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Test methods for soaps

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Foreword

This Japanese Industrial Standard has been revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by Japan Soap and Detergent Association (JSDA)/Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14.

Consequently **JIS K 3304**:2006 is replaced with this Standard.

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Test methods for soaps

Introduction

This Japanese Industrial Standard has been prepared based on **ISO 456:1973**, Edition 1, **ISO 457:1983**, Edition 2, **ISO 672:1978**, Edition 1, **ISO 673:1981**, Edition 2, **ISO 684:1974**, Edition 1, **ISO 685:1975**, Edition 1, **ISO 1066:1975**, Edition 1, **ISO 1067:1974**, Edition 1, **ISO 2272:1989**, Edition 2, **ISO 4318:1989**, Edition 2, **ISO 4323:1977**, Edition 1, **ISO 4325:1990**, Edition 2, and **ISO 8212:1986**, Edition 1 with some modifications of the technical contents in order to re-adopt the evaluation method of the detergency of powdery soaps as the test method in this Standard, which has been deleted in the previous revision, in view of the change in the market of detergents for clothing and the change in the laundering environment.

The vertical lines on both sides and dotted underlines indicate changes from the corresponding International Standard. A list of modifications with the explanations is given in Annex JB.

1 Scope

This Standard specifies the test methods for soaps except those containing a surface active agent or synthetic detergent.

NOTE The International Standards corresponding to this Standard and the symbol of degree of correspondence are as follows.

ISO 456:1973 *Surface active agents—Analysis of soaps—Determination of free caustic alkali*

ISO 457:1983 *Soaps—Determination of chloride content—Titrimetric method*

ISO 672:1978 *Soaps—Determination of moisture and volatile matter content—Oven method*

ISO 673:1981 *Soaps—Determination of content of ethanol-insoluble matter*

ISO 684:1974 *Analysis of soaps—Determination of total free alkali*

ISO 685:1975 *Analysis of soaps—Determination of total alkali content and total fatty matter content*

ISO 1066:1975 *Analysis of soaps—Determination of glycerol content—Titrimetric method*

ISO 1067:1974 *Analysis of soaps—Determination of unsaponifiable, unsaponified and unsaponified saponifiable matter*

ISO 2272:1989 *Surface active agents—Soaps—Determination of low contents of free glycerol by molecular absorption spectrometry*

ISO 4318:1989 *Surface active agents and soaps—Determination of water content—Azeotropic distillation method*

ISO 4323:1977 *Soaps—Determination of chlorides content—Potentiometric method*

ISO 4325:1990 *Soaps and detergents—Determination of chelating agent content—Titrimetric method*

ISO 8212:1986 *Soaps and detergents—Techniques of sampling during manufacture* (overall evaluation: MOD)

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standards and **JIS** are IDT (identical), MOD (modified), and NEQ (not equivalent) according to **ISO/IEC Guide 21-1**.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS K 0050 *General rules for chemical analysis*

JIS K 0113 *General rules for methods of potentiometric, amperometric, coulometric, and Karl Fischer titrations*

