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ISO RECOMMENDATION

R 894

SURFACE ACTIVE AGENTS
TECHNICAL SODIUM PRIMARY ALKYLSULPHATES
METHODS OF ANALYSIS

1st EDITION
December 1968

For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 894, *Surface active agents – Technical sodium primary alkylsulphates – Methods of analysis*, was drawn up by Technical Committee ISO/TC 91, *Surface active agents*, the Secretariat of which is held by the Association Française de Normalisation (AFNOR).

Detailed work on this question by the Technical Committee led, in 1963, to the adoption of a Draft ISO Recommendation.

In March 1967, this Draft ISO Recommendation (No. 1035) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia
Austria
Belgium
Brazil
Canada
Czechoslovakia
France
Germany
Greece
Hungary
Iran
Ireland
Israel
Italy
Japan
Netherlands
New Zealand
Portugal
Romania
South Africa, Rep. of
Spain
Switzerland
Turkey
U.A.R.
United Kingdom
Yugoslavia

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in December 1968, to accept it as an ISO RECOMMENDATION.

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ISO Recommendation R 894 December 1968

SURFACE ACTIVE AGENTS

TECHNICAL SODIUM PRIMARY ALKYLSULPHATES

METHODS OF ANALYSIS

INTRODUCTION

The word "primary", preceding the generic name for the products in the title, is intended to distinguish products from those which, in accordance with current scientific usage, could be designated as technical sodium secondary alkylsulphates. As shown in the general formula given below, the former are derived from *primary* alcohols, whereas the latter may be considered as derived from *secondary* alcohols.

It is therefore the former which are the subject of this ISO Recommendation. They are commonly known today as *technical sulphates of primary fatty alcohols*.

In order to simplify the text of this ISO Recommendation and avoid unecessary repetition, the word "primary" has been omitted from the term "sodium alkylsulphates", but it should be understood that only "sodium primary alkylsulphates" are covered.

The general formula of the products which are the subject of this ISO Recommendation is

$$R - CH_2 - O - SO_3Na$$

where R is an aliphatic radical.

PART I

GENERAL

1. SCOPE

This ISO Recommendation describes the methods of analysis of technical sodium primary alkylsulphates. It covers the following determinations :

(1) Determination of water content.

(2) Measurement of pH.

(3) Determination of free alkalinity or free acidity.

(4) Determination of total alkalinity.

(5) Determination of matter extractable by hexane.

(6) Determination of matter extractable by diethyl ether after acid hydrolysis (combined technical fatty alcohols).

(7) Determination of the combined sulphur content arising from sodium alkylsulfate.

(8) Determination of sodium sulphate content.

(9) Determination of sodium chloride content.

It also describes in Appendices,

(1) a general scheme of analysis;

(2) a method for the determination of water, using the Karl Fischer technique with two solutions.

2. FIELD OF APPLICATION

This ISO Recommendation applies only to technical sodium alkylsulphates in powder, paste or liquid form (aqueous solution), free from any product extraneous to their manufacture.

3. PREPARATION OF SAMPLE

3.1 Procedure

In all cases, and as a first step, prepare a representative sample. The procedure depends on the physical nature of the product.

3.1.1 *Products in powder form.* If a suitable conical divider is available, proceed in accordance with ISO Recommendation R 607, *Surface active agents in powder form – Preparation of a reduced sample*, until a reduced sample of approximately 300 g is obtained.

If such a divider is not available, spread a representative mass of 1000 g of the product in powder form on a sheet of white filter paper. Crush any large grains by means of a spatula. Then, using the method of alternate quarters, prepare a representative sample of approximately 300 g.

3.1.2 *Products in paste form.* First of all determine the pH value of the paste by means of contact electrodes or a suitable indicator paper. If the pH is not below 7, the following procedure may be adopted : Stir the paste with a stout glass rod, at the same time exposing it to a moderate heat of 40 to 50 °C (water bath or hot-plate) until a homogeneous mass is obtained. While still stirring it, let it cool to a temperature between 15 and 30 °C, obtaining, in this way a very thick paste, which may be regarded as a representative sample.

