 + 1) are added to 500 ml of water, are sufficiently mixed by shaking, and are allowed to stand still.

- (2) Since nonelectrolyte, suspended solid, etc. are hardly removed, the ion exchange water is filtered by using the membrane filter of 0.45 μm or under in pore size.

Remark: Distilled water is the water refined by using a distillation method as the final process for refining water. Further, ion exchange water is the water refined by using an ion exchange method as the final process for refining.

- (1.6) Water Not Containing Carbon Dioxide The water is prepared as follows: After boiling for approximately 5 min, attach the gas washing bottle containing potassium hydroxide solution (25 w/v %) ⁽³⁾ as shown in Fig. 1, intercept carbon dioxide in the air, and allow to stand to cool. This water is prepared when used.
- (1.7) Water Not Containing Dissolved Oxygen This water is prepared as follows: Put water in a flask. After boiling for about 5 min, attach the gas washing bottle containing alkaline pyrogallol solution ⁽⁴⁾ as shown in Fig. 1, intercept oxygen in the air, and allow to stand to cool. This water is prepared when used.

Fig. 1. Cooling and Preservation of Water Not Containing Carbon Dioxide or Dissolved Oxygen (An Example)



Notes ⁽³⁾ The potassium hydroxide solution (25 w/v %) is prepared by using the potassium hydroxide specified in JIS K 8574.

⁽⁴⁾ The alkaline pyrogallol solution is prepared by adding sodium hydroxide 30 w/v % solution prepared by using sodium hydroxide specified in JIS K 8576 to 10 g of pyrogallol specified in JIS K 8780 to make into 100 ml.

Further, this solution is prepared when used.

- (1.8) Dilution of Liquid Reagent When liquid reagent is diluted to be used, for instance, "hydrochloric acid (1 + 2)" is prepared by adding water at a rate of 2 ml of water to 1 ml of hydrochloric acid.

- (1.9) Content of Solute in Solution The symbol of w/v % is used to indicate the number of g of solute (converted to anhydride) in solution of 100 ml and further, the symbol of v/v % is used to indicate the number of ml.
- (1.10) Weighing out Accurately "Weighing out accurately" means generally to weigh out to the nearest 0.1 mg in the case of mass. However, by considering the number of figures to be indicated of an analytical value, it is weighed out to necessary figures.
- Further, in the case of volume, the volume is measured out by using a whole pipet, a measuring pipet or a buret.
- (1.11) Number of Drops When the amount of solution is indicated by the number of drops, about 1/20 ml is taken as one drop.
- (1.12) Acidity, Neutrality and Alkalinity of Solution The acidity, neutrality and alkalinity of solution, unless otherwise specified, are examined by using litmus test paper.
- (1.13) Regulation of pH Value of Solution The regulation of pH value of solution, unless otherwise specified, is performed by using pH test paper.
- (1.14) Constant Weight "Heating to constant weight" means a case wherein heating or ignition is performed successively further for 30 min, is left to cool in a desiccator and thereafter, a mass difference between the mass before the test and that after the test becomes 1.0 mg or under by using a chemical balance.
- (1.15) Drying For "drying", unless especially specified, drying is performed for a specific time with an electric constant temperature oven capable of keeping temperature within test temperature $\pm 2^{\circ}\text{C}$.
- (1.16) Desiccant in Desiccator Unless otherwise specified, silica gel is used.
- (1.17) Separation with a Centrifugal Separator Separation is performed under condition of 3000 to 15000 r.p.m. with the centrifugal separator having a glass or stainless steel precipitation tube, a round bottom, and a capacity of about 50 ml.
- (1.18) Grinding When a sample or a reagent is ground to be homogeneous, unless otherwise specified, grinding is performed by using an agate mortar.
- (1.19) Standing to Cool Unless otherwise specified, temperature is decreased to room temperature by allowing a sample to stand naturally.
- (1.20) Cooling Unless otherwise specified, temperature is decreased to room temperature by using cold water or the like.
- (1.21) Heating on a Water Bath Unless otherwise specified, heating is performed on a boiling water bath.

- (1.22) Heating in a Water Bath Unless otherwise specified, heating is performed in a boiling water bath.
- (1.23) Warm Water Warm water means water of 40 to 60°C in temperature.
- (1.24) Hot Water Hot water means water of 60°C or higher in temperature.
- (1.25) Room Temperature Ambient temperature in a laboratory, which is 5 to 35°C, is called room temperature.
- (1.26) To Remove Organic Solvent by Distillation A boiling stone is put into an Erlenmeyer flask and a letter shape tube and a Liebig condenser are connected thereto. The Erlenmeyer flask is placed on a water bath or a mantle heater which is regulated at a temperature 10 to 20°C higher than the boiling point of an organic solvent to be distilled, and the organic solvent is removed by distillation. When interchangeable ground joint glassware is used, the operation becomes easy.
- (1.27) Rounding Off of Numerical Values The numerical value of a measured result is obtained to the next place beyond the last right-hand place of significant figures, and is rounded off in accordance with JIS Z 8401.
- (1.28) Figure, Table, Note and Remark A Figure and a Table follow serial numbers through the whole. A Note and a Remark follow serial numbers in each item.
- (2) Cautions Related to Safety
- (a) When insanitary or irritant gas is generated, operation is performed at a place having an exhaustor or in a draft.
- (b) Wastes after being used for a test shall be treated sufficiently carefully in accordance with related ordinances and rules.
- (3) Preparation of Titrant, Indicator and Buffer Solution The titrant, indicator and buffer solution shall be prepared as follows:
- (3.1) Preparation of Titrant
- (3.1.1) 0.1 mol/l (0.1 N) Potassium Hydroxide Ethanol Solution (5.611 g KOH/l)
- (a) Preparation Weigh out 7 g of potassium hydroxide specified in JIS K 8574, dissolve in as little amount of water containing no carbon dioxide as possible, add ethanol (95) specified in JIS K 8102 to 1 l, and put in a polyethylene bottle. Intercept carbon dioxide and allow to stand as it is for several days. Thereafter, take the supernatant.
- (b) Standardization Weigh out accurately 25 ml of 0.05 mol/l sulfuric acid of 2. (3.1.15) into a conical beaker of 200 ml. Add 50 ml of water containing no carbon dioxide. Add two drops of phenolphthalein of 2. (3.2) (g) as an indicator, titrate with 0.1 mol/l potassium hydroxide ethanol solution, and take the point at which a light red remains before it disappears for 10 s as the end point. Standardize each service.

- (c) Calculation The factor shall be calculated from the following formula :

$$F_1 = \frac{25 \times F_2}{V}$$

where, F_1 : factor of 0.1 mol/l ethanol potassium hydroxide solution

25: amount of 0.05 mol/l sulfuric acid (ml)

F_2 : factor of 0.05 mol/l sulfuric acid

V : amount of 0.1 mol/l ethanol potassium hydroxide solution required for titration (ml).

(3.1.2) 0.05 mol/l Disodium Ethylenediaminetetraacetate Solution
(18.61 g $C_{10}H_{14}O_8N_2Na_2 \cdot 2H_2O/l$)

- (a) Preparation Weigh out 18.6 g of disodium ethylenediaminetetraacetate specified in JIS K 8107, dissolve in water to 1 l, and put in a polyethylene bottle to be preserved.
- (b) Standardization Weigh out accurately 25 ml of 0.05 mol/l zinc solution ⁽¹⁾ into a conical beaker of 200 ml, and add water to 100 ml. Regulate pH value at about 5.0 by adding hexamethylenetetramine specified in JIS K 8847. Add 0.3 ml of Xylenol Orange solution (0.1 w/v %) of 2. (3.2) (b) as an indicator, titrate with 0.05 mol/l disodium ethylenediaminetetraacetate solution, and take the point at which the color of solution turns from red purple through orange to yellow as an end point.

Note ⁽¹⁾ Wash zinc specified in JIS K 8005 successively with hydrochloric acid (1 + 3) prepared from hydrochloric acid specified in JIS K 8180, water, ethanol (99.5) specified in JIS K 8101, and diethyl ether specified in JIS K 8103. Immediately transfer to a desiccator containing magnesium perchlorate, and allow to stand for 12 h or over. Weigh out accurately about 1.7 g therefrom, and transfer to a conical beaker of 200 ml. Add 25 ml of water and 20 ml of nitric acid (1 + 1) prepared from nitric acid specified in JIS K 8541, cover with a watch glass, and dissolve by heating on a water bath. After removing nitrogen oxide by boiling, allow to stand to cool, transfer to a 500-ml one-mark volumetric flask, add water up to the mark, and prepare 0.05 mol/l zinc solution.