JIS K 0115 *General rules for molecular absorptiometric analysis*

JIS K 3211 *Technical terms for surface active agents*

JIS K 3331 *Hardened oils and fatty acids for industrial use*

JIS K 3362 *Test method of household synthetic detergent*

JIS K 8001 *General rules for test methods of reagents*

JIS K 8005 *Reference materials for volumetric analysis*

JIS K 8034 *Acetone (Reagent)*

JIS K 8101 *Ethanol (99.5) (Reagent)*

JIS K 8102 *Ethanol (95) (Reagent)*

JIS K 8103 *Diethyl ether (Reagent)*

JIS K 8105 *Ethylene glycol (Reagent)*

JIS K 8122 *Calcium chloride dihydrate (Reagent)*

JIS K 8136 *Tin(II) chloride dihydrate (Reagent)*

JIS K 8155 *Barium chloride dihydrate (Reagent)*

JIS K 8159 *Magnesium chloride hexahydrate (Reagent)*

JIS K 8180 *Hydrochloric acid (Reagent)*

JIS K 8256 *Sodium periodate (Reagent)*

JIS K 8271 *Xylene (Reagent)*

JIS K 8295 *Glycerol (Reagent)*

JIS K 8316 *Disodium chromotropate dihydrate (Reagent)*

JIS K 8541 *Nitric acid (Reagent)*

JIS K 8574 *Potassium hydroxide (Reagent)*

- JIS K 8576 *Sodium hydroxide (Reagent)*
 JIS K 8577 *Barium hydroxide octahydrate (Reagent)*
 JIS K 8585 *Stearic acid (Reagent)*
 JIS K 8593 *Petroleum ether (Reagent)*
 JIS K 8622 *Sodium hydrogen carbonate (Reagent)*
 JIS K 8625 *Sodium carbonate (Reagent)*
 JIS K 8643 *Thymol blue (Reagent)*
 JIS K 8756 *Palmitic acid (Reagent)*
 JIS K 8799 *Phenolphthalein (Reagent)*
 JIS K 8842 *Bromothymol blue (Reagent)*
 JIS K 8844 *Bromophenol blue (Reagent)*
 JIS K 8982 *Ammonium iron (III) sulfate 12-water (Reagent)*
 JIS K 8983 *Copper (II) sulfate pentahydrate (Reagent)*
 JIS K 8987 *Sodium sulfate (Reagent)*
 JIS L 0803 *Standard adjacent fabrics for staining of colour fastness test*
 JIS M 8100 *Particulate materials—General rules for methods of sampling*
 JIS P 3801 *Filter paper (for chemical analysis)*
 JIS R 3503 *Glass apparatus for chemical analysis*
- NOTE Corresponding International Standard: ISO 383:1976 *Laboratory glassware—Interchangeable conical ground joints (IDT)*
- JIS Z 8723 *Methods for visual comparison of surface colours*
 JIS Z 8801-1 *Test sieves—Part 1: Test sieves of metal wire cloth*
 JIS Z 8802 *Methods for determination of pH of aqueous solutions*

3 Terms and definitions

For the purpose of this Standard, the terms and definitions given in **JIS K 3211**, and the following apply.

3.1

soap

product mainly composed of sodium salt (sodium soap) or potassium salt (potassium soap) of higher fatty acid

4 Test items

The test items on the quality of soap and the applicable clauses are shown in Table 1. The evaluation method of detergency of soaps shall be as specified in Clause 7.

Table 1 Test items

Test item	Applicable clause
Water content	6.1
Petroleum ether soluble matter	6.2
Total alkali and total fatty matter	6.3
Pure soapy matter	6.4
Free alkali	6.5
Total free alkali	6.6
Ethanol-insoluble matter	6.7
Water-insoluble matter	6.8
Chloride	6.9
Glycerol	6.10
Ethylenediaminetetraacetate (edetic acid)	6.11
pH value	6.12

5 Common matters

5.1 General

General matters common to the tests shall be in accordance with **JIS K 0050**.

5.2 Preparation of test sample

5.2.1 Types of sample

Samples are classified as follows.

- a) **Solid soap** Soaps in such forms as bars, pieces and tablets
- b) **Powdery lump soap** Soaps in such forms as powder, grains, flakes and chips
- c) **Soft soap** Soaps in pasty state, gel state, etc.
- d) **Liquid soap** Soaps in transparent liquid state, emulsion state, etc.

5.2.2 Appliances

The appliances used for the preparation of samples shall be as follows.

- a) **Knife**
- b) **Electric mixer**
- c) **Riffle sampler** No. 6 riffle sampler specified in Table 4 of **JIS M 8100**.
- d) **Spatula for division** A flat plate made of stainless steel or synthetic resin, used for division by the coning and quartering method (see Figure 1).

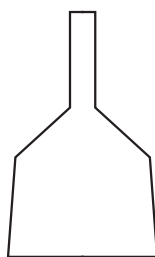


Figure 1 Spatula for division

e) **Agitation bar or agitator**

5.2.3 Reduction

The samples shall be reduced as follows depending on the types of soap.

