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Test methods for soaps

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Foreword

This translation has been made based on the original Japanese Industrial Standard 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**: 1997 is replaced with this Standard.

This revision has been made based on **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* and **ISO 8212**: 1986 *Soaps and detergents—Techniques of sampling during manufacture* for the purposes of making it easier to compare this Standard with International Standard; to prepare Japanese Industrial Standard conforming with International Standard; and to propose a draft of an International Standard which is based on Japanese Industrial Standard.

Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have technical properties. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have the said technical properties.

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Test methods for soaps

Introduction This Japanese Industrial Standard has been prepared based on the first edition of **ISO 456** *Surface active agents—Analysis of soaps—Determination of free caustic alkali* published in 1973, the second edition of **ISO 457** *Soaps—Determination of chloride content—Titrimetric method* published in 1983, the first edition of **ISO 672** *Soaps—Determination of moisture and volatile matter content—Oven method* published in 1978, the second edition of **ISO 673** *Soaps—Determination of content of ethanol-insoluble matter* published in 1981, the first edition of **ISO 684** *Analysis of soaps—Determination of total free alkali* published in 1974, the first edition of **ISO 685** *Analysis of soaps—Determination of total alkali content and total fatty matter content* published in 1975, the first edition of **ISO 1066** *Analysis of soaps—Determination of glycerol content—Titrimetric method* published in 1975, the first edition of **ISO 1067** *Analysis of soaps—Determination of unsaponifiable, unsaponified and unsaponified saponifiable matter* published in 1974, the second edition of **ISO 2272** *Surface active agents—Soaps—Determination of low contents of free glycerol by molecular absorption spectrometry* published in 1989, the second edition of **ISO 4318** *Surface active agents and soaps—Determination of water content—Azeotropic distillation method* published in 1989, the first edition of **ISO 4323** *Soaps—Determination of chlorides content—Potentiometric method* published in 1977, the second edition of **ISO 4325** *Soaps and detergents—Determination of chelating agent content—Titrimetric method* published in 1990 and the first edition of **ISO 8212** *Soaps and detergents—Techniques of sampling during manufacture* published in 1986 with some modifications of the technical contents.

The portions given continuous sidelines and dotted underlines are the matters in which the contents of the original International Standard have been modified. A list of modifications with explanations is given in Annex (informative).

1 Scope This Standard specifies the test methods for soaps (those containing surface active agent or synthetic detergent are excluded).

NOTE : The International Standards corresponding to this Standard are as follows.

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**.

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

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

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

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

- ISO 684:1974 *Analysis of soaps—Determination of total free alkali (MOD)*
- ISO 685:1975 *Analysis of soaps—Determination of total alkali content and total fatty matter content (MOD)*
- ISO 1066:1975 *Analysis of soaps—Determination of glycerol content—Titrimetric method (MOD)*
- ISO 1067:1974 *Analysis of soaps—Determination of unsaponifiable, unsaponified and unsaponified saponifiable matter (MOD)*
- ISO 2272:1989 *Surface active agents—Soaps—Determination of low contents of free glycerol by molecular absorption spectrometry (MOD)*
- ISO 4318:1989 *Surface active agents and soaps—Determination of water content—Azeotropic distillation method (MOD)*
- ISO 4323:1977 *Soaps—Determination of chlorides content—Potentiometric method (MOD)*
- ISO 4325:1990 *Soaps and detergents—Determination of chelating agent content—Titrimetric method (MOD)*
- ISO 8212:1986 *Soaps and detergents—Techniques of sampling during manufacture (MOD)*

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 3362 Testing methods for synthetic detergent

JIS K 8001 General rule for test methods of reagents

JIS K 8005 Reference materials for volumetric analysis

JIS K 8034 Acetone

JIS K 8101 Ethanol (99.5)

JIS K 8102 Ethanol (95)

JIS K 8103 Diethyl ether (Reagent)