The mass (including the glass rod) should be determined both before and after the operation in order to determine any losses due to evaporation of water, which should be taken into consideration.

If, on the other hand, the pH value of the paste is below 7, add for each 50 g of paste 5 drops of 0.2 % alcoholic solution of phenolphthalein, stir with a strong nickel spatula, then still stirring, add 0.5 N alcoholic potassium hydroxide drop by drop until a pink colour is obtained. Then proceed as specified above, including the determination of mass before and after the operation (see Part III, section 2 and Part IV, section 3 of this ISO Recommendation).

3.1.3 *Liquid products.* If the liquid is homogeneous and clear, it may be regarded as a representative sample. If the liquid is turbid, or if it contains a deposit, it should be stirred before the representative sample is taken.

If it contains a compact or crystalline deposit, it should be carefully heated to a temperature of about 30 °C until the deposit can be uniformly distributed by stirring, or until the crystals disappear.

3.2 Storage

Liquid products or those in paste form should be kept in wide-necked flasks with ground-glass stoppers and not in metallic containers.

4. GENERAL PRINCIPLE*

Preparation of an aqueous alcoholic solution of a test portion of the raw material, from which are isolated the products extractable by hexane.

Evaporation to dryness of the residual aqueous alcoholic liquor in the presence of propan-2-ol. Hot acid hydrolysis of the dry residue and isolation from the acid reaction liquor of all products extractable by diethyl ether (combined technical fatty alcohols).

On the residual acid aqueous liquid, determination of the total sulphate. The difference between the percentage of total sulphate and inorganic sulphate determined separately, makes it possible to calculate the content of combined sulphur arising from the sodium alkylsulphate.

On separate test portions of the representative sample of the raw material,

- determination of water content,
- measurement of pH,
- determination of free alkalinity or free acidity,
- determination of total alkalinity,
- determination of sodium sulphate content,
- determination of sodium chloride content.

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* See the general scheme of the method in Appendix Y.

PART II

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 up to 10 % of water;

(*b*) the azeotropic method, which should be used only for products containing more than 5 % of water.

1. KARL FISCHER METHOD

1.1 Introduction

The Karl Fischer reagent is a solution containing anhydrous pyridinium sulphite and iodine, which are transformed in the presence of the slightest trace of water into pyridinium sulphate and hydriodic acid respectively.

It is possible to proceed in one of two ways, as follows :

(*a*) by determining the water in the product directly with the Karl Fischer reagent as a single solution, but since this liquid has reduced stability unless it has been specially stabilized, this technique is recommended only when the determination has to be carried out frequently enough for the single reagent to be used up each day or when the stabilized reagent is available;

(*b*) by determining the water by means of the iodine solution after adding anhydrous pyridinium sulphite solution to the product. These two liquids can be kept much longer apart than when mixed and this technique, although also applicable in the other case, is particularly recommended when determinations have to be carried out at more or less regular intervals.

1.2 Method using a single solution

In a solution or suspension in methanol of a test portion of the representative sample of the raw material such that the water it contains requires a measurable quantity of Karl Fischer reagent, determine the water in accordance with ISO Recommendation R 760, *Determination of water in chemical products by the Karl Fischer method.*

Reproducibility

The difference between the results obtained on the same sample, in different laboratories, should not exceed 0.2 g of water for 100 g of sample.

1.3 Method using two solutions

See Appendix Z.

2. AZEOTROPIC METHOD

2.1 Principle

Entrainment of the water in the vapour state, by boiling xylene.

2.2 Reagent

Xylene, commercial quality (any isomer or mixture of isomers in any proportion) saturated with water.