- a) **Solid soap** Solid soaps shall be reduced using the quartering method shown below. The solid soap shall be equally divided into eight parts by cutting with a knife so that each of its sections passes through the centre of the sample and intersects at right angles to each other. Two parts of one-eighth content opposing to each other on the diagonal of sample shall be taken and combined into a quarter quantity. After slicing or cutting into dice, it shall be regarded as a sample (see Figure 2).

NOTE 1 In the case of dried soap, a “fretsaw” or the like should be used for cutting.

NOTE 2 Samples may be crushed with an electric mixer. In the case of soap containing much moisture which can adhere to the mixer vessel wall and make the uniform grinding difficult, means other than an electric mixer may be used.

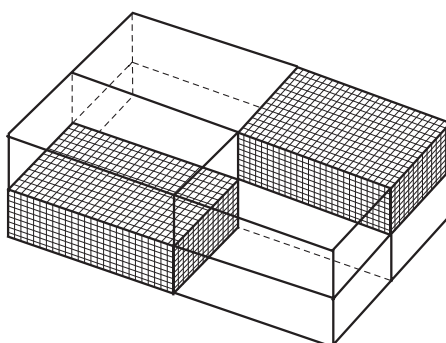


Figure 2 Quartering method

- b) **Powdery lump soap** Powdery lump soaps shall be reduced using either of the method shown below, which shall be repeated until a suitable amount of test sample is obtained.
- 1) **Riffle sampler method** The sample shall be put into a beaker or any suitable vessel after mixing, allowed to fall uniformly in the body of riffle sampler, and divided into two portions. One of the two portions shall be selected at random.

For soaps of grain state, flake state and chip state, this method shall be used after previously ground with an electric mixer.

- 2) **Coning and quartering method** The sample shall be put into a beaker or any suitable vessel after mixing, and conically piled on a hard clean flat surface. This operation shall be repeated one or two times by changing the position. After confirming the complete symmetry of cone, the top part of cone shall be flattened by rotating the spatula for division held horizontally. Then, the cone shall be cut by vertically pressing down the spatula for division so that it passes through the centre of the cone and equally divided into four sectors. The opposing two parts of quadrisection shall be discarded and the remaining two parts shall be mixed (see Figure 3).

For soaps of grain state, flake state and chip state, this method shall be used after previously ground with an electric mixer.