- (c) Calculation The factor shall be calculated from the following formula:

$$F = \frac{m \times \frac{25}{500}}{0.003270 \times V} \times \frac{A}{100}$$

where, F : factor of 0.05 mol/l disodium ethylenediaminetetraacetate solution

m : mass of zinc (g)

$\frac{25}{500}$: aliquot ratio of 0.05 mol/l zinc solution

A : content of zinc (%)

0.003270: mass of zinc equivalent to 1 ml of 0.05 mol/l disodium ethylenediaminetetraacetate solution (g)

V : amount of 0.05 mol/l disodium ethylenediaminetetraacetate solution required for titration (ml).

(3.1.3) 0.01 mol/l Disodium Ethylenediaminetetraacetate Solution
(3.722 g $C_{10}H_{14}O_8N_2Na_2 \cdot 2H_2O/l$)

- (a) Preparation Weigh out 3.8 g of disodium ethylenediaminetetraacetate specified in JIS K 8107, dissolve in water to 1 l, and put in a polyethylene bottle to be preserved.
- (b) Standardization Allow the standardization to be as described in 2. (3.1.2) (b). In that case, prepare 0.01 mol/l zinc solution by using about 0.33 g of zinc and 5 ml of nitric acid (1 + 1).
- (c) Calculation The factor shall be calculated from the following formula:

$$F = \frac{m \times \frac{25}{500}}{0.0006539 \times V} \times \frac{A}{100}$$

where, F : factor of 0.01 mol/l disodium ethylenediaminetetraacetate solution

m : mass of zinc (g)

$\frac{25}{500}$: aliquot ratio of 0.01 mol/l zinc solution

A : content of zinc (%)

0.0006539: mass of zinc equivalent to 1 ml of 0.01 mol/l disodium ethylenediaminetetraacetate solution (g)

V : amount of 0.01 mol/l disodium ethylenediaminetetraacetate solution required for titration (ml).

(3.1.4) 0.1 mol/l (0.2 N) Iodine Chloride-Acetic Acid Solution

Weigh out 8.7 g of iodine specified in JIS K 8920. Dissolve by adding iodine in 500 ml of acetic acid specified in JIS K 8355 heating on a water bath. Separately weigh out 7.9 g of iodine trichloride specified in JIS K 8403 to be dissolved in 400 ml of acetic acid. Mix both solutions, further add acetic acid to 1 l, and put into a brown bottle to be preserved at a dark place.

(3.1.5) 0.02 mol/l (0.1 N) Potassium Permanganate Solution (3.161 g KMnO_4/l)

- (a) Preparation Weigh out 3.2 g of potassium permanganate specified in JIS K 8247 to an Erlenmeyer flask of 2000 ml. Add 1050 ml of water. After quietly boiling for 1 to 2 h, allow to stand at a dark place for one night. After washing with water preliminarily boiled, filter the supernatant with a dry sintered-glass filter (17G4 or 25G4), and put into a brown bottle washed with steam for about 30 min to be preserved at a dark place.
- (b) Standardization Heat sodium oxalate specified in JIS K 8005 at a temperature of about 200°C for 1 h, and allow to stand to cool in a desiccator containing magnesium perchlorate. Weigh out accurately 0.20 to 0.24 g therefrom into a conical beaker of 500 ml, and dissolve in 200 ml of water. Add 20 ml of sulfuric acid (1 + 1) prepared by using sulfuric acid specified in JIS K 8951. Regulate the temperature of solution at 25 to 30°C, and titrate with 0.02 mol/l potassium permanganate solution stirring quietly. Add to this side about 2 ml of a required titre by opening fully the cock of a burette, and allow to stand until red of the solution disappears. Then, heat to 55 to 60°C in temperature, successively titrate, and take the point at which the light red is maintained for 30 s or over as the end point.

Perform a blank test keeping the solution to which 200 ml of water and 20 ml of sulfuric acid (1 + 1) are separately added, at 55 to 60°C in temperature.

- (c) Calculation The factor shall be calculated from the following formula:

$$F = \frac{m}{0.006700 \times (V_1 - V_2)} \times \frac{A}{100}$$

where, F : factor of 0.02 mol/l potassium permanganate solution

m : mass of sodium oxalate (g)

A : content of sodium oxalate (%)

0.006700: mass of sodium oxalate equivalent to 1 ml of 0.02 mol/l potassium permanganate solution

V_1 : amount of 0.02 mol/l potassium permanganate solution required for titration (ml)

V_2 : amount of 0.02 mol/l potassium permanganate solution required for blank test (ml).

(3.1.6) 0.1 mol/l (0.1 N) Silver (I) Nitrate Solution (16.99 g AgNO_3/l)

- (a) Preparation Weigh out 17 g of silver nitrate specified in JIS K 8550, dissolve in water to 1 l, and put in a brown bottle to be preserved.

- (b) Standardization Put sodium chloride specified in JIS K 8005 into a platinum crucible, heat at a temperature of about 600°C for 1 h, and allow to stand to cool in a desiccator containing magnesium perchlorate. Weigh out accurately 0.14 to 0.17 g therefrom into a conical beaker of 200 ml, and dissolve by adding 50 ml of water. Add 3 drops of uranine solution of 2. (3.2) (a) as an indicator, titrate with 0.1 mol/l silver (I) nitrate solution, and take a point at which the color of solution is tinted with reddish as the end point.

- (c) Calculation The factor shall be calculated from the following formula:

$$F = \frac{m}{0.005844 \times V} \times \frac{A}{100}$$

where, F : factor of 0.1 mol/l silver (I) nitrate solution

m : mass of sodium chloride (g)

A : content of sodium chloride (%)

0.005844: mass of sodium chloride equivalent to 1 ml of 0.1 mol/l silver (I) nitrate solution (g)

V : mass of 0.1 mol/l silver (I) nitrate solution required for titration (ml).

(3.1.7) 0.01 mol/l (0.01 N) Silver (I) Nitrate Solution (1.699 g AgNO₃/l)

- (a) Preparation Weigh out 1.7 g of silver nitrate specified in JIS K 8550, dissolve in water to 1 l, and put in a brown bottle to be preserved.

- (b) Standardization Put sodium chloride specified in JIS K 8005 into a platinum crucible, heat it at a temperature of about 600°C for 1 h, and allow to stand to cool in a desiccator containing magnesium perchlorate. Weigh out accurately 0.14 to 0.17 g therefrom, dissolve in water, transfer to a 250-ml one-mark volumetric flask, and add water up to the mark. Separately take accurately 25 ml from the solution into a conical beaker of 200 ml, and add 25 ml of water. Add 3 drops of uranine solution of 2. (3.2) (a) as an indicator, titrate with 0.01 mol/l silver (I) nitrate solution, and take a point at which the color of solution is tinted with red as the end point.

- (c) Calculation The factor shall be calculated from the following formula:

$$F = \frac{m \times \frac{25}{250}}{0.0005844 \times V} \times \frac{A}{100}$$

where, F : factor of 0.01 mol/l silver (I) nitrate solution

m : mass of sodium chloride (g)

$\frac{25}{250}$: aliquot ratio of sodium chloride solution

A : content of sodium chloride (%)

0.0005844: mass of sodium chloride equivalent to 1 ml of 0.01 mol/l silver (I) nitrate solution (g)

V : mass of 0.01 mol/l silver (I) nitrate solution required for titration (ml).

(3.1.8) 0.05 mol/l Lead (II) Nitrate Solution [16.56 g Pb (NO₃)₂/l]

- (a) Preparation Weigh out 16.6 g of lead nitrate specified in JIS K 8563, and dissolve in water to 1 l.
- (b) Standardization Weigh out accurately 30 ml of 0.05 mol/l disodium ethylenediaminetetraacetate solution of 2. (3.1.2) into a conical beaker of 200 ml, add 65 ml of water and 5 ml of acetic acid-sodium acetate buffer solution (pH value 5) of 2. (3.3), and add 0.4 ml of Xylenol Orange solution (0.1 w/v %) of 2. (3.2) (b) as and indicator. Titrate with 0.05 mol/l lead (II) nitrate solution, and take a point at which the yellow of the solution is tinted with slight red as the end point.
- (c) Calculation The factor shall be calculated from the following formula:

$$F_1 = \frac{F_2 \times 30}{V}$$

where, F_1 : factor of 0.05 mol/l lead (II) nitrate solution

F_2 : factor of 0.05 mol/l disodium ethylenediaminetetraacetate solution

30: amount of 0.05 mol/l disodium ethylenediaminetetraacetate solution (ml)

V : amount of 0.05 mol/l lead (II) nitrate solution required for titration (ml).