2.3 Apparatus

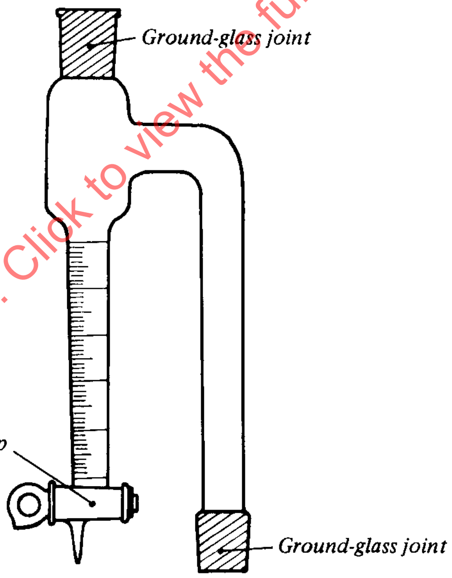
2.3.1 *Apparatus* comprising the following :

(*a*) *a short-necked round-bottomed flask* not less than 500 ml, connected to the tube, shown below, by means of a ground-glass joint;

(*b*) *a graduated cylindrical tube*, shown diagramatically below, with or without discharge tap;

(*c*) *a straight reflux condenser*, connected to the tube by means of a ground-glass joint.

2.3.2 *Analytical balance.*



NOTE. – Before use, clean the graduated tube and the interior of the reflux condenser of all trace of fatty matter, for example, by washing them successively in chromic/sulphuric acid mixture, then with distilled water, and finally with acetone. Dry them. Perfect cleanliness of the apparatus is essential for the success of the test.

2.4 Procedure

2.4.1 *Test portion*. Weigh, to the nearest 0.01 g, 10 to 50 g of the representative sample of the raw material into the flask according to whether it is a liquid, a paste or a powder.

2.4.2 *Determination*. Add to the test portion 100 to 300 ml of xylene and a dry boiling aid, for example a few fragments of pumice stone.

Attach the flask to the rest of the apparatus.

Heat gradually to boiling (2 to 3 drops per second) and maintain at this temperature until the distilled xylene becomes clear and water no longer separates from it.

If drops of water adhere to the sides of the tube, remove them by warming the tube carefully, using a small flame.

If foaming is troublesome during the determination, it can be eliminated by adding, for example, dry paraffin wax or dry oleic acid to the flask.

Leave to settle until the water has completely separated without an emulsified layer.

Read the volume of the water in the graduated tube, at the standard reference temperature of 20 °C.

2.5 Expression of results

2.5.1 *Method of calculation and formula*. The percentage of water in the sample is equal to

$$V \times \frac{100}{M}$$

where

M is the mass, in grammes, of the test portion;

V is the volume, in millilitres, of the aqueous layer.

2.5.2 *Reproducibility*. The difference between results obtained on the same sample, in two different laboratories, should not exceed 1 g of water for 100 g of sample.

PART III

MEASUREMENT OF pH

1. PROCEDURE

Using distilled or de-ionized water of pH between 6.5 and 7, prepare a solution 5 % (m/m) of the representative sample of the raw material (see section 2 below). Measure the pH to the nearest 0.1 unit, by means of a pH meter suitable for this purpose.

Read the meter as rapidly as possible, i.e. between 30 seconds and 1 minute after immersion of the electrodes.

2. NOTE

In the case of products in paste form, a distinction has to be made between two cases, i.e. during preparation of the sample (see Part I, clause 3.1.2 of this ISO Recommendation), the raw material in paste form, to be analysed, has shown on preliminary examination either

(*a*) *a pH not below 7*, in which case, it is possible to apply the procedure described in section 1, to a test portion of the representative sample of the raw material, prepared in accordance with Part I, clause 3.1.2 of this ISO Recommendation, or

(*b*) *a pH below 7*, in which case it is necessary to apply the procedure described in section 1 to a test portion of the raw material as it is, whatever its state of homogeneity.

PART IV

DETERMINATION OF FREE ALKALINITY OR FREE ACIDITY

1. DEFINITION

By "free alkalinity" or "free acidity" is understood alkalinity or acidity determined using phenolphthalein as indicator under specified conditions. It is expressed as the alkali value or the acid value as the case may be.