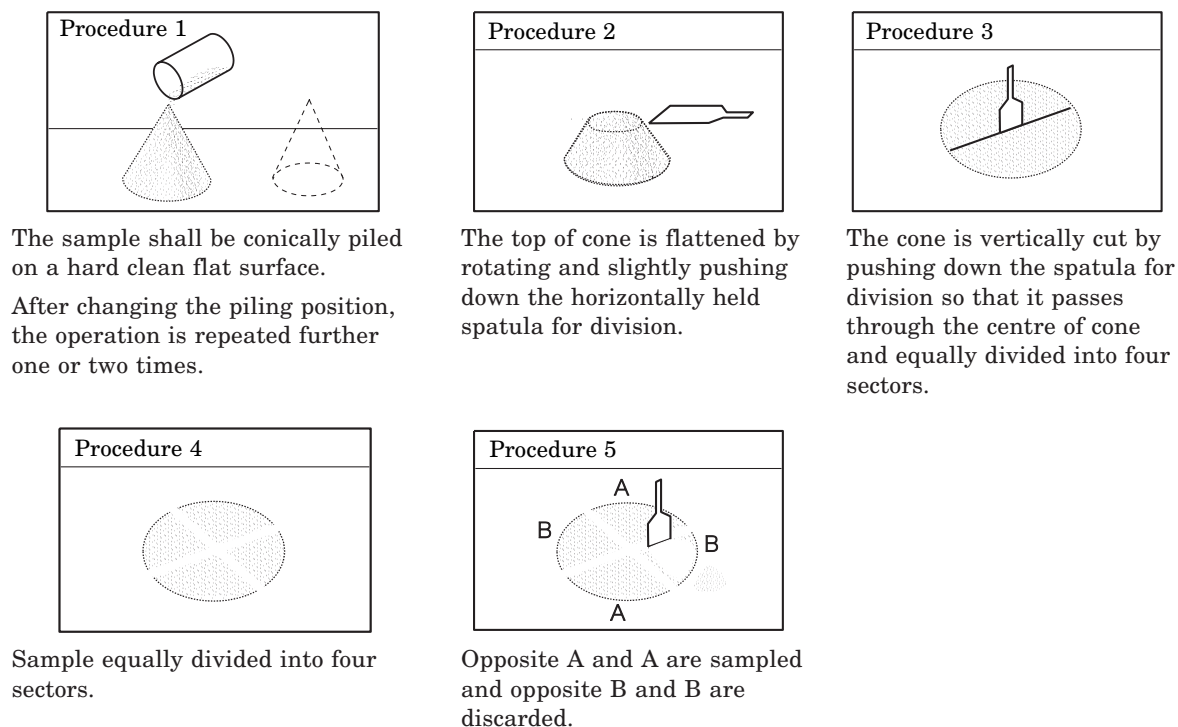


Figure 3 Coning and quartering method

- c) **Soft soap and liquid soap** Soft soaps and liquid soaps shall be reduced using the uniform mixing method shown below.

The total amount of sample is transferred into a beaker or the like, uniformly mixed with an agitation bar or agitator, and a suitable amount of the portion shall be regarded as a sample.

6 Test methods

6.1 Water content

6.1.1 General

The heating dry method and the distillation method are used.

6.1.2 Heating dry method

6.1.2.1 Summary

The loss when the sample is dried at 105°C is measured and made the water content thereof.

6.1.2.2 Appliances

The appliances shall be as follows.

- a) **Oven** That capable of being regulated at $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$.
- b) **Dish or weighing bottle** A flat bottom evaporating dish of 50 mm to 90 mm in outer diameter and 30 mm to 45 mm in depth, or flat weighing bottle.
- c) **Stirring rod** That made of glass.
- d) **Sand** Prepared by drying after cleaning, for example, sea sand.

6.1.2.3 Operation

The operation shall be as follows.

- a) Put a stirring rod into a dish or weighing bottle, and dry it in the oven regulated at $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Leave it to cool as it is in a desiccator.
- b) In the case of soaps liquefied at a drying temperature, operate it after previously putting the stirring rod and sand of about 10 g to the dish or weighing bottle. Dry them in the oven regulated at $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$, and leave them to cool as they are in a desiccator.
- c) Weigh out about 5 g of the sample in the dish or weighing bottle of **a)** or **b)** to the nearest 1 mg [m (g)], and put it in the oven regulated at $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Mix the sample and sand with a stirring rod in the case of using sand.
- d) After heating for 1 h, take it out from the oven and powder the sample using the stirring rod after cooling.
- e) Put the sample again in the oven regulated at $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$, and take it out therefrom after 1 h. Leave it to cool as it is in the desiccator, and measure its mass.
- f) Repeat the operations of **d)** and **e)** until the difference between two successive weighings becomes not more than 10 mg. The measured value at this time shall be regarded as the mass after drying [m_1 (g)].

6.1.2.4 Calculation

The water content shall be determined according to Formula (1).

$$C = \frac{m - m_1}{m} \times 100 \dots\dots\dots (1)$$

where, C : water content (mass fraction %)

m_1 : mass after drying (g)

m : mass of sample (g)

6.1.3 Distillation method

6.1.3.1 Summary

The sample is distilled together with xylene, and the content of water separated by distillation and the volatile matter soluble in water but insoluble in xylene are measured and recorded as the water content. This method is applicable to the sample containing not less than 5 % moisture in mass fraction, but not applicable to the sample containing water-soluble volatile compounds, for example, ethanol.

6.1.3.2 Reagents and apparatus

The reagents and apparatus shall be as follows.

