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Surface active agents — Technical sodium alkylarylsulphonates (excluding benzene derivatives) — Methods of analysis

*Agents de surface — Alkylarylsulfonates de sodium techniques (excepté ceux dérivant du benzène) —
Méthode d'analyse*

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 1104 was developed by Technical Committee ISO/TC 91, *Surface active agents*, and was circulated to the member bodies in November 1975.

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It has been approved by the member bodies of the following countries :

| | | |
|---------------------|-------------|-----------------------|
| Australia | India | South Africa, Rep. of |
| Austria | Iran | Spain |
| Belgium | Italy | Switzerland |
| Brazil | Japan | Turkey |
| Canada | Mexico | United Kingdom |
| Egypt, Arab Rep. of | Netherlands | U.S.A. |
| France | New Zealand | U.S.S.R. |
| Germany | Portugal | |
| Hungary | Romania | |

No member body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 1104-1969, of which it constitutes a technical revision.

Surface active agents – Technical sodium alkylarylsulphonates (excluding benzene derivatives) – Methods of analysis

0 INTRODUCTION

Sodium alkylarylsulphonates have the general formula



and are used as surface active agents in a vast range of processes. They are sodium salts of alkylarylsulphonic acids in which the saturated aliphatic radical **R** may have a mean chain length of about 12 carbon atoms; *n* may be equal to 1, 2 or 3 and **Ar** is an aromatic derivative of either toluene or naphthalene.

1 SCOPE

This International Standard specifies methods of analysis of technical sodium alkylarylsulphonates. It covers the following determinations :

- Measurement of pH.
- Determination of water content.
- Determination of free alkalinity or free acidity.
- Determination of matter extractable by light petroleum.
- Determination of sodium alkylarylsulphonates content.
- Determination of sodium sulphite content.
- Determination of sodium sulphate content.
- Determination of sodium chloride content.

It also sets out, in annexes

- A : a general scheme of analysis;
- B : a rapid method for the determination of sodium alkylarylsulphonates.

2 FIELD OF APPLICATION

This International Standard is applicable to technical sodium alkylarylsulphonates, in paste or powder form, and free from any products extraneous to their manufacture, excluding sodium alkylbenzenesulphonates.

It is not applicable to liquid products as these often contain either solvents or additives or both, which would interfere with the methods of analysis specified in this International Standard.

3 REFERENCES

ISO 607, *Surface active agents – Detergents – Methods of sample division.*¹⁾

ISO 894, *Surface active agents – Technical sodium primary alkylsulphates – Method of analysis.*

ISO 4314, *Surface active agents – Determination of free alkalinity or free acidity – Titrimetric method.*

ISO 4316, *Surface active agents – Determination of the pH of aqueous solutions – Potentiometric method.*

ISO 4317, *Surface active agents – Determination of water content – Karl Fischer method.*

ISO 4318, *Surface active agents and soaps – Determination of water content – Azeotropic distillation method.*

ISO . . . , *Surface active agents – Determination of sulphate content – Titrimetric method.*²⁾

4 SAMPLING

Prepare and store a laboratory sample of approximately 300 g according to the instructions given in ISO 607.

5 GENERAL PRINCIPLE³⁾

Preparation of an aqueous alcoholic solution of a test portion, from which are isolated the products extractable by light petroleum.

Dilution of an aliquot portion of the remaining liquid with an appropriate quantity of 2-propanol, saturation with anhydrous sodium carbonate and separation of the sodium alkylarylsulphonates in solution in 2-propanol by salting out.

1) In preparation. (Revision of ISO/R 607.)

2) In preparation.

3) See the general scheme of analysis in annex A.

On separate test portions :

- measurement of pH;
- determination of water content;
- determination of free alkalinity or free acidity;
- determination of sodium sulphite content;
- determination of sodium sulphate content;
- determination of sodium chloride content.

6 METHODS OF ANALYSIS

6.1 Measurement of pH

Carry out the measurement of pH by the method specified in ISO 4316, on a 1 % (*m/m*) solution of the laboratory sample.

6.2 Determination of water content

Depending on the amount of water in the product, carry out the determination according to one of the two following methods :

- a) the Karl Fischer method, applicable to products having less than 10 % (*m/m*) of water;
- b) the azeotropic distillation method, applicable only to products containing more than 5 % (*m/m*) of water.

6.2.1 Karl Fischer method

Carry out the determination of water content by the method specified in ISO 4317.