2. PROCEDURE

Dilute or, where applicable, dissolve 10 g of the representative sample of the raw material (see section 3 below) in 100 ml of propan-2-ol, 50 % (v/v), previously neutralized in the presence of phenolphthalein, and determine either the acidity or the alkalinity, by titration with a suitable 0.1 N standard volumetric solution (0.1 N potassium hydroxide or 0.1 N hydrochloric or sulphuric acid).

3. NOTE

In the case of products in paste form, a distinction has to be made between two cases, i.e. during preparation of the sample (see Part I, clause 3.1.2 of this ISO Recommendation) the raw material in past form, to be analysed, has shown on preliminary examination

(*a*) *a pH not below 7*, in which case it is possible to apply the procedure described in section 2 to a test portion of the representative sample of the raw material, prepared in accordance with Part I, clause 3.1.2 of this ISO Recommendation;

(*b*) *a pH below 7*, in which case it is necessary to apply the procedure described in section 2, to a test portion of the raw material as it is, whatever its state of homogeneity.

4. EXPRESSION OF RESULTS

Express the results as the acid value or alkali value, whichever is appropriate, i.e. in milligrammes of potassium hydroxide required to neutralize 1 g of the representative sample of the raw material.

The acid value (or alkali value) is equal to

$$V \times \frac{5.61}{M}$$

where

M is the mass, in grammes, of the test portion;

V is the volume, in millilitres, of the standard volumetric solution used.

NOTES

1. If the standard volumetric solution used is not exactly of strength 0.1 N, a suitable correction factor should be used in calculating the results.
2. When the value found is less than 0.3, express the result as follows :

Acid value or alkali value (as appropriate) < 0.3.

PART V

DETERMINATION OF TOTAL ALKALINITY

1. INTRODUCTION

It may happen that on measuring the pH value, in accordance with Part III of this ISO Recommendation, a pH which is significantly greater that 7 is observed, and that on determining the alkalinity, in accordance with Part IV, an alkali value significantly greater than 0.3 is obtained. In such a case, it is advisable to carry out the determination of the total alkalinity as below.

2. DEFINITION

By "total alkalinity" is understood the alkalinity determined using methyl orange (or bromophenol blue) as indicator under specified conditions. It is expressed as alkali value.

3. PROCEDURE

Weigh, to the nearest 0.001 g, about 5 g of the representative sample of the raw material into a 100 ml beaker. Transfer the product quantitatively with 100 ml of distilled water into a 250 ml conical flask, heating if necessary.

Add 5 drops of methyl orange (or bromophenol blue) and titrate at room temperature with a solution of 0.1 N sulphuric acid until a colour change to a brownish red (or to yellow depending on the indicator used) is observed.

4. EXPRESSION OF RESULTS

Express the results as the alkali value as defined in Part IV of this ISO Recommendation.

The total alkalinity, expressed as the alkali value, is equal to

$$V \times \frac{5.61}{M}$$

where

M is the mass, in grammes, of the test portion;

V is the volume, in millilitres, of the standard volumetric solution used.

NOTE. – If the standard volumetric solution used is not exactly of strength 0.1 N specified above, a suitable correction factor should be used in calculating the results.

PART VI

DETERMINATION OF MATTER EXTRACTABLE BY HEXANE

1. INTRODUCTION

Matter extractable by hexane consists of sulphur-free products as well as products containing sulphur, which, when present, are non-ionizable in aqueous solution.

2. PRINCIPLE

Extraction, by means of hexane, of the products specified in section 1, in the aqueous alcoholic solution of the test portion, taking into consideration the volatility of the products in question.

3. REAGENTS

3.1 *Ethanol*, 50 % (v/v) solution in distilled water.

3.2 *Ethanol*, 96 % (v/v) solution.

3.3 *Hexane*, not less than 95 % (v/v).*

3.4 *Sodium hydroxide*, approximately 0.1 N solution in distilled water.

3.5 *Sodium sulphate*, neutral and anhydrous, analytical reagent grade.

3.6 *Phenolphthalein*, 0.2 % solution in ethanol.