- a) **Xylene** That specified in JIS K 8271.
- b) **Apparatus for water distillation** As specified in Figure 10 of JIS K 3362.

The inside of apparatus for water distillation shall be previously cleaned with the mixed solution of nitric acid and hydrogen peroxide water [e.g. mixed solution of nitric acid (6 mol/L) and hydrogen peroxide water (30 %) by the volume ratio 3:1]. Thereafter, it is washed with water and dried before use.

6.1.3.3 Operation

The operation shall be as follows.

- a) Weigh out about 10 g to 15 g of the sample, of which the amount of distilled water becomes 2 ml to 5 ml, into a distillation flask to the nearest 10 mg. Add 100 ml to 300 ml of xylene and boiling stone free from moisture, and assemble the apparatus for water distillation.
- b) Pour additional xylene from the upper end of cooler into the water measuring tube of distillation flask until it overflows, and stopper lightly the upper end of cooler with cotton.
- c) Heat the distillation flask and distil it at a speed of about 100 drops per minute. After the distillation of most of water, allow the distillation speed to be about 200 drops per minute.
- d) When the distillation of water is no longer observed in the water measuring tube, further continue to distil it for 30 min, and then stop heating. When water drops adhere to the inside of cooling tube and water measuring tube, remove and fall them with a spiral wire into the water measuring tube, and wash down with about 5 ml of xylene.
- e) Leave it as it is for 5 min or longer. When the layer of xylene has become transparent, read the amount of water in the water measuring tube to the nearest 0.05 ml at $20\text{ }^{\circ}\text{C} \pm 1.5\text{ }^{\circ}\text{C}$ and record it as the distilled amount of water [V (ml)].

6.1.3.4 Calculation

The water content shall be determined according to Formula (2).

$$C = \frac{1.0 \times V}{m} \times 100 \dots\dots\dots (2)$$

where, C : water content (mass fraction %)
 V : distilled amount of water (ml)
 m : mass of sample (g)
 1.0 : density of water

NOTE When the contents in the distillation flask foams by heating, adding the oleic acid or oleyl alcohol dehydrated by heating often brings about a good result.

6.2 Petroleum ether soluble matter

6.2.1 Summary

The content of matter soluble in petroleum ether is measured among the components except free fatty acid contained in the sample (total amount of unsaponifiable matter and unsaponifiable part of saponifiable matter), and made the petroleum ether soluble matter content. The extracted fatty acid content is titrated with potassium hydroxide and converted to oleic acid content, then it is subtracted from the petroleum ether soluble matter content.

6.2.2 Reagents

The reagents shall be as follows.

- a) **Neutral ethanol (95)** Ethanol (95) specified in JIS K 8102 is slowly boiled for 5 min to remove carbon dioxide, rapidly cooled to room temperature in running water, and neutralized by several drops of phenolphthalein solution (10 g/L) using 0.1 mol/L alcoholic potassium hydroxide solution until it keeps faint red colour for 30 s. It shall be prepared immediately before use.
- b) **Sodium hydrogen carbonate solution (10 g/L)** Prepared by dissolving 10 g of sodium hydrogen carbonate specified in JIS K 8622 in water to make 1 000 ml.
- c) **Petroleum ether** That specified in JIS K 8593, distilled at 30 °C to 60 °C.
- d) **0.1 mol/L alcoholic potassium hydroxide solution** The supernatant of the solution prepared in a way that 7 g of potassium hydroxide specified in JIS K 8574 is dissolved in 5 ml water, diluted to 1 000 ml by adding ethanol (95) specified in JIS K 8102 and left as it is for two to three days while keeping out carbon dioxide.

Standardization Titrate 25 ml of the solution above with several drops of phenolphthalein solution (10 g/L) until the red colour vanishes, using 0.1 mol/L hydrochloric acid solution prepared by the method specified in JIS K 8001.

- e) **Phenolphthalein solution (10 g/L)** Prepared as specified in JA.5 of JIS K 8001.
- f) **Sodium sulfate** That specified in JIS K 8987.