JIS K 8105 Ethylene glycol

JIS K 8136 Tin (II) chloride dihydrate

JIS K 8155 Barium chloride dihydrate

JIS K 8180 Hydrochloric acid

JIS K 8256 Sodium periodate

- JIS K 8271 *Xylene (Reagent)*
JIS K 8295 *Glycerol (Reagent)*
JIS K 8316 *Disodium chromotropate dihydrate*
JIS K 8541 *Nitric acid*
JIS K 8574 *Potassium hydroxide*
JIS K 8576 *Sodium hydroxide*
JIS K 8577 *Barium hydroxide octahydrate (Reagent)*
JIS K 8593 *Petroleum ether (Reagent)*
JIS K 8622 *Sodium hydrogen carbonate (Reagent)*
JIS K 8643 *Thymol blue*
JIS K 8799 *Phenolphthalein*
JIS K 8842 *Bromothymol blue*
JIS K 8844 *Bromophenol blue*
JIS K 8982 *Ammonium iron (III) sulfate 12-water*
JIS K 8983 *Copper (II) sulfate pentahydrate*
JIS K 8987 *Sodium sulfate*
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 : **ISO 383**:1976 *Laboratory glassware—Interchangeable conical ground joints* is equivalent to the said standard.
JIS Z 8802 *Methods for determination of pH of aqueous solutions*

3 Term and definition For the purpose of this Standard, the following term and definition shall apply.

- a) **soap** product mainly composed of sodium salt or potassium salt of high grade fatty acid

4 Test items

4.1 Chemical test The chemical test shall be as follows:

- a) **Water content**
- b) **Petroleum ether soluble matter**
- c) **Total alkali and total fatty matter**
- d) **Pure soapy matter**
- e) **Free alkali**
- f) **Total free alkali**

- g) **Ethanol-insoluble matter**
- h) **Water-insoluble matter**
- i) **Chlorides**
- j) **Glycerol**
- k) **Ethylenediaminetetraacetate (edetic acid)**

4.2 pH value

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

6 Preparation of test sample

6.1 Subject The subject of test sample shall be 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.

6.2 Appliances The appliances shall be as follows:

- a) **Knife**
- b) **Electric mixer**
- c) **Riffle sampler** The riffle sampler is in accordance with No. 6 of table 4 specified in JIS M 8100.
- d) **Spatula for division** The spatula for division is a flat plate made of stainless steel or synthetic resin used for division of conical quartering (see figure 1).

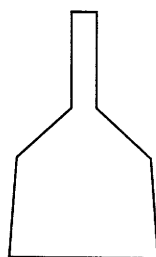


Figure 1 Spatula for division

- e) **Agitation bar or agitator**

6.3 Reduction The reduction shall be repeated using either one of the following methods until a suitable amount of test sample can be obtained.

- a) **Quartering** The quartering is the method applied to solid soap. 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 intersect 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 crushed with an electric mixer to make the test sample (see figure 2).

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

- 2 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.

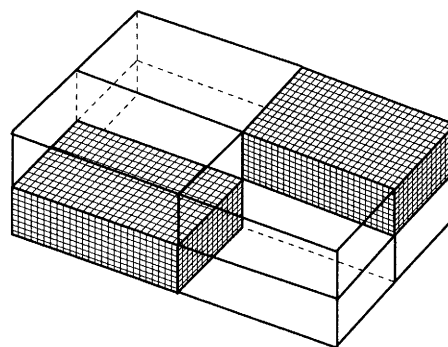
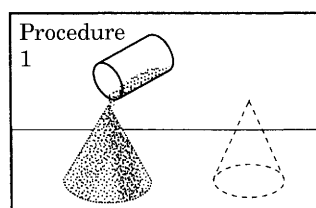


Figure 2 Quartering

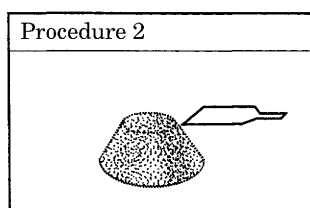
- b) **Method by riffle sampler** The method by riffle sampler applies to powdery lump soaps. The sample is put into a beaker or any suitable vessel after mixing, allowed to fall uniformly in the body of riffle sampler and divided into two portions. One of the two portions shall be selected at random. For soaps of grain state, flake state and chip state, this method is used after previously grinding with an electric mixer.
- c) **Conical quartering** The conical quartering is the method applied to powdery lump soaps. 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 is repeated one or two times. 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 4 sectors. The opposing two parts of quadrisection shall be discarded and the remaining two parts thereof shall be mixed (see figure 3).