6.2.2 Azeotropic distillation method

Carry out the determination of water content by the method specified in ISO 4318.

6.3 Determination of free alkalinity or free acidity

Carry out the determination of free alkalinity or free acidity by the method specified in ISO 4314.

6.4 Determination of matter extractable by light petroleum

6.4.1 Introduction

Matter extractable by light petroleum consists of sulphur-free products (non-sulphonated compounds and those which cannot be sulphonated) as well as, where applicable, products containing sulphur which, when present, do not ionize in aqueous solution.

6.4.2 Principle

Extraction with light petroleum of the products specified in 6.4.1 from an aqueous alcoholic solution of the

test portion, taking into consideration the volatility of the products in question.

6.4.3 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

6.4.3.1 Sodium sulphate, anhydrous.

6.4.3.2 2-Propanol, 98 % (*V/V*) solution.

6.4.3.3 2-Propanol, 15 % (*V/V*) solution.

6.4.3.4 Light petroleum, boiling between 40 and 60 °C.

The residue on evaporation shall not be greater than 0,002 % (*m/m*).

6.4.3.5 Sodium hydroxide, approximately 0,1 N solution.

6.4.3.6 Sodium chloride, 200 g/l solution.

6.4.3.7 Phenolphthalein, 1 g/l solution in ethanol.

6.4.4 Apparatus

Ordinary laboratory apparatus and

6.4.4.1 Round-bottomed flask, of capacity 250 ml, with ground glass neck.

6.4.4.2 Fractionating column, of length 20 cm and inside diameter approximately 10 mm, having a ground glass cone at its lower end to fit into the neck of the flask (6.4.4.1).

6.4.4.3 Glass condenser, of nominal (jacket) length 160 mm, complying with the requirements of ISO 4799.

6.4.4.4 Three separating funnels, of capacity 500 ml, with ground glass stoppers complying with the requirements of ISO 4800.

6.4.4.5 Conical flask, of capacity 250 ml, complying with the requirements of ISO 1773.

6.4.4.6 One-mark volumetric flask, of capacity 500 ml, complying with the requirements of ISO 1042, with ground glass stopper.

6.4.5 Procedure

6.4.5.1 TEST PORTION

Weigh, to the nearest 0,01 g, a mass of the laboratory sample containing approximately 6 g of sodium alkylaryl-sulphonates, in a 250 ml beaker.

6.4.5.2 DETERMINATION

Dissolve the test portion (6.4.5.1) in 100 ml of water. Heat if necessary, but not to above 50 °C.

Check that the liquid is slightly alkaline to the phenolphthalein solution (6.4.3.7) and, if necessary, render it alkaline with the sodium hydroxide solution (6.4.3.5) until a pale pink colour is obtained with the indicator. Transfer the solution quantitatively to one of the separating funnels (6.4.4.4) and add 40 ml of the 2-propanol solution (6.4.3.2).

Carry out five extractions, using two of the separating funnels alternately, with 50 ml of the light petroleum (6.4.3.4) each time.

During each extraction, shake vigorously and allow the phases to settle well after each separation. Retain both phases after the extractions.

Combine the hydrocarbon phases in the third separating funnel (6.4.4.4) and wash four times with 40 ml portions of the 2-propanol solution (6.4.3.3). Run off the light petroleum phase. If an emulsion forms during the washing procedure, add the sodium chloride solution (6.4.3.6) 5 ml at a time, shaking the flask and allowing the contents to settle after each addition, until the emulsion is destroyed and complete separation occurs. One 5 ml portion will usually be sufficient.

Combine the aqueous 2-propanol washings with the aqueous alcoholic phase remaining after the extractions with light petroleum.

Transfer the light petroleum phases quantitatively from the third separating funnel to the conical flask (6.4.4.5) containing approximately 10 g of the sodium sulphate (6.4.3.1). Shake for at least 3 min and then allow to stand for about 5 min.

Transfer the combined aqueous alcoholic phases that remain, quantitatively to the one-mark volumetric flask (6.4.4.6). Rinse each of the three separating funnels with 20 ml of the 2-propanol solution (6.4.3.3) three times.

Add the rinsing solution to the one-mark volumetric flask, and dilute to the mark with the 2-propanol solution (6.4.3.3).

This solution, L_1 , is used for determination of the sodium alkylarylsulphonates content.

Filter the light petroleum layer through fast-running filter paper into the flask (6.4.4.1), previously tared and containing a few glass beads. Wash the sodium sulphate, the conical flask (6.4.4.5) and the filter with the light petroleum until no grease marks remain on the filter paper.