4. APPARATUS

Ordinary laboratory apparatus not otherwise specified, and the following items:

(*a*) *round-bottomed distillation flask*, 250 ml, with ground-glass neck;

(*b*) *fractionating column* 20 cm long and inside diameter approximately 8 mm, with a ground-glass cone at its lower end to fit into the neck of the distillation flask;

(*c*) *three separating funnels*, 500 ml, with ground-glass stoppers;

(*d*) *vertical condenser*, 80 cm long;

(*e*) *beakers*, 100 and 400 ml;

(*f*) *conical flask*, 300 ml;

(*g*) *hotplate* or *water bath*;

(*h*) *analytical balance*.

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* If hexane is not available, use freshly distilled light petroleum spirit, of boiling range 30 to 60 °C. In reporting the results, it should be stated which of the two solvents has been used.

5. PROCEDURE

5.1 Test portion

Weigh, to the nearest 0.01 g, a quantity of the representative sample of the raw material, such that it contains approximately 4 g of sodium primary alkylsulphate, into the 100 ml beaker.

Let M_0 be the mass weighed.

5.2 Determination

Dissolve or, if necessary, dilute the test portion in 50 ml of very hot distilled water (approximately 70 °C) stirring with a glass rod, then add 50 ml of ethanol (3.2) while continuing to stir. Introduce the liquid obtained into one of the 500 ml separating funnels (A). Wash the beaker alternately with small equal portions of very hot distilled water and of ethanol (3.2) until the final volume is approximately 300 ml.

Check that the liquid is slightly alkaline to phenolphthalein, and if necessary render it alkaline by means of the sodium hydroxide solution (3.4) until a pale pink colour is obtained with the indicator (3.6).

Shake to render the mixture homogeneous. Allow to cool.

Add 50 ml of hexane (3.3).

Shake vigorously for approximately 30 seconds and allow to separate. Add the minimum quantity of ethanol (3.2) necessary to break any emulsion that may be formed.

Transfer the lower layer into a second separating funnel (B).

Extract with another 25 ml portion of hexane (3.3).

Collect the lower layer in a third separating funnel (C) and transfer the upper layer into the first separating funnel (A).

Extract the aqueous alcoholic phase three more times, each time using 25 ml of hexane (3.3).

Combine the hydrocarbon phases in the separating funnel (A) and transfer the aqueous alcoholic phase into the 400 ml beaker after the last extraction.

Wash the separating funnels (B) and (C) three times, each time using 10 ml of ethanol (3.1).

Add the washings to the aqueous alcoholic phase in the beaker.

Wash the hydrocarbon extract with portions of ethanol (3.1) until the washings are no longer alkaline; add the washings to the aqueous alcoholic phase.

This solution L_1 is used for the determination of the content of combined technical fatty alcohols and of the content of combined sulphur originating from the sodium alkylsulphate.

Transfer the hydrocarbon layer quantitatively into a 300 ml conical flask containing anhydrous neutral sodium sulphate (3.5). Shake the liquid and allow it to stand for 30 minutes, then filter through filter paper into the distillation flask, previously tared. Wash the conical flask, the sodium sulphate and the filter five times, each time using 10 ml of hexane (3.3). Pay particular attention to the edges of the filter paper, which should not show any greasy marks.

Fit the fractionating column to the distillation flask, place the assembly on the hotplate or in the water bath and distil until almost all the solvent has passed over. Remove the fractionating column, cool to about 30 °C and eliminate the last traces of solvent by a gentle current of air.

To do this maintain the current of air and rotate the flask by hand in an inclined position above the hotplate or water bath. In this way, the liquid in the flask will spread over the interior in a thin film and so assist in the removal of the last traces of solvent.

It is advisable to bear in mind that lower alcohols, such as lauric alcohol for example, are fairly volatile, particularly in the presence of traces of moisture, which, in some circumstances, may cause quite considerable losses. Therefore, particular care is necessary in evaporating the solvent, especially while passing the current of air.