(3.1.9) 0.01 mol/l Lead (II) Nitrate Solution [3.312 g Pb (NO₃)₂/l]

- (a) Preparation Weigh out 3.4 g of lead nitrate specified in JIS K 8563, and dissolve in water to 1 l.
- (b) Standardization Weigh out accurately 30 ml of 0.01 mol/l disodium ethylenediaminetetraacetate solution of 2. (3.1.3) into a conical beaker of 200 ml, add 65 ml of water and 5 ml of acetic acid-sodium acetate buffer solution (pH value 5) of 2. (3.3), and add 0.4 ml of Xylenol Orange solution (0.1 w/v %) of 2. (3.2) (b) as an indicator. Titrate with 0.01 mol/l lead (II) nitrate solution, and take a point at which the yellow of the solution is tinted with slight red as the end point.
- (c) Calculation The factor shall be calculated from the following formula:

$$F_1 = \frac{F_2 \times 30}{V}$$

where, F_1 : factor of 0.01 mol/l lead (II) nitrate solution

F_2 : factor of 0.01 mol/l disodium ethylene-diaminetetraacetate solution

30 : amount of 0.01 mol/l disodium ethylene-diaminetetraacetate solution (ml)

V : amount of 0.01 mol/l lead (II) nitrate solution required for titration (ml).

(3.1.10) 0.1 mol/l (0.1 N) Sodium Hydroxide Solution (4.000 g Na OH/l)

(a) Preparation Weigh out 30 ml of water not containing carbon dioxide into a polyethylene bottle of 100 ml. Weigh out 35 g of sodium hydroxide specified in JIS K 8576, and dissolve by gradually adding it while cooling the polyethylene bottle. Thereafter, tightly stopper it, and allow to stand for 4 to 5 days. Weigh out 5 ml of this supernatant, add water not containing carbon dioxide to 1 l, and attach a soda lime tube thereto to be preserved.

(b) Standardization Dry amidosulfuric acid specified in JIS K 8005 in a desiccator containing magnesium perchlorate for 48 h. Weigh out accurately 0.24 to 0.29 g of amidosulfuric acid into a conical beaker of 200 ml, and dissolve by adding 25 ml of water. Add 2 drops of Bromothymol Blue solution (0.1 w/v %) of 2. (3.2) (h), titrate with 0.1 mol/l sodium hydroxide solution, and take a point at which the color of the solution turns to bluish green as the end point. Standardize at each service.

(c) Calculation Calculate the factor from the following formula:

$$F = \frac{m}{0.009710 \times V} \times \frac{A}{100}$$

where, F : factor of 0.1 mol/l sodium hydroxide solution

m : mass of amidosulfuric acid (g)

A : content of amidosulfuric acid (%)

0.009710 : mass of amidosulfuric acid equivalent to 1 ml of 0.1 mol/l sodium hydroxide solution (g)

V : mass of 0.1 mol/l sodium hydroxide solution required for titration (ml).

(3.1.11) 0.1 mol/l (0.1 N) Ammonium Thiocyanate Solution (7.612 g NH₄SCN/l)

- (a) Preparation Weigh out 8 g of ammonium thiocyanate specified in JIS K 9000, dissolve in 1 l of water, and put it into a brown bottle to be preserved.
- (b) Standardization Weigh out accurately 25 ml of 0.1 mol/l (0.1 N) silver (I) nitrate solution of 2. (3.1.6) into a conical beaker of 200 ml, and add 25 ml of water and 2 ml of nitric acid specified in JIS K 8541. Add 1 ml of ammonium iron (III) sulfate solution of 2. (3.2) (m), titrate with 0.1 mol/l ammonium thiocyanate solution, and take a point at which the color of the solution turns to light red brown as the end point.
- (c) Calculation The factor shall be calculated from the following formula:

$$F_1 = \frac{F_2 \times 25}{V}$$

where, F_1 : factor of 0.1 mol/l ammonium thiocyanate solution

F_2 : factor of 0.1 mol/l silver (I) nitrate solution

25: amount of 0.1 mol/l silver (I) nitrate solution (ml)

V : amount of 0.1 mol/l ammonium thiocyanate solution required for titration (ml).

(3.1.12) 0.01 mol/l (0.01 N) Ammonium Thiocyanate Solution (0.7612 g NH₄SCN/l)

- (a) Preparation Weigh out 0.8 g of ammonium thiocyanate specified in JIS K 9000, either dissolve in 1 l of water, or weigh out 100 ml of 0.1 mol/l (0.1 N) ammonium thiocyanate solution of 2. (3.1.11), add 900 ml of water, and put into a brown bottle to be preserved.
- (b) Standardization Weigh out accurately 25 ml of 0.01 mol/l (0.01 N) silver (I) nitrate solution of 2. (3.1.7) into a conical beaker of 200 ml, and add 25 ml of water and 2 ml of nitric acid specified in JIS K 8541. Add 1 ml of ammonium iron (III) sulfate solution of 2. (3.2) (m), titrate with 0.01 mol/l ammonium thiocyanate solution, and take a point at which the color of the solution turns to thin red brown as the end point.
- (c) Calculation The factor shall be calculated from the following formula:

$$F_1 = \frac{F_2 \times 25}{V}$$

where, F_1 : factor of 0.01 mol/l ammonium thiocyanate solution

F_2 : factor of 0.01 mol/l silver (I) nitrate solution

25: amount of 0.01 mol/l silver (I) nitrate solution (ml)

V : amount of 0.01 mol/l ammonium thiocyanate solution required for titration (ml).

(3.1.13) 0.1 mol/l (0.1 N) Sodium Thiosulfate Solution (24.82 g $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}/\text{l}$)

- (a) Preparation Weigh out 26 g of sodium thiosulfate specified in JIS K 8637 and 0.2 g of sodium carbonate (anhydride) specified in JIS K 8625. Dissolve in 1 l of water not containing dissolved oxygen, mix by sufficiently shaking, stopper, and allow to stand for 2 days. Thereafter, take the supernatant.
- (b) Standardization Dry potassium iodate specified in JIS K 8005 at a temperature of about 130°C for 2 h, and allow to stand to cool in a desiccator containing magnesium perchlorate. Weigh out accurately 0.09 to 0.11 g of potassium iodate into an interchangeable ground joint Erlenmeyer flask of 200 ml, and dissolve in 25 ml of water. Add 2 g of potassium iodide specified in JIS K 8913 and 5 ml of sulfuric acid (1 + 5) prepared by using sulfuric acid specified in JIS K 8951. Immediately stopper, moisten the interchangeable ground joint part with potassium iodide solution (10 w/v %), tightly stopper, and mix by gently shaking. Thereafter, allow to stand in a dark place for 5 min. Titrate with 0.1 mol/l sodium thiosulfate solution. When the solution turns from red brown to light yellow, add about 0.5 ml of starch solution of 2. (3.2) (e), further continue to titrate, and take a point at which the color of the solution turns from blue to colorless as the end point. Separately add 2 g potassium iodide and 5 ml of sulfuric acid (1 + 5) to 25 ml of water, and perform a blank test of this solution.
- (c) Calculation The factor shall be calculated from the following formula:

$$F = \frac{m}{0.003567 \times (V_1 - V_2)} \times \frac{A}{100}$$

where, F : factor of 0.1 mol/l sodium thiosulfate solution

m : mass of potassium iodate (g)

A : content of potassium iodate (%)

0.003567: mass of potassium iodate equivalent to 1 ml of 0.1 mol/l sodium thiosulfate solution (g)

V_1 : amount of 0.1 mol/l sodium thiosulfate solution required for titration (ml)

V_2 : amount of 0.1 mol/l sodium thiosulfate solution required for blank test (ml).

(3.1.14) 0.05 mol/l (0.1 N) Iodine Solution (12.69 g I/l)

- (a) Preparation Dissolve 40 g of potassium iodide specified in JIS K 8913 in 25 ml of water. Then, dissolve 13 g of iodine specified in JIS K 8920, add water to 1 l, add 3 drops of hydrochloric acid specified in JIS K 8180, and put into a brown bottle to be preserved.
- (b) Standardization Weigh out accurately 25 ml of 0.05 mol/l iodine solution into an interchangeable ground joint Erlenmeyer flask of 200 ml, and titrate with 0.1 mol/l (0.1 N) sodium thiosulfate solution of 2. (3.1.13). When the color of the solution turns from red brown to light yellow, add 0.5 ml of starch solution of 2. (3.2) (e), further continue to titrate, and take a point at which the color of the solution turns from blue to colorless as the end point.
- (c) Calculation The factor shall be calculated from the following formula:

$$F_1 = \frac{V \times F_2}{25}$$

where, F_1 : factor of 0.05 mol/l iodine solution

V : amount of 0.1 mol/l sodium thiosulfate solution required for titration (ml)

F_2 : factor of 0.1 mol/l sodium thiosulfate solution

25: amount of 0.05 mol/l iodine solution (ml).