Fit the fractionating column (6.4.4.2) and condenser (6.4.4.3) to the flask and distil, using a water bath. If water is present in the light petroleum extract, redissolve the material in the light petroleum, filter and repeat the distillation. After distillation, allow the flask to cool and remove the last traces of the solvent under a reduced pressure of 2 kPa (about 15 mmHg) at 30 °C so that the difference between subsequent weighings made at 20 min intervals is only apparent in the third significant figure.

NOTE — If only very little water is found in the extract (and in droplet form), an attempt can be made to remove it by adding about 3 ml of acetone and repeating the process of evaporation as indicated above.

6.4.6 Expression of results

6.4.6.1 METHOD OF CALCULATION

The content of matter extractable by light petroleum is given, as a percentage by mass, by the formula

$$m_1 \times \frac{100}{m_0}$$

where

m_0 is the mass, in grams, of the test portion (6.4.5.1);

m_1 is the mass, in grams, of the residue obtained.

6.4.6.2 REPRODUCIBILITY

The difference between the results obtained on the same sample, in two different laboratories, should not exceed 1 %.

6.5 Determination of sodium alkylarylsulphonates content

6.5.1 Principle

Addition to 2-propanol of an aliquot portion of the aqueous alcoholic liquor L_1 from the previous determination (see 6.4.5.2), and saturation with sodium carbonate at 45 to 50 °C.

Salting out of the sodium alkylarylsulphonates and dissolution in the separated 2-propanol.

Isolation of the sodium alkylarylsulphonates by evaporation of the solvent.

6.5.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

6.5.2.1 Sodium carbonate, anhydrous.

6.5.2.2 1,4-Dioxan.

6.5.2.3 2-Propanol, 98 % (V/V) solution.

6.5.2.4 Sulphuric acid, 0,1 N standard volumetric solution.

6.5.2.5 Phenolphthalein, 1 g/l solution in ethanol.

6.5.3 Apparatus

Ordinary laboratory apparatus and

6.5.3.1 Two crystallizing dishes, of diameter 80 mm and capacity 200 ml.

6.5.3.2 One-mark volumetric flask, of capacity 250 ml, complying with the requirements of ISO 1042.

6.5.3.3 Conical flask, of capacity 250 ml, complying with the requirements of ISO 1773, with ground glass stopper.

6.5.3.4 Separating funnel, of capacity 250 ml, complying with the requirements of ISO 4800.

6.5.3.5 Sintered glass filter funnel, of porosity P40 (pore diameter 16 to 40 μm).

6.5.3.6 Oven, capable of being controlled at 100 to 105 °C.

6.5.3.7 Pipettes, of capacity 50 and 100 ml, complying with the requirements of ISO 648.

6.5.4 Procedure

6.5.4.1 TEST PORTION

Using one of the pipettes (6.5.3.7), take 100 ml of the aqueous alcoholic solution L_1 produced during the previous determination (see 6.4.5.2) and place it in the conical flask (6.5.3.3).

6.5.4.2 DETERMINATION

Add 70 ml of the 2-propanol solution (6.5.2.3) to the test portion (6.5.4.1). Warm a water bath to 45 to 50 °C. Stirring all the time, add the sodium carbonate (6.5.2.1), 4 to 5 g at a time with continuous stirring, until no more will dissolve (30 to 35 g is generally sufficient).

Stopper the conical flask and shake vigorously for 5 min, keeping the temperature at 45 to 50 °C. Allow the phases to separate at this temperature; if separation is unsatisfactory, add 2 ml of the 1,4-dioxan (6.5.2.2).

Allow to cool, without stirring, for 2 h at 30 °C. It is advisable not to allow the temperature to fall below this value, in order to prevent the sodium carbonate from crystallizing out when the solution is transferred to the separating funnel, as this makes separation very difficult. For the same reason, it is advisable to warm the separating funnel to 40 to 50 °C before transferring the contents of the conical flask to it.

Transfer the liquid in the conical flask to the separating funnel (6.5.3.4). Run off the lower aqueous phase. Wash the sediment in the conical flask twice with 20 ml of the 2-propanol solution (6.5.2.3) and add the washings to the separating funnel.

If necessary, remove carefully all the aqueous phase. Transfer the 2-propanol phase to the volumetric flask (6.5.3.2), filtering on the sintered glass filter (6.5.3.5) if necessary. Wash the filter and the separating funnel with the 2-propanol solution (6.5.2.3), add the washings to the volumetric flask and dilute to the mark with the 2-propanol solution.