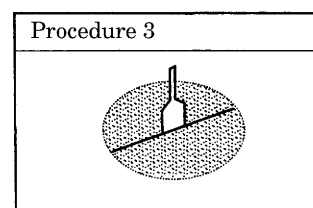
For soaps of grain state, flake state and chip state, this method shall be used after previously grinding with the electric mixer.



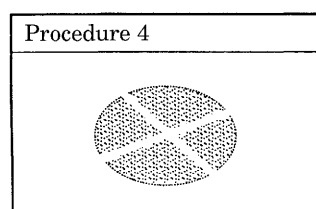
The sample is conically piled on a hard clean flat surface. After changing the piling position, the operation is repeated further 1 or 2 times.



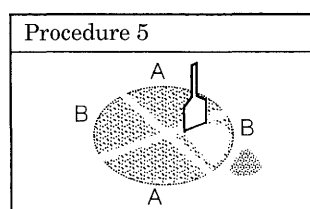
The top of cone is flattened by rotating and slightly pushing down the horizontally held spatula for division.



The cone is vertically cut by pushing down the spatula for division so that it passes through the centre of cone and equally divided into 4 sectors.



Sample equally divided into 4 sectors.



Opposite A and A are sampled and opposite B and B are discarded.

Figure 3 Conical quartering

- d) **Method by uniform mixing** The method by uniform mixing is the method applied to soft soap and liquid soap. The total amount of sample is transferred into a beaker or the like, uniformly mixed with an agitating bar or agitator and a portion thereof shall be made the test sample.

7 Chemical test

7.1 Water content

7.1.1 Classification of test methods There are the following two types of test methods.

- Heating dry method**
- Distillation method**

7.1.2 Heating dry method The heating dry method shall be as follows:

- Summary** The loss when the sample is dried at 105 °C is measured and made the water content thereof.
- Appliances** The appliances shall be as follows:
 - Oven** The oven capable of being regulated at 105 °C ± 2 °C.
 - Dish or weighing bottle** A flat bottom evaporating dish 50 mm to 90 mm in outer diameter and 30 mm to 45 mm in depth or flat weighing bottle.
 - Stirring rod** A stirring rod made of glass.
 - Sand** Sand prepared by drying after cleaning, for example, sea sand.

c) **Operation** The operation shall be as follows:

- 1) Put a stirring rod into a dish or weighing bottle and dry it with the oven regulated at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$. Leave it to cool as it is in a desiccator and measure the mass.
- 2) In the case of soaps to be liquefied at a drying temperature, operate it after previously adding about 10 g of sand to the dish or weighing bottle.
- 3) Weigh out about 5 g of the sample into the dish or weighing bottle to the nearest 1 mg and mix it with a stirring rod in the case of using sand. Thereafter, put it into the oven regulated at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.
- 4) After heating for 1 h, take it out from the oven and powder the sample using the stirring rod after cooling.
- 5) Put it into the oven again and take it out therefrom after 1 h. Leave it to cool as it is in the desiccator and measure its mass.
- 6) Repeat the operations of 4) and 5) until the difference between the weighings of successive 2 times becomes not more than 10 mg.

d) **Calculation** The water content shall be calculated from the formula of (1).

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

where, C : water content (mass fraction %)

m_1 : loss (g)

m : mass of sample (g)

7.1.3 Distillation method The distillation method shall be as follows:

a) **Summary** The sample is distilled together with xylene, and the water separated by distillation and the volatile matter soluble in water but insoluble in xylene are measured and recorded as the water content. Though this method is applicable to the sample containing not less than 5 % of water content, it is not applicable to the sample containing water soluble volatile matter, for example, ethanol.

b) **Reagent** The reagent shall be the xylene specified in JIS K 8271.

c) **Apparatus** The apparatus shall be the apparatus for distillation water content specified in figure 10 of **7.21.2** of JIS K 3362.

d) **Operation** The operation shall be as follows:

- 1) Weigh out about 10 g to 15 g of the sample, of which the amount for distilled water content 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.
- 2) Let xylene flow into a water measuring tube until it overflows in the distillation flask from the upper end of cooler and stopper lightly the upper end of cooler with cotton.