(3.1.15) 0.05 mol/l (0.1 N) Sulfuric Acid (4.904 g H₂SO₄/l)

- (a) Preparation After gradually adding 3 ml of sulfuric acid specified in JIS K 8951 to 1 l of water while mixing by stirring, allow to stand to cool.
- (b) Standardization Put sodium carbonate (anhydride) specified in JIS K 8005 into a platinum crucible, heat at a temperature of about 600°C for 1 h, and allow to stand to cool in a desiccator containing magnesium perchlorate. Weigh out accurately 0.13 to 0.16 g of sodium carbonate into a conical beaker of 200 ml, and dissolve in 20 ml of water. Add 2 drops of Bromophenol Blue solution (0.1 w/v %) of 2. (3.2) (i), and titrate with 0.05 mol/l sulfuric acid. After the color of the solution has turned from blue purple to blue green, gently boil to remove carbon dioxide. After quenching, further continue to titrate, and take a point at which the color of the solution turns to blue green as the end point.

- (c) Calculation The factor shall be calculated from the following formula:

$$F = \frac{m}{0.005300 \times V} \times \frac{A}{100}$$

where, F : factor of 0.05 mol/l sulfuric acid

m : mass of sodium carbonate (anhydride) (g)

A : content of sodium carbonate (anhydride) (%)

0.005300: mass of sodium carbonate equivalent to 1 ml of 0.05 mol/l sulfuric acid (g)

V : mass of 0.05 mol/l sulfuric acid required for titration (ml).

(3.1.16) 0.1 mol/l (0.1 N) Ammonium Iron (III) Sulfate [48.22 g $\text{FeNH}_4(\text{SO}_4)_2 \cdot 12 \text{H}_2\text{O}/\text{l}$]

- (a) Preparation Weigh out 49 g of ammonium iron (III) sulfate (dodecahydrate) specified in JIS K 8982. After dissolving in 300 ml of water containing 10 ml of hydrochloric acid (1 + 1) prepared by using hydrochloric acid specified in JIS K 8180, add water to 1 l.
- (b) Standardization Weigh out accurately 25 ml of 0.1 mol/l ammonium iron (III) sulfate solution into an interchangeable ground joint Erlenmeyer flask of 200 ml, and add 35 ml of water and 3 ml of hydrochloric acid (1 + 1). Then add 3 g of potassium iodide specified in JIS K 8913, immediately stopper, and gently mix by shaking. Thereafter, allow to stand to cool in a dark place for 10 min, and titrate free iodine with 0.1 mol/l (0.1 N) sodium thiosulfate solution of 2. (3.1.13). When the color of the solution turns light yellow, add either 10 ml of sodium acetate solution (25 w/v %) prepared by using sodium acetate trihydrate specified in JIS K 8371 or 10 ml of ammonium acetate solution (25 w/v %) prepared by using ammonium acetate specified in JIS K 8359 and 0.5 ml of starch solution of 2. (3.2) (e), continue to titrate, and take a point at which the color of the solution turns from blue to colorless as the end point.

Separately, add 3 ml of hydrochloric acid (1 + 1) to 60 ml of water, then add 3 g of potassium iodide, immediately stopper, and gently mix by shaking. Thereafter, allow to stand in a dark place for 10 min, and perform a blank test.

- (c) Calculation The factor shall be calculated from the following formula:

$$F_1 = \frac{(V_1 - V_2) \times F_2}{25}$$

where, F_1 : factor of 0.1 mol/l ammonium iron (III) sulfate solution

V_1 : amount of 0.1 mol/l sodium thiosulfate solution required for titration (ml)

V_2 : amount of 0.1 mol/l sodium thiosulfate solution required for blank test (ml)

F_2 : factor of 0.1 mol/l sodium thiosulfate solution

25: amount of 0.1 mol/l ammonium iron (III) sulfate solution (ml).

(3.1.17) 0.1 mol/l (0.1 N) Diammonium Iron (II) Sulfate Solution
[39.21 g $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}/\text{l}$]

- (a) Preparation After gradually adding 30 ml of sulfuric acid specified in JIS K 8951 to 300 ml of water while mixing by stirring, allow to stand to cool. Then, dissolve 40 g of ferrous ammonium sulfate specified in JIS K 8979, and add water to make 1 l.
- (b) Standardization Weigh out accurately 25 ml of 0.1 mol/l diammonium iron (II) sulfate solution into a conical beaker of 200 ml, and add 25 ml of water and 5 ml of phosphoric acid specified in JIS K 9005. Titrate with 0.02 mol/l (0.1 N) potassium permanganate solution of 2. (3.1.5), and take a point at which the color of the solution turns light red as the end point. Separately add 5 ml of phosphoric acid to 25 ml of water, and perform a blank test of this solution. Standardize each service.
- (c) Calculation The factor shall be calculated from the following formula:

$$F_1 = \frac{(V_1 - V_2) \times F_2}{25}$$

where, F_1 : factor of 0.1 mol/l diammonium iron (II) sulfate solution

V_1 : amount of 0.02 mol/l potassium permanganate solution required for titration (ml)

V_2 : amount of 0.02 mol/l potassium permanganate solution required for blank test (ml)

F_2 : factor of 0.02 mol/l potassium permanganate solution

25: amount of 0.1 mol/l diammonium iron (II) sulfate solution (ml)

(3.2) Preparation of Indicators

- (a) Uranine Solution (0.2 w/v %) Weigh out 0.2 g of uranine specified in JIS K 8830, and dissolve in water to 100 ml.
- (b) Xylenol Orange Solution (0.1 w/v %) Weigh out 0.1 g of Xylenol Orange specified in JIS K 9563, dissolve in 100 ml of water, and put in a brown bottle to be preserved.

- (c) Potassium Thiocyanate Solution (Saturated) Weigh out about 65 g of potassium thiocyanate specified in JIS K 9001, and dissolve in 30 ml of water.
- (d) Thymol Blue Solution (0.1 w/v %) Weigh out 0.1 g of Thymol Blue specified in JIS K 8643, dissolve in 50 ml of ethanol (95) specified in JIS K 8102, dilute to 100 ml by adding water, and put in a brown bottle to be preserved.
- (e) Starch Solution Weigh out 1.0 g of starch specified in JIS K 8659. After mixing by adding about 10 ml of water, add into 100 ml of hot water while mixing by stirring. Boil for about 1 min. After cooling, take the supernatant or filter with filter paper. Prepare each service.
- (f) N-Paramethoxyphenyl-Phenylenediamine Hydrochloride Diluted Powder Weigh out 1 g of N-paramethoxyphenyl-phenylenediamine hydrochloride and 100 g of sodium chloride specified in JIS K 8150, grind with a glass mortar and a glass pestle, and put into a brown bottle to be preserved.
- (g) Phenolphthalein Solution (1 w/v %) Weigh out 1 g of phenolphthalein specified in JIS K 8799, dissolve in 90 ml of ethanol (95) specified in JIS K 8102, and add water to 100 ml.
- (h) Bromothymol Blue Solution (0.1 w/v %) Weigh out 0.1 g of Bromothymol Blue specified in JIS K 8842. After dissolving in 50 ml of ethanol (95) specified in JIS K 8102, add water to make 100 ml, and put into a brown bottle to be preserved.
- (i) Bromophenol Blue Solution (0.1 w/v %) Weigh out 0.1 g of Bromophenol Blue specified in JIS K 8844. After dissolving in 50 ml of ethanol (95) specified in JIS K 8102, add water to make 100 ml, and put into a brown bottle to be preserved.
- (j) Methyl Orange Solution (0.1 w/v %) Weigh out 0.1 g of Methyl Orange specified in JIS K 8893, dissolve in 100 ml of water, and put into a brown bottle to be preserved.
- (k) Methyl Red Solution (0.1 w/v %) Weigh out 0.1 g of Methyl Red specified in JIS K 8896, dissolve in 100 ml of ethanol (95) specified in JIS K 8102, and put into a brown bottle to be preserved.
- (l) Methyl Red-Methylene Blue Mixed Solution Weigh out 0.1 g of Methyl Red specified in JIS K 8896 and 0.1 g of Methylene Blue specified in JIS K 8897, dissolve in 200 ml of ethanol (95) specified in JIS K 8102, and put into a brown bottle to be preserved.
- (m) Ammonium Iron (III) Sulfate Solution Weigh out 20 g of ammonium iron (III) sulfate-dodecahydrate specified in JIS K 8982. After dissolving in 80 ml of water, add 20 ml of sulfuric acid (1 + 3) prepared by using sulfuric acid specified in JIS K 8951.

- (3.3) Preparation of Buffer Solution, Acetic Acid-Sodium Acetate Buffer Solution (pH Value 5) Weigh out 66 g of sodium acetate trihydrate specified in JIS K 8371. After dissolving in 600 ml of water, add 10 ml of acetic acid specified in JIS K 8355. Adjust pH value to 5 by adding sodium acetate or acetic acid.

3. Sampling Method

The sampling method shall be in accordance with 2. of JIS K 5400.

4. Non-Volatile Matter

- (1) Summary A residue when evaporated components are removed by heating a sample is taken as a non-volatile matter, which is obtained as the percentage in the sample.