Add to each of the two crystallizing dishes (6.5.3.1), previously weighed, exactly 50 ml of the 2-propanol extract, using one of the 50 ml pipettes (6.5.3.7). Evaporate nearly to dryness on a water bath in a fume cupboard and add with the pipette a further 50 ml of the 2-propanol extract to each of the dishes. Evaporate again to dryness on a water bath in a fume cupboard and dry in the oven (6.5.3.6), controlled at 100 to 105 °C, until the

difference between the results of two successive weighings carried out at intervals of 30 min is apparent only in the third significant figure.

Dissolve one of the two residues so obtained in water, heating if necessary to achieve complete solution. Check that this solution is alkaline to the phenolphthalein (6.5.2.5), and determine the content of any sodium carbonate carried over during the extraction, by titration with the sulphuric acid solution (6.5.2.4), using the phenolphthalein solution (6.5.2.5) as indicator. Then determine the content of chloride (Cl^-) ions in the remaining aqueous liquor, from any sodium chloride which may have been carried over, using the potentiometric method (see 6.8) and all the remaining aqueous liquor as the test portion.

6.5.5 Expression of results

6.5.5.1 METHOD OF CALCULATION

The sodium alkylarylsulphonates content is given, as a percentage by mass, by the formula

$$\left(\frac{m_1 + m_2}{2} \right) \times \left(1 - \frac{0,010\ 6\ V_0 + 0,005\ 85\ V_1}{m_2} \right) \times \frac{250}{100} \times \frac{500}{100} \times \frac{100}{m_0} = \left(\frac{m_1 + m_2}{2} \right) \times$$

$$\left(1 - \frac{0,010\ 6\ V_0 + 0,005\ 85\ V_1}{m_2} \right) \times \frac{1\ 250}{m_0}$$

where

m_0 is the mass, in grams, of the test portion (6.4.5.1);

m_1 is the mass, in grams, of the first residue obtained;

m_2 is the mass, in grams, of the second residue obtained;

V_0 is the volume, in millilitres, of the sulphuric acid solution (6.5.2.4) used in the determination of sodium carbonate content;

V_1 is the volume, in millilitres, of the silver nitrate solution (6.8.2.3 in ISO 894) used in the determination of Cl^- ions content (see 6.8).

NOTE — If the standard volumetric solutions used are not exactly of the strengths specified in the list of reagents, suitable correction factors should be applied when calculating the results.

6.5.5.2 REPRODUCIBILITY

The difference between the results obtained on the same sample in two different laboratories should not exceed 1 %.

NOTE — For a check on the values obtained by the method specified above (content in raw material of technical sodium alkylarylsulphonates), see annex B.

6.6 Determination of sodium sulphite content

6.6.1 Principle

Iodometric titration of an aqueous solution.

6.6.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

6.6.2.1 Hydrochloric acid, approximately 0,1 N solution.

6.6.2.2 Iodine, 0,1 N standard volumetric solution.

6.6.2.3 Sodium thiosulphate, 0,1 N standard volumetric solution.

6.6.2.4 Starch solution.

6.6.3 Apparatus

Ordinary laboratory apparatus and

6.6.3.1 Pipette, of capacity 25 ml, complying with the requirements of ISO 648.

6.6.3.2 Conical flask, of capacity 500 ml, complying with the requirements of ISO 1773.

6.6.4 Procedure**6.6.4.1 TEST PORTION**

Weigh, to the nearest 0,001 g, approximately 2 g of the laboratory sample into a 250 ml beaker.

6.6.4.2 DETERMINATION

Dissolve the test portion (6.6.4.1) in 100 ml of water. Heat if necessary, but not to above 50 °C.

Using the pipette (6.6.3.1), transfer 25 ml of the iodine solution (6.6.2.2) to the conical flask (6.6.3.2) and acidify with 100 ml of the hydrochloric acid solution (6.6.2.1). Stir, and add the dissolved test portion. Stir again and then back-titrate with the sodium thiosulphate solution (6.6.2.3), using the starch solution (6.6.2.4) as indicator.

6.6.4.3 BLANK TEST

Carry out a blank test under the same conditions as the determination, but omitting the test portion.