6.2.3 Appliances

The appliances shall be as follows.

- a) **Microburette** That of 2 ml in capacity with 0.05 ml graduation.
- b) **Oven** That specified in **6.1.2.2 a)**.

6.2.4 Operation

The operation shall be as follows.

- a) Weigh out about 5 g of the sample into a 200 ml beaker to the nearest 1 mg. Add 50 ml of neutral ethanol (95) and 50 ml of sodium hydrogen carbonate solution (10 g/L), dissolve the sample by heating at 70 °C or under, and leave it to cool as it is.
- b) When there is some insoluble matter, filter and transfer the solution into a 500 ml Separating funnel A. Wash the beaker of **a)** and the insoluble matter on the filter paper with a small amount of a mixture of equal volumes of neutral ethanol (95) and sodium hydrogen carbonate solution (10 g/L), then combine the washings to Separating funnel A.
- c) Add 50 ml of petroleum ether to Separating funnel A, and mix it by shaking vigorously for 1 min for extraction. Leave it as it is until it is apparently separated into two layers.
- d) Transfer the water layer into Separating funnel A to a 500 ml Separating funnel B, add 50 ml of petroleum ether, shake and mix it for extraction. Thereafter, leave it as it is until it is apparently separated into two layers.
- e) Transfer the water layer in Separating funnel B to a 500 ml Separating funnel C, add 50 ml of petroleum ether, shake and mix it for extraction. Thereafter, leave it as it is until it is apparently separated into two layers.
- f) Transfer the water layer in Separating funnel C to a 300 ml Erlenmeyer flask.
- g) Transfer the petroleum ether extract solution in Separating funnel B and Separating funnel C to Separating funnel A. Wash Separating funnel B and Separating funnel C with a small amount of petroleum ether, and combine the washings to Separating funnel A.
- h) Each time add 50 ml of a mixture of equal volumes of neutral ethanol (95) and water to Separating funnel A, and wash it until the washings no longer turn faintly red with several drops of phenolphthalein solution (10 g/L).
- i) Remove the washings from Separating funnel A, and dehydrate the petroleum ether extract solution by adding sodium sulfate thereto. Using a dry filter paper, filter it into a 300 ml Erlenmeyer flask having known mass preliminarily dried to constant weight.
- j) Wash Separating funnel A and the filter paper two or three times with a small amount of petroleum ether, and add the washings to the Erlenmeyer flask of **i)**.
- k) Remove traces of solvent from most of petroleum ether in the Erlenmeyer flask of **j)** on water bath with slowly flowing dry air or nitrogen gas.

- l) Heat the Erlenmeyer flask of **k**) in the oven regulated at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for 5 min, leave it to cool as it is in a desiccator and measure the mass.
- m) Repeat the operation of **l**) until the difference between two successive weighings becomes not more than 2 mg. The mass at this time shall be regarded as the amount of petroleum ether extract [m_1 (g)].
- n) Dissolve the petroleum ether extract obtained in **m**) by adding several ml of neutral ethanol (95) hereto, and add several drops of phenolphthalein solution (10 g/L). Titrate it by 0.1 mol/L alcoholic potassium hydroxide solution using a microburette, and take the point when faint red colour is kept for 30 s as the end point. Record the amount of 0.1 mol/L alcoholic potassium hydroxide solution required for titration as [V (ml)].

6.2.5 Calculation

The petroleum ether soluble matter content shall be determined according to Formula (3).

$$C = \frac{m_1 - \frac{V \times M}{1000} \times \frac{f}{10}}{m} \times 100 \dots\dots\dots (3)$$

where, C : petroleum ether soluble matter content (mass fraction %)

m_1 : amount of petroleum ether extract (g)

V : amount of 0.1 mol/L alcoholic potassium hydroxide solution required for titration (ml)

f : factor of 0.1 mol/L alcoholic potassium hydroxide solution

M : average molecular weight of fatty acid

m : mass of sample (g)

NOTE For the average molecular weight of fatty acid, the numeric value of 282 of oleic acid is generally used.