When a small amount of non-volatile matter in a volatile matter is to be obtained, most of the volatile matter is removed by distillation and then the sample is heated.

- (2) Apparatus The apparatus shall be as follows:

- (a) Weighing Bottle A flat type weighing bottle of 60 mm × 30 mm specified in JIS R 3503.
- (b) Glass Rod A glass rod of about 3 mm in diameter and about 60 to 70 mm in length.
- (c) Drying Oven An electric thermostat with an exhaust hole capable of keeping at a temperature $\pm 2^{\circ}\text{C}$, which is specified in a product Standard.
- (d) Evaporating Dish ⁽¹⁾ An evaporating dish with flat bottom of 90 mm × 45 mm specified in JIS R 3503.
- (e) Distillation Apparatus ⁽¹⁾ A distillation apparatus specified in 10.

Note ⁽¹⁾ This is used when non-volatile content of volatile matter is obtained.

- (3) Operation The operation shall be carried out as follows.

(3.1) When Non-volatile Content of Sample Is High

- (a) Dry a weighing bottle and a glass rod in a drying oven at a temperature of $105 \pm 2^{\circ}\text{C}$. After standing to cool in a desiccator, put the glass rod on the weighing bottle, and weigh the mass.
- (b) Quickly take a sample ⁽²⁾ into the weighing bottle ⁽³⁾ ⁽⁴⁾, put the glass rod thereon, and weigh the mass.
- (c) Spread the sample on the bottom face of container with glass rod and unless otherwise specified, heat in the drying oven containing ⁽⁵⁾ the glass rod as it is at a temperature of $105 \pm 2^{\circ}\text{C}$ for 3 h. When a skin is formed on the surface of the sample during heating, break the skin with the glass rod occasionally.

- (d) After ending heating, take the weighing bottle and the glass rod out of the drying oven. After standing to cool in the desiccator, weigh the mass and obtain the amount of residue in the weighing bottle.

Notes (2) The amount of the sample is about 2 g for that containing pigment and about 1.5 g for that containing no pigment.

- (3) Put a dish prepared by using a foil IN 30 of 0.015 mm in thickness specified in JIS H 4160 inside the weighing bottle, and allow the sample to be weighed out in the said dish.

- (4) In the case of the sample of slow evaporation speed, use any one of a container of 60 mm in diameter of a form like a Petri dish of which material is glass, tinplate, or aluminium.

- (5) In the case of paint containing much solvent of a low flash point, for the weighing bottle containing a sample, remove the cover and put it in a room for 30 min before putting into a drying oven.

(3.2) When Non-volatile Content of Sample Is Low

- (a) In order to obtain a small amount of non-volatile content in volatile matter, weigh out 100 ml of a sample with a measuring cylinder, and put into a distillation flask.
- (b) After carrying out distillation until 75 ml is distilled in accordance with 10., stop heating, and remove the flask.
- (c) Transfer contents of the flask to an evaporation dish (6) of which the mass is known, successively erect upside down for 15 s, and transfer residual contents sticking to the inner wall.
- (d) Heat an evaporation dish on a water bath, and evaporate most of the residue.
- (e) Heat in a drying oven at a temperature of $105 \pm 2^\circ\text{C}$ for 2 h. After standing to cool in a desiccator, weigh the mass of the evaporation dish, and obtain the mass of non-volatile matter.

Note (6) Preliminarily put a boiling stone on the evaporation dish, and heat in a drying oven at a temperature of $105 \pm 2^\circ\text{C}$ to constant mass.

(4) Calculation The calculation shall be carried out as follows:

- (4.1) When the non-volatile matter in the sample is much, the non-volatile matter shall be calculated from the following formula:

$$A = \frac{m_2}{m_1} \times 100$$

where, A: non-volatile matter (g)

m_2 : residual amount in weighing bottle (g)

m_1 : mass of sample (g).

(4.2) When the heating residue of a sample is little, the mass of the heating residue of (3.2) (e) is the heating residue (g/100 ml) per 100 ml of the sample.

5. Volatile Matter

(1) Summary An evaporation component when a sample is heated is taken volatile matter, which is obtained as the percentage in the sample.

(2) Apparatus The apparatus shall be as follows:

(a) Weighing Bottle A flat type weighing bottle of 60 mm × 30 mm specified in JIS R 3503.

(b) Glass Rod A glass rod of about 3 mm in diameter and about 60 to 70 mm in length.

(c) Drying Oven An electric oven with an exhaust hole capable of keeping at a temperature $\pm 2^\circ\text{C}$ by thermostat, which is specified in a product Standard.

(3) Operation The operation shall be carried out as follows:

(a) Dry a weighing bottle and a glass rod in a drying oven at $105 \pm 2^\circ\text{C}$ in temperature. After standing to cool in a desiccator, put the glass rod on the weighing bottle, and weigh the mass.

(b) Quickly take a sample ⁽¹⁾ into the weighing bottle ⁽²⁾ ⁽³⁾, put the glass rod thereon, and weigh the mass.

(c) Spread the sample on the bottom face of a container with the glass rod and unless otherwise specified, heat in the drying oven ⁽⁴⁾ containing the glass rod as it is at a temperature of $105 \pm 2^\circ\text{C}$ for 3 h. When a skin is formed on the surface on the sample during heating, break the skin with the glass rod at times.

(d) After ending heating, take the weighing bottle and the glass rod out of the drying oven. After standing to cool in the desiccator, weigh the mass and obtain the amount of residue in the weighing bottle.

Notes ⁽¹⁾ The amount of the sample is about 2 g for that containing pigment and about 1.5 g for that containing no pigment.

⁽²⁾ Put a dish prepared by using foil IN 30 of 0.015 mm in thickness specified in JIS H 4160 inside the weighing bottle, and allow the sample to be weighed out in the said dish.

- (³) In the case of a container of slow evaporation speed, use any one of a container of 60 mm in diameter of a form like a Petri dish of which material is glass, tinplate, or aluminium.
- (⁴) In the case of paint containing much solvent of a low flash point, for the weighing bottle containing a sample, remove the cover and put it into a room for 30 min before putting into a drying oven.
- (4) Calculation The volatile matter shall be calculated from the following formula:

$$A = \frac{m_1 - m_2}{m_1} \times 100$$

where, A: volatile matter (%)

m_1 : mass of sample (g)

m_2 : residual amount in weighing bottle (g).

6. Solvent Insoluble Matter

- (1) Summary Solvent soluble matter is dissolved by adding solvent to a sample, and solid matter obtained by centrifugal separation is taken as solvent insoluble matter, which is obtained as the percentage in the sample.
- (2) Apparatus The apparatus shall be as follows:
- (a) Centrifugal Separator A centrifugal separator of 3000 to 15000 r.p.m. in function.
- (b) Precipitation Tube A glass or stainless steel precipitation tube with a round bottom of about 50 ml in capacity.
- (3) Operation The operation shall be carried out as follows:
- (a) Weigh out accurately 10 g of a sample into a precipitation tube of which the mass is preliminarily weighed.
- (b) Add about 20 ml of solvent specified in product Standard (¹), and sufficiently mix with a glass rod. Wash solid matter sticking to the glass rod (²) out into a precipitation tube with solvent, and add solvent until the amount of solution becomes about four-fifths of the precipitation tube.
- (c) After covering the precipitation tube with a cork stopper to prevent evaporation of solvent, separate at 3000 to 15000 r.p.m. with a centrifugal separator for 20 to 30 min (³).
- (d) Take out the supernatant in the precipitation tube to be preserved, and use for a test of solvent soluble matter.

- (e) Further use 30 ml of solvent each time, and repeat the operations of mixing, precipitation, and outflow three times in accordance with (b) to (d).
- (f) After dipping the bottom of the precipitation tube into hot water, lightly strike the bottom against 5 to 6 sheets of cotton cloth piled on a wooden board several times, repeat this operation, and separate the precipitate from the inner wall.
- (g) Dry the precipitation tube in a drying oven at a temperature of $105 \pm 2^\circ\text{C}$ for 2 h ⁽⁴⁾. After standing to cool in a desiccator, weigh the mass and obtain the mass of the precipitate.

When this solvent insoluble matter is used as the sample of an analysis, after sufficiently grinding with an agate mortar to homogeneity, transfer to a weighing bottle. After drying in the drying oven at a temperature of $105 \pm 2^\circ\text{C}$ ⁽⁴⁾ for 30 min, use it.