6.6.5 Expression of results**6.6.5.1 METHOD OF CALCULATION**

The sodium sulphite content is given, as a percentage by

mass, by the formula

$$(V_0 - V_1) \times 0,006\ 304 \times \frac{100}{m_0}$$

where

V_0 is the volume, in millilitres, of the sodium thiosulphate solution (6.6.2.3) used in the blank test;

V_1 is the volume, in millilitres, of the sodium thiosulphate solution (6.6.2.3) used in the determination;

m_0 is the mass, in grams, of the test portion (6.6.4.1).

NOTE — If the standard volumetric solutions used are not exactly of the strengths specified in the list of reagents, suitable correction factors should be applied when calculating the results.

6.6.5.2 REPRODUCIBILITY

The difference between the results obtained on the same sample in two different laboratories should not exceed 0,2 %.

NOTE — If the sodium sulphite content is lower than 0,1 % (m/m), record the result as "traces".

6.7 Determination of sodium sulphate content

Carry out the determination of sodium sulphate content by the method specified in ISO . . .

6.8 Determination of sodium chloride content

Carry out the determination of sodium chloride content by the method specified in sub-clause 6.8 of ISO 894.

7 TEST REPORT

The test report shall include the following particulars :

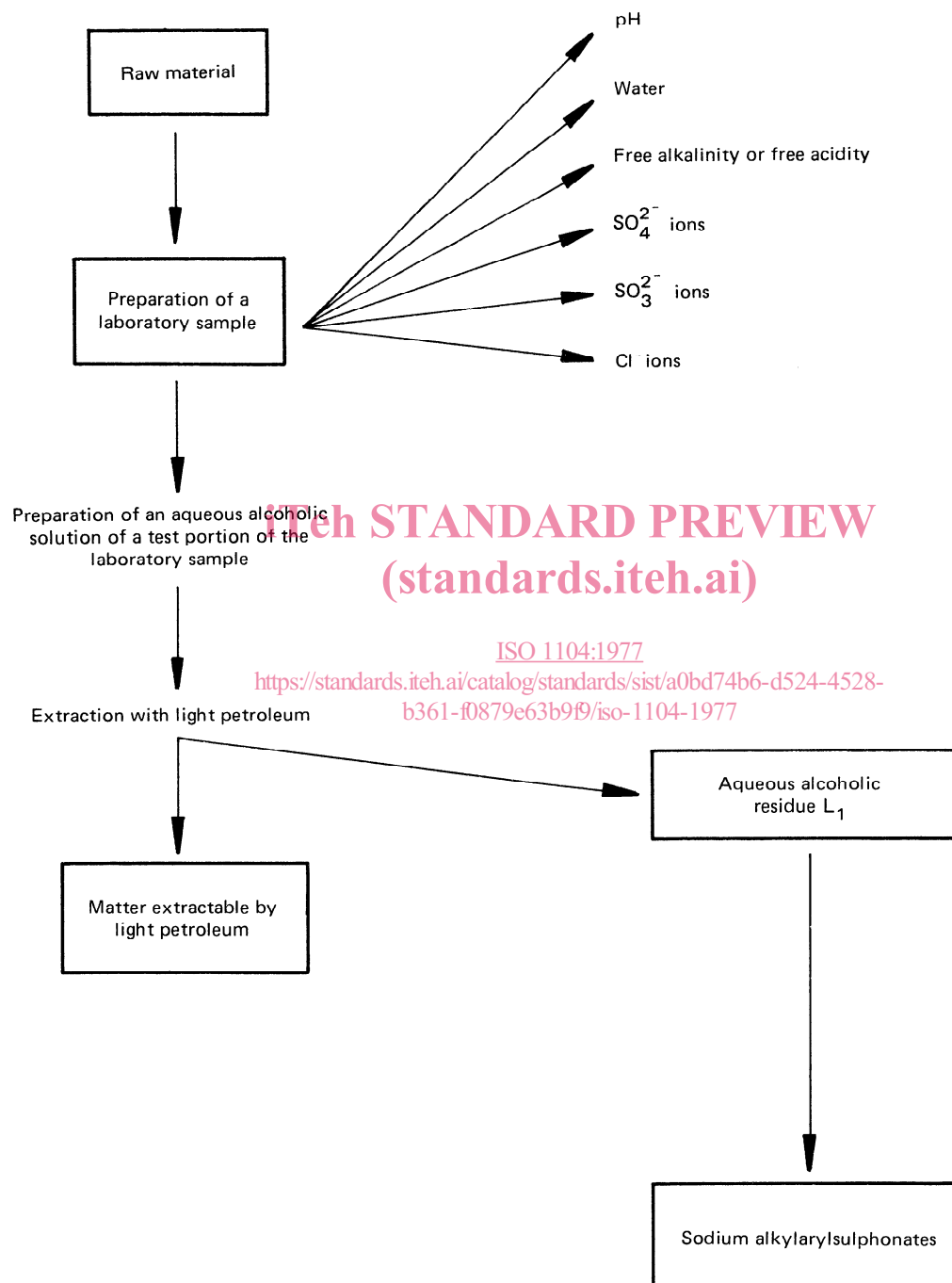
- all information necessary for the complete identification of the sample;
- the reference of the method used (reference to this International Standard);
- the results and the method of expression used;
- the test conditions;
- any operational details not specified in this International Standard or regarded as optional, as well as all incidents likely to have affected the results.