6.3 Total alkali and total fatty matter

6.3.1 Summary

This method is for the simultaneous determination of total alkali and total fatty matter contained in the sample, and is not applicable to the soaps in which the surface active agents except the salts of fatty acid are contained.

6.3.2 Total alkali

6.3.2.1 Summary

Total amount of alkali matter in the soap titrated with inorganic acid is determined. This includes the alkali matter bonded as soap, the hydroxides or carbonates of alkaline metal, and the alkali content of some silicates incidentally titrated under this test conditions.

The sample is decomposed with a definite amount of inorganic acid standard solution, and the liberated fatty matter is separated by extraction with petroleum ether. The excessive acid contained in the water layer is titrated with sodium hydroxide solution, and the total alkali content is determined.

6.3.2.2 Reagents

The reagents shall be as follows.

- a) **Petroleum ether** That specified in **6.2.2 c)**.
- b) **0.5 mol/L sulfuric acid** That specified in **JA.6** of **JIS K 8001**.
- c) **1 mol/L hydrochloric acid** That specified in **JA.6** of **JIS K 8001**.
- d) **1 mol/L sodium hydroxide solution** That specified in **JA.6** of **JIS K 8001**.
- e) **Methyl orange solution** That specified in **JA.5** of **JIS K 8001**.

6.3.2.3 Operation

The operation shall be as follows.

- a) Weigh out about 5 g of the sample into a 200 ml beaker to the nearest 1 mg. Add 100 ml of water thereto and dissolve it by heating.
- b) Transfer it into a 500 ml Separating funnel A. Wash the beaker of **a)** with a small amount of water, and combine the washings to Separating funnel A.
- c) Using the methyl orange solution as an indicator, add 0.5 mol/L sulfuric acid or 1 mol/L hydrochloric acid to Separating funnel A while mixing it by shaking vigorously until it becomes acid.
- d) Add 5 ml of 0.5 mol/L sulfuric acid or 1 mol/L hydrochloric acid, and record the amount of 0.5 mol/L sulfuric acid or 1 mol/L hydrochloric acid required for decomposition as $[V_1 \text{ (ml)}]$.
- e) After leaving it to cool as it is, add 100 ml of petroleum ether, and mix it by shaking vigorously for 1 min for extraction. Leave it as it is until it is apparently separated into two layers.
- f) Transfer the water layer in Separating funnel A to a 500 ml Separating funnel B, add 50 ml of petroleum ether, and shake to mix for extraction. Leave it as it is until it is apparently separated into two layers.
- g) Transfer the water layer in Separating funnel B to a 500 ml Separating funnel C, add 30 ml of petroleum ether, and shake to mix for extraction. Leave it as it is until it is apparently separated into two layers.
- h) Transfer the water layer in Separating funnel C to a 300 ml Erlenmeyer flask.
- i) Transfer the petroleum ether extract solutions in Separating funnel B and Separating funnel C to Separating funnel D. Wash respective separating funnels with a small amount of petroleum ether, and combine the washings to Separating funnel A.

- j) Each time use 25 ml of water for the washing of petroleum ether extract solution. Wash Separating funnel A and Separating funnel B, then transfer the washings to 500 ml Separating funnel A and wash. Continue the washing until the washings become neutral, using the methyl orange solution.
- k) Use 25 ml of water to wash the petroleum ether extract solution in Separating funnel B and Separating funnel C, respectively. Transfer both washings into Separating funnel A, and wash the petroleum ether extract solution. Continue to wash by changing water until the washings become neutral using the methyl orange solution.
- l) Collect all washings into the 300 ml Erlenmeyer flask containing water layer of **h**), titrate it with 1 mol/L sodium hydroxide solution using methyl orange solution as an indicator. Record the amount of 1 mol/L sodium hydroxide solution required for titration as $[V_2 \text{ (ml)}]$.