- 3) Heat the flask and distill it at a speed of about 100 drops per minute. After distillation of most of water, allow the distillation speed to be about 200 drops per minute.
- 4) When distillation of water is no longer observed in the water measuring tube, further continue to distill it for 30 min, and then stop heating. When water drops adhere to the inside of cooling tube and water measuring tube, remove it with a spiral wire and wash it down with about 5 ml of xylene.
- 5) Leave it as it is for 5 min or longer and when the layer of xylene has become transparent, read the amount of water at 20 °C and record it as the distilled amount of water.

e) **Calculation** The water content shall be calculated from the 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

- NOTES 1 The inside of apparatus for distillation of water content is previously cleaned with the mixed solution of nitric acid and hydrogen peroxide water (for example: 3 parts of 6 mol/L nitric acid + 1 part of 30 % H_2O_2). Thereafter, it is washed with water and dried before use.
- 2 Although the error of one scale interval shall be not more than 0.05 ml, calibration should be carried out for a measuring apparatus on the market.

Information : 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.

7.2 Petroleum ether soluble matter

7.2.1 Summary This method aims at the extraction and determination of the matter soluble in petroleum ether having components except free fatty acid contained in the sample (total amount of unsaponifiable matter and unsaponifiable content of saponifiable matter). The extracted fatty acid is titrated with potassium hydroxide, converted to oleic acid and subtracted.

7.2.2 Reagents The reagents shall be as follows:

- a) **Neutral ethanol (95)** Ethanol (95) specified in **JIS K 8102** is slowly boiled for 5 min and carbon dioxide is removed. The sample is rapidly cooled to room temperature in running water, added with several drops of phenolphthalein solution (10 g/L) and neutralized using 0.1 mol/L alcoholic potassium hydroxide solution until it keeps faint red colour for 30 s. It shall be prepared just before use.

- b) **Sodium hydrogencarbonate solution** (10 g/L) The solution prepared by dissolving 10 g of sodium hydrogencarbonate specified in JIS K 8622 in water to make 1 000 ml.
- c) **Petroleum ether** The petroleum ether specified in JIS K 8593, distilled at 30 °C to 60 °C.
- d) **0.1 mol/L alcoholic potassium hydroxide solution** The supernatant wherein 7 g of potassium hydroxide specified in JIS K 8574 is dissolved with 5 ml water, diluted to 1 000 ml by adding ethanol (95) specified in JIS K 8102 and left as it is for 2 days to 3 days while keeping out carbon dioxide.

Standardization 25 ml of this reagent several drops of phenolphthalein solution (10 g/L) and titrate using 0.1 mol/L hydrochloric acid solution specified in JIS K 8001 until the red colour vanishes.

- e) **Phenolphthalein solution** (10 g/L) The solution in accordance with 4.4 of JIS K 8001.
- f) **Sodium sulphate** That specified in JIS K 8987.

7.2.3 Appliances The appliances shall be as follows:

- a) **Microburette** The microburette of 2 ml in capacity with 0.05 ml of graduation.
- b) **Oven** The oven in accordance with 7.1.2 b) 1).

7.2.4 Operation The operation shall be as follows:

- a) Weigh out about 5 g of the sample into a beaker of 200 ml capacity to the nearest 1 mg. Add 50 ml of neutral ethanol (95) and 50 ml of sodium hydrogencarbonate solution (10 g/L) and dissolve it by heating at 70 °C max. and leave it to cool as it is.
- b) When there is some insoluble matter, filter it, and transfer it to a separating funnel. Wash the insoluble matter on the beaker and the filter paper with a small amount of a mixture of equal volumes of the neutral ethanol (95) and the sodium hydrogencarbonate solution (10 g/L) and combine the washing to the separating funnel.
- c) Then, add 50 ml of petroleum ether to the separating funnel, mix it by violently shaking it for 1 min and extract it. Thereafter, leave it as it is until it is apparently separated into 2 layers.
- d) Transfer the layer of water to the second separating funnel of 500 ml capacity, add 50 ml of petroleum ether and mix it by shaking in the same way. Thereafter leave it as it is.
- e) Further, transfer the layer of water to the third separating funnel of 500 ml capacity, and add 50 ml of petroleum ether. Mix it again by shaking in the same way and extract it.