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ANNEX A

GENERAL SCHEME OF ANALYSIS



ANNEX B

DETERMINATION OF SODIUM ALKYLARYLSULPHONATES CONTENT
(Rapid method)

B.0 PRELIMINARY NOTE

If all parties to a transaction agree, the following more rapid analytical method for the determination of sodium alkylarylsulphonates content may be used.

B.1 PRINCIPLE

The principle is the same as that for 6.5.1. Only the procedure differs.

B.2 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

B.2.1 Sodium carbonate, anhydrous.

B.2.2 1,4-Dioxan.

B.2.3 2-Propanol, 98 % (V/V) solution.

B.2.4 Sulphuric acid, 0,1 N standard volumetric solution.

B.2.5 Phenolphthalein, 1 g/l solution in ethanol.

B.3 APPARATUS

Ordinary laboratory apparatus and

B.3.1 Graduated cylinder, of capacity 250 ml, with ground glass stopper, complying with the requirements of ISO 4788.

B.3.2 Pipettes, of capacity 25 and 100 ml, complying with the requirements of ISO 648.

B.3.3 Two anti-creep crystallizing dishes, of capacity 75 ml.

B.4 PROCEDURE**B.4.1 Test portion**

Pipette 100 ml of the aqueous alcoholic solution L_1 , produced during the determination of matter extractable by light petroleum (see 6.4.5.2), into the 250 ml graduated cylinder (B.3.1).

B.4.2 Determination

Add 70 ml of the 2-propanol solution (B.2.3) to the test portion (B.4.1) and place the cylinder in a water bath

maintained at 45 to 50 °C. Add the sodium carbonate (B.2.1), 4 to 5 g at a time, with continuous stirring, until no more will dissolve (30 to 35 g is usually sufficient).

Stopper the cylinder, shake vigorously and place in the water bath at 45 to 50 °C for the phases to separate. If the separation is unsatisfactory, add 2 ml of the 1,4-dioxan (B.2.2).

Allow to cool, without stirring, for 2 h at ambient temperature.

Measure the volume of the upper alcoholic layer in the cylinder.

Remove 25 ml from the middle of this layer with one of the pipettes (B.3.2).

If the solution is cloudy, filter it through paper into one of the crystallizing dishes (B.3.3), both of which have been tared previously. Wash the filter with the 2-propanol solution (B.2.3).

If the solution is clear, transfer it directly to the crystallizing dish.

Repeat this operation for the other crystallizing dish.

Evaporate the solvent on a water bath and dry in an oven controlled at 100 to 105 °C, until the difference between the results of two successive weighings made at 15 min intervals is apparent only in the third significant figure.

Dissolve one of the two residues so obtained in water and, if necessary, warm until it dissolves completely. Determine the sodium carbonate carried over during the extraction by titration with the sulphuric acid solution (B.2.4), using the phenolphthalein solution (B.2.5) as indicator.

B.5 EXPRESSION OF RESULTS**B.5.1 Method of calculation**

The sodium alkylarylsulphonates content is given, as a percentage by mass, by the formula

$$\begin{aligned} & \left(\frac{m_1 + m_2}{2} \right) \times \left(1 - 0,0106 \frac{V_0}{m_2} \right) \times \frac{V_1}{25} \times \frac{500}{100} \times \frac{100}{m_0} = \\ & = \left(\frac{m_1 + m_2}{2} \right) \times \left(1 - 0,0106 \frac{V_0}{m_2} \right) \times \frac{20 V_1}{m_0} \end{aligned}$$

where

m_0 is the mass, in grams, of the test portion (B.4.1);

m_1 is the mass, in grams, of the first residue obtained;

m_2 is the mass, in grams, of the second residue obtained;