6.3.2.4 Calculation

The total alkali content shall be determined according to Formula (4) or Formula (5).

a) In the case of sodium soap

$$C = 0.040 \times (V_1 f_1 - V_2 f_2) \times \frac{100}{m} \dots\dots\dots (4)$$

b) In the case of potassium soap

$$C = 0.056 \times (V_1 f_1 - V_2 f_2) \times \frac{100}{m} \dots\dots\dots (5)$$

where, C : total alkali content (mass fraction %)
 V_1 : added amount of 0.5 mol/L sulfuric acid or 1 mol/L hydrochloric acid (ml)
 V_2 : amount of 1 mol/L sodium hydroxide solution required for titration (ml)
 f_1 : factor of 0.5 mol/L sulfuric acid or 1 mol/L hydrochloric acid
 f_2 : factor of 1 mol/L sodium hydroxide solution
 m : mass of sample (g)

6.3.3 Total fatty matter

6.3.3.1 Summary

Total amount of water-insoluble fatty matter obtained by decomposing the soap using inorganic acid is determined. This includes the unsaponifiable matters, glycerides and any rosin acids except the salts of fatty acid contained in soaps.

After the evaporation of the petroleum ether from the petroleum ether extract solution of **6.3.2**, the residue is dissolved in ethanol, and the fatty acid is neutralized with a potassium hydroxide solution. The ethanol is evaporated, and total fatty matter content is determined by measuring the mass of generated potassium soap.

6.3.3.2 Reagents

The reagents shall be as follows.

- a) **Neutral ethanol (99.5)** Ethanol (99.5) specified in JIS K 8101, which is slowly boiled for 5 min to remove carbon dioxide, rapidly cooled to room temperature in running water, and neutralized by several drops of phenolphthalein solution (10 g/L) using 0.1 mol/L alcoholic potassium hydroxide solution until it keeps faint red colour for 30 s. It shall be prepared immediately before use.
- b) **0.5 mol/L alcoholic potassium hydroxide solution** The supernatant of the solution prepared in a way that 33 g of potassium hydroxide specified in JIS K 8574 is dissolved in 25 ml water, diluted to 1 000 ml by adding ethanol (95) specified in JIS K 8102 and left as it is for two to three days while keeping out carbon dioxide.

Standardization Titrate 25 ml of above solution with several drops of phenolphthalein solution (10 g/L) until the red colour vanishes, using 0.5 mol/L hydrochloric acid solution specified in JIS K 8001.

- c) **Phenolphthalein solution (10 g/L)** That specified in 6.2.2 e).
- d) **Acetone** That specified in JIS K 8034.
- e) **Sodium sulfate** That specified in 6.2.2 f).
- f) **Petroleum ether** That specified in 6.2.2 c).

6.3.3.3 Appliance

The appliance shall be as follows.

- a) **Oven** That specified in 6.1.2.2 a).

6.3.3.4 Operation

The operation shall be as follows.

- a) Dehydrate the petroleum ether extract solution obtained in 6.3.2 by adding sodium sulfate thereto, and using a dry filter paper, filter it into a 300 ml Erlenmeyer flask having known mass preliminarily dried to constant weight.
- b) Wash a 500 ml separating funnel and filter paper two or three times with a small amount of petroleum ether, and add the washings to the 300 ml Erlenmeyer flask of a).
- c) Evaporate the petroleum ether on water bath, and allow the remaining amount to be about 10 ml. Thereafter, leave it to cool as it is, and dissolve it by adding about 20 ml of neutral ethanol (99.5).
- d) Add several drops of phenolphthalein solution (10 g/L), titrate it with 0.5 mol/L alcoholic potassium hydroxide solution, and take the point when it keeps faint red colour for 30 s as the end point. Record the amount of 0.5 mol/L alcoholic potassium hydroxide solution at this time as [V (ml)]
- e) Then, evaporate the neutral ethanol (99.5) on water bath. When the evaporation becomes near its end, spread potassium soap thinly to the wall and bottom part of Erlenmeyer flask by rotating the 300 ml Erlenmeyer flask. After most of the neutral ethanol (99.5) is evaporated, leave it to cool as it is.