- Notes
- ⁽¹⁾ When a sample is emulsion paint, use water two times, then acetone specified in JIS K 8034 two times, and further a mixed solution of toluene specified in JIS K 8680 and acetone by 1 to 1 in volume ratio, repeat the operation of mixing, precipitation and outflow two times, and remove the solvent soluble matter.
 - ⁽²⁾ When solid matter sticks to a glass rod, always weigh the mass by combining the glass rod and a precipitation tube.
 - ⁽³⁾ Generally apply an centrifugal separator at 3000 to 4000 r.p.m. for 20 to 30 min. However, when pigments difficult to be separated such as carbon black, chromate, organic pigment, etc. are contained, apply the centrifugal separator at 8000 to 15000 r.p.m. for 30 min.
 - ⁽⁴⁾ When copper (I) oxide is contained, dry in a drying oven at a temperature of $55 \pm 2^\circ\text{C}$ for 30 min, allow to stand in a desiccator for 24 h, and obtain the mass of the precipitate.

- (4) Calculation Calculate solvent insoluble matter in a sample from the following formula:

$$A = \frac{m_2}{m_1} \times 100$$

where, A : solvent insoluble matter (%)

m_2 : mass of precipitate (g)

m_1 : mass of sample (g).

7. Alcohol Insoluble Matter

- (1) Summary A sample is mixed in ethanol. After dissolution by boiling, extraction is performed by using an extractor, and its residue is taken as alcohol insoluble matter, which is obtained as the percentage in the sample.

(2) Reagent Ethanol (95) Ethanol (95) specified in JIS K 8102.

(3) Apparatus The apparatus shall be as follows:

(a) Filter An example of the shape and dimensions thereof is shown in Fig. 2.

(b) Water Bath A copper or stainless steel water bath of 100 mm in width. An example of the shape and dimensions thereof is given in Fig. 3.

(c) Extractor An extractor is composed of a condenser, a syphon cup, and an Erlenmeyer flask. An example of the shape and dimensions thereof is given in Fig. 4. They are assembled as follows:

Attach a cork stopper to an under-tube of the condenser, insert the top of the under-tube into the upper part of the syphon cup, pass a piece of a straight wire through holes provided thereto, connect them, hang the syphon cup in the Erlenmeyer flask, and attach it to the lower tube of the condenser with the cork stopper so as not to be leaked. Pass water through the condenser, put several boiling stones and ethanol (95) into the Erlenmeyer flask, heat the Erlenmeyer flask in a water bath, and make the syphon cup be wrapped by vapor of ethanol at a temperature near a boiling point.

(d) Weighing Bottle A cylindrical weighing bottle of 40 mm × 80 mm specified in JIS R 3503.

(e) Extraction Thimble An extraction thimble of about 25 mm in outer diameter and about 55 mm in height. Preliminarily put in the syphon cup of an extractor, use ethanol (95), and extract for about 30 min by operation in the same way as in (c). Put the extraction thimble into a weighing bottle, and dry at a temperature of $105 \pm 2^\circ\text{C}$ to constant mass.

Fig. 2. An Example of Filter

Material: Hard glass

Unit: mm

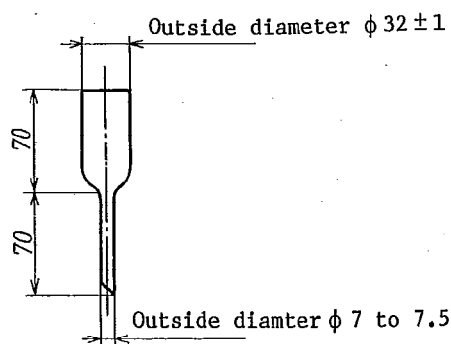


Fig. 3. An Example of Water Bath

Unit: mm

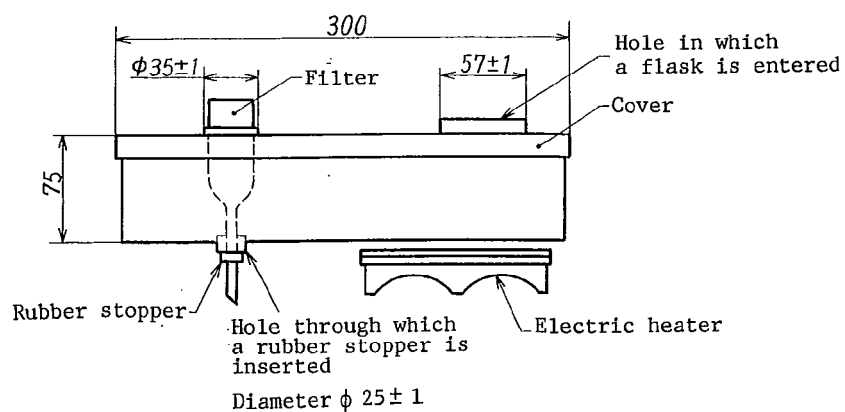
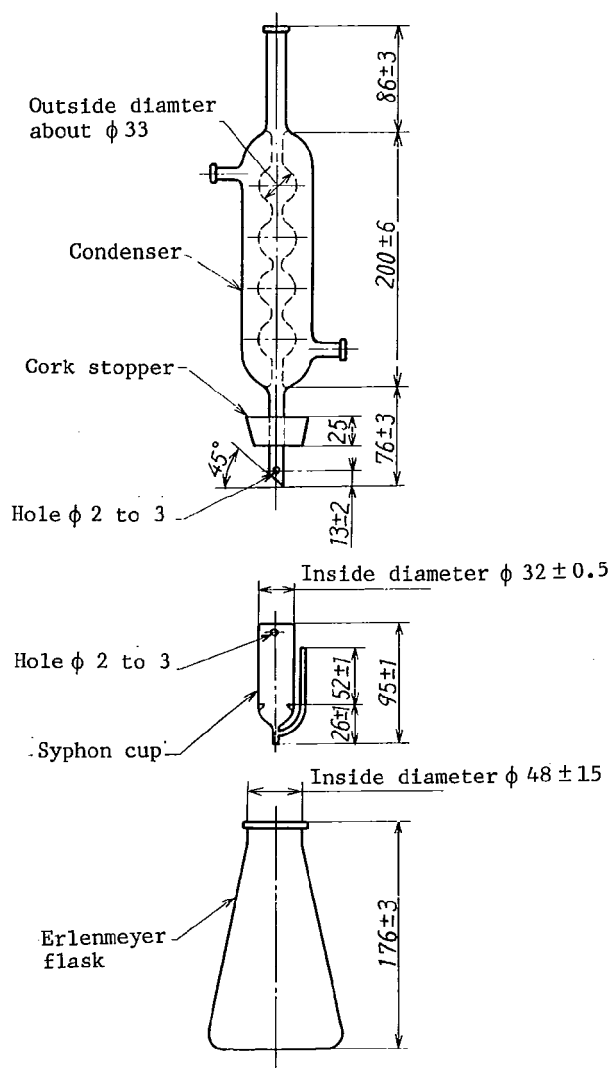


Fig. 4. An Example of Extractor

Unit: mm



(4) Operation Carry out the operation as follows:

- (a) Sufficiently mix by stirring a sample, and weigh out accurately about 15 g therefrom into a conical beaker of 100 ml. Separately obtain heating residue in accordance with 4.
- (b) Add 60 ml of ethanol (95), cover with a watch glass, dissolve the sample by boiling ethanol (95) on a water bath, and keep at a temperature near the boiling point for 30 min.
- (c) Insert an extraction thimble of which the mass is preliminarily weighed into a filter, and moisten with boiling ethanol (95). After quickly filtering the sample solution into the extraction thimble so that temperature does not decrease, use 75 ml of boiling ethanol (95), and transfer the residue in the beaker to the extraction thimble by washing.
- (d) Put 125 ml of ethanol (95) into an Erlenmeyer flask containing boiling stones, put the extraction thimble into a syphon cup, boil and extract in a water bath for 1 h.
- (e) Put the extraction thimble into a weighing bottle, put into a drying oven kept at a temperature of $105 \pm 2^\circ\text{C}$, and heat for 1 h. After standing to cool in a desiccator, weigh the mass.
- (f) Put it into the drying oven kept at a temperature of $105 \pm 2^\circ\text{C}$ again, dry by heating for 1 h, and allow to stand to cool in a desiccator. Thereafter, weigh the mass. Repeat until a difference in mass between the former and the latter becomes 2 mg or under, weigh the mass of the extraction thimble, and obtain the mass of the residue.

(5) Calculation Calculate alcohol insoluble matter from the following formula:

$$A = \frac{m_2}{m_1 \times B} \times 100 \times 100$$

where, A : alcohol insoluble matter (%)

m_2 : mass of residue (g)

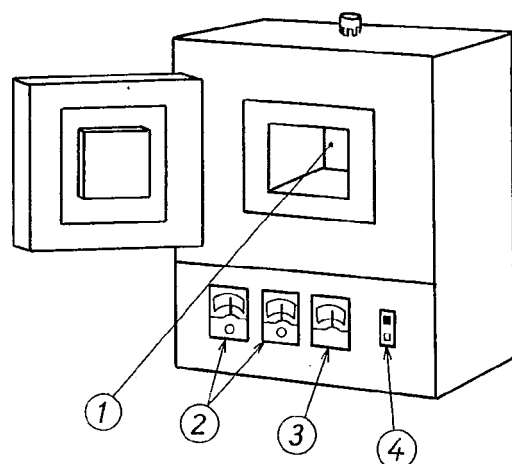
m_1 : mass of sample (g)

B : heating residue of sample according to 4. (%).