- f) Transfer the petroleum ether extracted solution in the second and third separating funnels to the first separating funnel. Wash respective separating funnels with a small amount of petroleum ether and combine the washings to the first funnel.
- g) Each time add 50 ml of the mixture of equal volumes of neutral ethanol (95) and water to the first separating funnel and wash it until the washings no longer turn faintly red with several drops of phenolphthalein solution (10 g/L).
- h) Extract the washings and dehydrate the petroleum ether extracted solution by adding sodium sulphate thereto. Filter it into the 300-ml Erlenmeyer flask having known mass preliminarily dried to constant weight using a dry filter paper.
- i) Wash the separating funnel and the filter paper with a small amount of petroleum ether two or three times and add the washings to the Erlenmeyer flask.
- j) Remove trace of solvent from most of petroleum ether on water bath with slowly flowing dry air or nitrogen gas.
- k) Heat the Erlenmeyer flask with the oven regulated at $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for 5 min, leave it to cool as it is in a desiccator and measure the mass.
- l) Repeat the operation of **k**) until the difference between the weighings of successive two times becomes not more than 2 mg.
- m) Then, dissolve the residue 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.

7.2.5 Calculation The petroleum ether soluble matter shall be calculated from the 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 (mass fraction %)
 m_1 : extracted amount of petroleum ether (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.

7.3 Total alkali and total fatty matter

7.3.1 Summary This method is for the simultaneous determination of total alkali and total fatty matter of soap and is not applicable to the case where the surface active agents except the salt of fatty acid are contained.

NOTES 1 Total alkali It is the total amount of alkali content in the soap titrated with inorganic acid. The alkali content bonded as soap, the hydroxide or carbonate of alkaline metal and the alkali content of silicates of some kind or the like titrated under this test conditions are contained therein.

2 Total fatty matter The fatty matter insoluble in water obtained by dissolving soaps using inorganic acid under specified conditions. Unsaponifiable matter, fats and oils, resin acid, etc. contained in soaps except the salt of fatty acid are contained therein.

7.3.2 Total alkali The total alkali shall be as follows:

- a) **Summary** The sample is dissolved 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 layer of water is titrated with sodium hydroxide solution and the total alkali is determined. This method is not applicable to the coloured soap of which the colour obstructs the judgement of the end point of methyl orange.
- b) **Reagents** The reagents shall be as follows:
 - 1) **Petroleum ether** The petroleum ether in accordance with **7.2.2 c)**.
 - 2) **0.5 mol/L sulphuric acid** The sulphuric acid specified in **4.5** of **JIS K 8001**.
 - 3) **1 mol/L hydrochloric acid** The hydrochloric acid specified in **4.5** of **JIS K 8001**.
 - 4) **1 mol/L sodium hydroxide solution** The sodium hydroxide solution specified in **4.5** of **JIS K 8001**.
 - 5) **Methyl orange solution** Methyl orange solution specified in **4.4** of **JIS K 8001**.
- c) **Operation** The operation shall be as follows:
 - 1) Weigh out about 5 g of the sample into a 200-ml beaker to the nearest 1 mg. Add 100 ml of water to it and dissolve it by heating.
 - 2) Transfer it to a 500-ml separating funnel. Wash the beaker with a small amount of water and combine the washing to the separating funnel.
 - 3) Using methyl orange solution as an indicator, add 0.5 mol/L sulphuric acid or 1 mol/L hydrochloric acid with a burette while mixing it by shaking vigorously until it becomes acid.
 - 4) Further, add 5 ml in excess and accurately measure the amount of acid required for decomposition.
 - 5) After leaving it to cool as it is, add 100 ml of petroleum ether, and mix it by shaking it vigorously for 1 min to extract it. Leave it as it is until it is apparently separated into two layers.