For this purpose, first weigh the flask, cooled to room temperature and carefully dried, while there is still a detectable odour of solvent. Note the mass, then reheat the flask to approximately 30 °C so as to liquefy the contents and pass a current of air over them for a further minute. After cooling and drying the flask, weigh it again and note the mass.

By repeating these operations and plotting successive weighings on a graph on millimetre squared paper, it will be noted that, after a rapid drop, the curve reaches a practically horizontal lower level. The second weighing on the horizontal part is regarded as the end of the operation and the mass noted is recorded as that of the final dry residue.

Any difference between the last two weighings should be apparent only in the third significant figure.

6. EXPRESSION OF RESULTS

6.1 Method of calculation and formula

The percentage of matter extractable by hexane in the representative sample of the raw material is equal to

$$M_1 \times \frac{100}{M_0}$$

where

M_0 is the mass, in grammes, of the test portion;

M_1 is the mass, in grammes, of the residue obtained.

6.2 Reproducibility

The difference between results obtained on the same sample, in two different laboratories, should not exceed 1 g of matter extractable by hexane for 100 g of sample.

PART VII

DETERMINATION OF MATTER EXTRACTABLE BY DIETHYL ETHER AFTER ACID HYDROLYSIS (combined technical fatty alcohols)

1. PRINCIPLE

Evaporation, to a fifth of its volume, of the aqueous alcoholic liquid resulting from the previous determination, then, after the addition of propan-2-ol, evaporation to dryness. It is very important that the solution remain alkaline throughout the whole of this determination.

Dissolution of the dry residue in distilled water, then, after the addition of hydrochloric acid, acid hydrolysis, at boiling point. When the hydrolysis is complete, cold extraction by diethyl ether of all the fatty substances released.

These fatty substances are constituted almost entirely of combined fatty alcohols, but they may, in some cases, contain sulphonic derivatives (true sulphonics) and fatty acids, generally in very small proportions.

2. REAGENTS

2.1 *Propan-2-ol*, 98 % solution (v/v), rendered slightly alkaline with a 0.1 N sodium hydroxide solution in the presence of phenolphthalein.

2.2 *Hydrochloric acid*, (ρ_{20} = 1.19 g/ml) analytical reagent grade, sulphate free.

2.3 *Diethyl ether.*

2.4 *Hydrochloric acid*, approximately 1 N solution in distilled water.

2.5 *Sodium sulphate*, neutral and anhydrous.

3. APPARATUS

Ordinary laboratory apparatus not otherwise specified, and the following items

(*a*) *saponification flask*, 250 ml, fitted with a condenser;

(*b*) *three separating funnels*, 500 ml;

(*c*) *conical flask*, 300 ml;

(*d*) *volumetric flask*, 250 ml;

(*e*) *round-bottomed distillation flask*, 250 ml, with ground-glass neck;

(*f*) *fractionating column*, 20 cm long, inside diameter approximately 8 mm, with a ground-glass cone at its lower end, which can be fitted to the neck of the flask;

(*g*) *straight condenser*, 80 cm long;

(*h*) *glass beads*;

(*i*) *beaker*, 400 ml;

(*j*) *analytical balance*, accurate to 0.1 mg.

4. PROCEDURE

4.1 Test portion

Use the aqueous alcoholic solution, L_1, resulting from the determination of matter extractable by hexane (see Part VI of this ISO Recommendation) and corresponding to the test portion M_0.

4.2 Determination

Reduce the solution to approximately one fifth of its volume by evaporation on the water bath, using a current of air. Add 20 ml of propan-2-ol (2.1) and evaporate to dryness.

Dissolve, heating if necessary, in 50 ml of distilled water and transfer quantitatively to the saponification flask by means of distilled water, bringing the final volume to approximately 100 ml. Add two glass beads and then, very carefully, 35 ml of hydrochloric acid (2.2). (Care is necessary to avoid losses from the flask due to overflow of the foam resulting from decomposition of any carbonates formed during the evaporation to dryness).