8. Ash Content

- (1) Summary A sample is incinerated by heating, and ash content is obtained as percentage from its residue.
- (2) Apparatus Electric Furnace An electric furnace capable of keeping at a temperature of about 550°C . An example thereof is shown in Fig. 5.

Fig. 5. An Example of Electric Furnace



- ① : Heat-resisting brick furnace
- ② : Current regulating dial
- ③ : Temperature indicator
- ④ : Power source switch

(3) Operation Carry out the operation as follows:

- (a) Weigh out accurately about 10 g of a sample into a porcelain crucible PC 1 B type of 30 ml which is preliminarily heated at a temperature of about 550°C to constant mass. In order to obtain ash content in heating residue, separately obtain the non-volatile matter preliminarily in accordance with 4.
- (b) When a sample contains volatile components, heat on a water bath, and remove most of the volatile components.
- (c) Gradually heat with a gas burner so that the sample is not scattered, and carbonize the sample.
- (d) Put the porcelain crucible into an electric furnace ⁽¹⁾ kept at a temperature of about 550°C, and heat for 1 h. After standing to cool in a desiccator, weigh the mass.
- (e) Put the porcelain crucible into the electric furnace again, heat for 30 min, and allow to stand to cool in the desiccator. Thereafter, weigh the mass. Repeat this operation until a difference in mass between the former and the latter becomes 1 mg or under.
- (f) Obtain the residue in the porcelain crucible.

Note ⁽¹⁾ The porcelain crucible may be heated with a gas burner instead of the electric furnace.

(4) Calculation Obtain ash content as follows:

- (a) Calculate the ash content in the whole sample from the following formula:

$$A_1 = \frac{m_1}{m_2} \times 100$$

where, A_1 : ash content in the whole sample (%)

m_1 : residue in porcelain crucible (g)

m_2 : mass of sample (g).

- (b) Calculate the ash content in the non-volatile matter from the following formula:

$$A_2 = \frac{m_3}{m_4 \times B} \times 100 \times 100$$

where, A_2 : ash content in heating residue (%)

m_3 : residue in porcelain crucible (g)

m_4 : mass of sample (g)

B : non-volatile matter of sample obtained in accordance with 4. (%)

9. Determination of Water Content (Karl Fischer's Method)

A Karl Fischer's reagent is used, and water content contained in a sample is measured to be represented by percentage. There are volumetric titration method and coulometric titration method.

General matters common to Karl Fischer's method shall be in accordance with JIS K 0113.

9.1 Volumetric Titration Method

- (1) Summary Titration is performed by using Karl Fischer's reagent, and water content is obtained from its consumption.

- (2) Reagents The reagents shall be as follows:

- (2.1) Mixed Solvent ⁽¹⁾ A mixture of methanol dehydrated in accordance with JIS K 0113 and chloroform specified in JIS K 8322 by 1 to 3 in volumetric ratio, and otherwise a mixed solvent suitable for titration by dissolving a sample. When there is a possibility for presence of aldehydes or ketones in the sample, a mixture of pyridine specified in JIS K 8777 which is dehydrated in accordance with JIS K 0113 and ethylene glycol specified in JIS K 8105 by 4 to 1 in volumetric ratio.

Note ⁽¹⁾ Products on the market equal thereto in quality may be used.

- (2.2) Karl Fischer's Reagent ⁽¹⁾ A Karl Fischer's reagent specified in 6.2 (4) of JIS K 0113. When aldehydes or ketones exist in a sample, a reagent not containing methanol.

(3) Apparatus The apparatus shall be as follows:

- (3.1) Apparatus for Volumetric Titration An apparatus for volumetric titration is composed of a titration part, a measuring control part, and an indication part, and an example thereof is shown in Fig. 6.

Remark: Handling of the apparatus is in accordance with the instruction manual.

- (3.2) Vaporization Apparatus Water content in a sample is vaporized by heating the sample, and is conducted to a titration apparatus by dry nitrogen or the like. An example thereof is given in Fig. 7.

Remark: Handling of the apparatus is in accordance with the instruction manual.

- (3.3) Sampler A sampler capable of weighing out a sample under no influence of outside air water content.

Fig. 6. An Example of Structure of Apparatus for Volumetric Titration

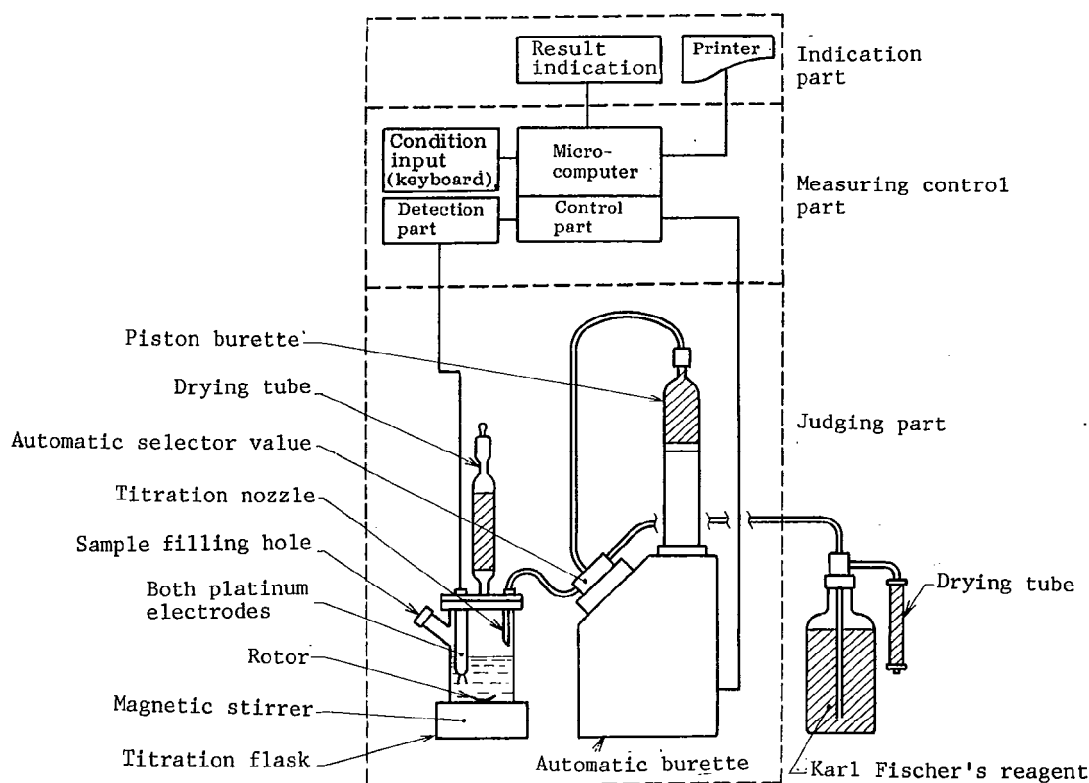
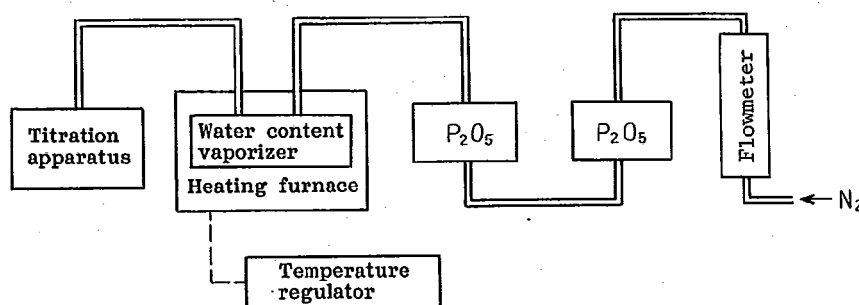


Fig. 7. An Example of Schematic Vaporization Apparatus

**(4) Operation** The operation shall be carried out as follows:

- (a) Take 100 ml of mixed solvent into a titration flask. After stoppering, actuate a stirrer. Then, start titration, titrate to the end point with Karl Fischer's reagent, and make the inside of the titration flask be under an anhydrous state.
- (b) Use a sampler⁽²⁾ which is preliminarily dried at a temperature of about 150°C for 1 h or over and is allowed to stand to cool in a desiccator, sample an amount containing water content equivalent to 5 to 10 ml of Karl Fischer's reagent of a sample⁽³⁾, and quickly add into a titration flask. Obtain a sampling amount by weighing a difference in mass of the sampler between before and after addition of the sample to the nearest 0.1 mg.