- 6) Transfer the layer of water to the second separating funnel of 500 ml capacity, add 50 ml of petroleum ether and mix it by shaking in the same way. Thereafter, leave it as it is.
 - 7) Transfer the layer of water to the third separating funnel of 500 ml capacity, and add 30 ml of petroleum ether. Mix it again by shaking in the same way and extract it.
 - 8) Extract the layer of water into a 300-ml Erlenmeyer flask.
 - 9) After transferring the petroleum ether extracted solution in the second and the third separating funnels to the first separating funnel, wash respective separating funnels with a small amount of petroleum ether and combine the washings to the first separating funnel.
 - 10) Each time use 25 ml of water for the washing of petroleum ether extracted solution, at first wash the second and third separating funnels, successively transfer it to the first separating funnel and carry out the operation. Continue the washing until the washings become neutral by methyl orange solution.
 - 11) Collect all washings into the Erlenmeyer flask with the layer of water and titrate it with 1 mol/L sodium hydroxide solution using methyl orange solution as an indicator.
- d) **Calculation** Total alkali shall be calculated from the formula (4) or (5).

1) **In the case of sodium soap**

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

2) **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 (mass fraction %)
 V_1 : added amount of 0.5 mol/L sulphuric 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 sulphuric acid or 1 mol/L hydrochloric acid
 f_2 : factor of 1 mol/L sodium hydroxide solution
 m : mass of sample (g)

7.3.3 Total fatty matter The total fatty matter shall be as follows:

- a) **Summary** After distilling petroleum ether from the extracted solution obtained for total alkali, the residue is dissolved with ethanol and the fatty acid is neutralized with potassium hydroxide solution. The ethanol is evaporated, and total fatty matter is determined by measuring the mass of generated potassium soap.

b) **Reagents** The reagents shall be as follows:

- 1) **Neutral ethanol (99.5)** Ethanol (99.5) specified in **JIS K 8101** is slowly boiled for 5 min and carbon dioxide is removed. The sample is rapidly cooled to room temperature with running water, added with several drops of phenolphthalein solution (10 g/L) and neutralized using 0.1 mol/L alcoholic potassium hydroxide solution until it keeps faint red colour for 30 s. It shall be prepared just before use.
- 2) **0.5 mol/L alcoholic potassium hydroxide solution** The supernatant liquid prepared so that 33 g of the potassium hydroxide specified in **JIS K 8574** is dissolved in 25 ml of water, made to be 1 000 ml by adding the ethanol (95) specified in **JIS K 8102** and left as it is for 2 days to 3 days while keeping out carbon dioxide.

Standardization Several drops of phenol phthalein solution (10 g/L) are added in 25 ml of alcoholic potassium hydroxide solution adjusted and titration is carried out by using 0.5 mol/L hydrochloric acid solution specified in **JIS K 8001** until crimson colour vanishes.

- 3) **Phenolphthalein solution (10 g/L)** The solution in accordance with 7.2.2 e).
- 4) **Acetone** The acetone specified in **JIS K 8034**.
- 5) **Sodium sulphate** The sodium sulphate in accordance with 7.2.2 f).

c) **Appliance** The appliance shall be the oven in accordance with 7.1.2 b) 1).

d) **Operation** The operation shall be as follows:

- 1) Dehydrate the petroleum ether extracted solution obtained in 7.3.2 by adding sodium sulphate thereto and filter it into the 300-ml Erlenmeyer flask having known mass preliminarily dried to constant weight using a dry filter paper.
- 2) Wash the separating funnel and the filter paper 2 or 3 times with a small amount of petroleum ether, and add the washings to the Erlenmeyer flask.
- 3) Distill the petroleum ether on water bath and allow the remaining amount to be about 10 ml. Thereafter, leave it to cool as it is, dissolve it by adding about 20 ml of neutral ethanol (99.5).
- 4) 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 required for titration.
- 5) Then, evaporate the ethanol 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 Erlenmeyer flask. After most of ethanol is evaporated, leave it to cool as it is.
- 6) Then, add 10 ml to 15 ml of acetone to remove moisture, evaporate it on water bath and dry potassium soap with slowly flowing dry air or nitrogen gas.