When there is a possibility for containing interfering substance in the sample, preliminarily adjust in accordance with Table 1. When non-volatile interfering substance is contained, use a vaporization apparatus⁽⁴⁾.

An example of substance using the vaporization apparatus is given in Table 2.

Notes (2) When a syringe is used as a sampler, take the sample to about one-tenth of the syringe so as to remove leak of air from the grinding fit part of the syringe. Then, make the needle upward, remove bubbles, insert a silicone rubber piece to the top of the syringe, and moisten the grinding fit part of the syringe with the sample by adding pressure. Thereafter discard the sample. After repeating this operation two or three times, gradually take the sample into the syringe.

Weigh the mass while the silicone rubber piece is attached thereto as it is, then remove the silicone rubber piece, and put the sample into a titration flask. Thereafter, weigh the mass by attaching the used silicone rubber piece, and take the difference between the two weighings as the sampling amount of the sample.

(³) Since the sample is apt to be influenced by open air water content or the like, take care for the following points.

(1) A sample container is unsealed after the temperature of a sample becomes room temperature.

(2) A test is carried out just after unsealing.

(⁴) For conditions of a vaporization apparatus, select conditions suitable for the sample.

Table 1. Example of Substance Capable of Removing Interference by Adjusting Condition for Solution

Name of substance	Condition for solution
Aldehydes and ketones	Use a mixed solution of pyridine and ethylene glycol as a solvent, and titrate with Karl Fischer's reagent not containing methanol
Amines	After neutralizing with acetic acid, further add in excess.

Table 2. Example of Substance Using Vaporization Apparatus

Division	Name of substance
Metallic oxide substance (⁵)	Zinc oxide, calcium oxide, silver (I) oxide, mercury (II) oxide, copper (I) oxide, lead (II) oxide, lead (IV) oxide, lead (II) lead (IV) oxide, magnesium oxide, and manganese (IV) oxide

Note (⁵) Aluminium oxide, copper (II) oxide, nickel (II) oxide, and barium oxide do not interfere.

Remark: For other interfering substances, refer to 6.5 of JIS K 0113.

(c) Titrate again until the end point in the same way as in (a), and read the amount of Karl Fischer's reagent required for this titration to the nearest 0.01 ml.

(d) When a sample is cast by unstoppering a sample filling hole in accordance with (b), carry out a blank test by performing the operation on and after (b) in the same way.

(5) Calculation Calculate water content from the following formula:

$$A_1 = \frac{(V_1 - V_2) \times F_1}{m_1 \times 10^3} \times 100$$

where, A_1 : water content (%)

V_1 : amount of Karl Fischer's reagent required for titration (ml)

V_2 : amount of Karl Fischer's reagent required for blank test (ml)
(only when the blank test is carried out)

F_1 : potency of Karl Fischer's reagent (mg H_2O /ml)

m_1 : mass of sample (g).

9.2 Coulometric Titration Method

(1) Summary Coulometric titration is performed by using Karl Fischer's reagent in which iodide ion is mixed instead of iodine, and the amount of water content is read from the consumed quantity of electricity.

(2) Reagents The reagents shall be as follows:

(2.1) Electrolytic Solution The following electrolytic solutions are combined to be used.

(a) Anolyte ⁽¹⁾ A mixed solution of organic solvents such as iodide ion, sulfur dioxide, pyridine or base in the place thereof, methanol, etc.

(b) Catholyte ⁽¹⁾ A mixed solution of electrolytes such as sulfur dioxide, pyridine or base or the like in the place thereof and organic solvents such as methanol or the like.

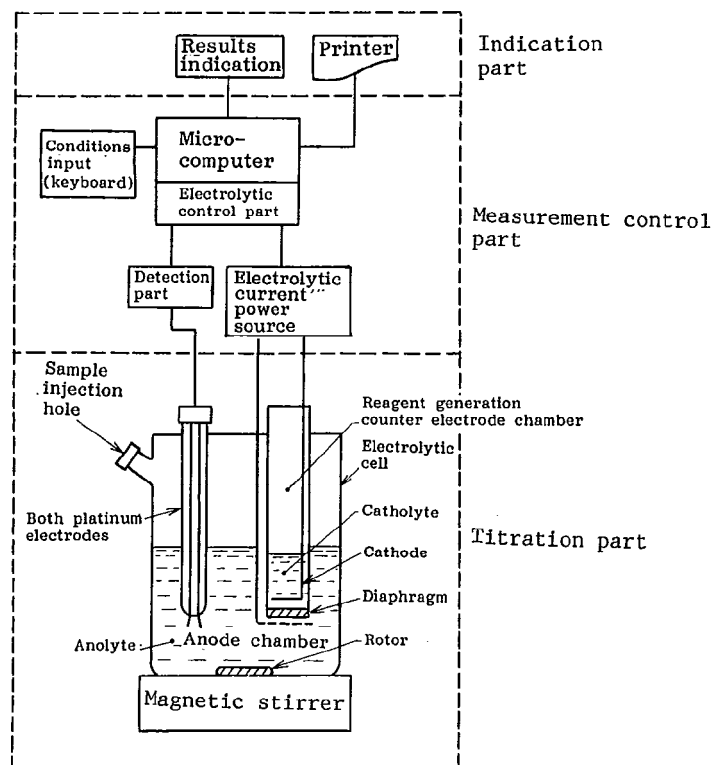
Informative Reference: For the composition of electrolytic solution, refer to 6.4.2 (1) Note ⁽⁹⁾ of JIS K 0113.

(3) Apparatus The apparatus shall be as follows:

(3.1) Apparatus for Coulometric Titration An apparatus for coulometric titration is composed of an electrolytic cell, a current control part, a detector, and an indication part. An example thereof is shown in Fig. 8.

Remark: Handling of the apparatus shall be in accordance with the instruction manual.

Fig. 8. An Example of Structure of Apparatus for Coulometric Titration



(3.2) Vaporization Apparatus A vaporization apparatus specified in (3.2) of 9.1.

(4) Operation The operation shall be carried out as follows:

- (a) Put about 100 ml of analyte into the reagent generation electrode chamber of an electrolytic cell and 5 to 10 ml of catholyte into the reagent generation counter electrode chamber. After sealing the electrolytic cell, attach it to the apparatus.
- (b) Cause electrolytic current to flow while stirring the analyte in the electrolytic cell, generate iodine at the analyte side, and make the inside of the electrolytic cell be under an anhydrous state.

Remark: Calibrate a tester as follows:

After making the inside of the electrolytic cell be under an anhydrous state in accordance with (4) (a) and (4) (b), add accurately 5 μ l of water from the sample injection hole with a microsyringe, perform coulometric titration to the end point, and obtain the amount of water content. Repeat this operation two times or over, and obtain the average of the amount of

water content. When the average value is within a range of $5000 \pm 250 \mu\text{g}$, take the tester as being normally operated. When it exceeds the range of $5000 \pm 250 \mu\text{g}$, adjust the tester again in accordance with the instruction manual, and perform the calibrating operation again.

- (c) Use the sampler ⁽²⁾ which is preliminarily dried at a temperature of about 150°C for 1 h or over and is allowed to stand to cool in a desiccator, and quickly add the sample ⁽³⁾ into the reagent generation electrode chamber of an electrolytic cell so as to contain about 20 mg of water content by using a sampler ⁽²⁾, obtain the sampling amount by weighing the mass of the sampler before and after adding the sample to the nearest 0.1 mg.

In the case where there is a probability of containing an interfering substance in the sample, preliminarily adjust anolyte in the same way as in Table 1 of 9.1 (4) (b). When nonvolatile interfering substance exists, allow a vaporization apparatus ⁽⁴⁾ to be used preferably. An example of substance using vaporization apparatus is shown in Table 2.

- (d) Allow an electrolytic current to flow while stirring anolyte, perform coulometric titration to the end point, and obtain the amount of water content.
- (5) Calculation Calculate water content from the following formula:

$$A_2 = \frac{m_2}{m_3 \times 10^6} \times 100$$

where, A_2 : water content (%)

m_2 : amount of water content of sample (μg)

m_3 : mass of sample (g).

10. Distillation Test

- (1) Summary A sample is distilled by a distillation test method used for paint, and the relation between the amount of distillate and the temperature of distillate is obtained.
- (2) Definitions The meanings of principal terms used in this clause shall be as follows:
- (a) initial boiling point ($^\circ\text{C}$) A reading of the thermometer when the initial one drop of distillate drops from the bottom end of a condenser tube.
- (b) end point ($^\circ\text{C}$) A reading of the highest temperature obtained in the last process of distillation test, which generally appears after the sample in the bottom part of a flask is thoroughly gasified.
- (c) dry point ($^\circ\text{C}$) A reading of the thermometer when the lowest part in a flask is dried. At that time, the inner surface of the flask and the surface of a thermometer may be under a wet state.