Attach the condenser and reflux for at least four hours. Cool and transfer quantitatively to one of the 500 ml separating funnels (A). Wash the condenser and flask carefully and quantitatively with diethyl ether (2.3) and distilled water, which is then added to the liquid in the separating funnel.

Shake in order to homogenize thoroughly.

Add 30 ml of diethyl ether (2.3). Shake vigorously and allow to settle.

Transfer the lower layer to a second separating funnel (B). Extract once again with 30 ml of diethyl ether (2.3). Collect the lower layer in a third separating funnel (C) and transfer the upper layer to the first separating funnel (A).

Extract the aqueous phase three more times, each time using 30 ml of diethyl ether (2.3).

Combine the ethereal phases in separating funnel (A) and transfer the aqueous phase after the last extraction into a 400 ml beaker.

Wash funnels (B) and (C) three times, each time using 20 ml of distilled water. Add the washings to the aqueous phase contained in the beaker.

Wash the ethereal extract three times, each time using 15 ml of dilute hydrochloric acid (2.4), and add the washings to the aqueous phase.

Evaporate the latter on the boiling water bath to less than 200 ml, then transfer quantitatively to the 250 ml volumetric flask. Using distilled water, dilute to the mark; the solution thus obtained, L_2, is used for the determination of the content of combined sulphur arising from the sodium alkylsulphate (see Part VIII of this ISO Recommendation).

Transfer the ethereal extract quantitatively into the 300 ml conical flask containing sodium sulphate (2.5). Shake, allow to settle for 30 minutes, then filter through a filter paper into the distillation flask previously tared. Wash the conical flask, the sodium sulphate and the filter five times, each time using 10 ml of diethyl ether (2.3). Pay particular attention to the edges of the filter paper, which should not show any greasy marks.

Fit the fractionating column to the distillation flask, place the assembly on the water bath and distill until almost all the solvent has passed over. Remove the fractionating column and eliminate the last traces of solvent by a gentle current of air. For this purpose, while maintaining the current of air, rotate the flask by hand in an inclined position about its own axis outside the water bath. In this way the liquid in the flask is spread over the walls in a thin film which facilitates the elimination of the last traces of solvent.

It is advisable to bear in mind that lower alcohols, such as lauric alcohols, are fairly volatile, particularly in the presence of traces of moisture, which, in some circumstances, may cause quite considerable losses. Therefore, particular care is necessary in evaporating the solvent, especially while passing the current of air.

For this purpose, first weigh the flask, cooled to room temperature and carefully dried, while there is still a detectable odour of solvent. Note the mass, then reheat the flask to approximately 30 °C so as to liquefy the contents and pass a current of air over them for a further minute. After cooling and drying the flask, weigh it again and note the mass.

By repeating these operations and plotting successive weighings on a graph on millimetre squared paper, it will be noted that, after a rapid drop, the curve reaches a practically horizontal lower level, The second weighing on the horizontal part is regarded as the end of the operation and the mass noted is recorded as that of the final dry residue.

Any difference between the last two weighings should be apparent only in the third significant figure.

5. EXPRESSION OF RESULTS

5.1 Method of calculation and formula

The percentage of matter in the raw material extractable by diethyl ether after acid hydrolysis is equal to

$$M_1 \times \frac{100}{M_0}$$

where

M_0 is the mass, in grammes, of the test portion (see Part VI of this ISO Recommendation);

M_1 is the mass, in grammes, of the residue obtained.

5.2 Reproducibility

The difference between results obtained on the same sample, in two different laboratories, should not exceed 1 g of matter extractable by diethyl ether for 100 g of sample.

NOTES

1. The determination of the alkali value and the acid value should be carried out on the combined technical fatty alcohols, isolated as described in Part VII of this ISO Recommendation.
2. The acid value should be less than or, at most, equal to 2.

PART VIII

DETERMINATION OF THE COMBINED SULPHUR CONTENT ARISING FROM THE SODIUM ALKYLSULPHATE

1. PRINCIPLE

Determination of the total content of SO_4^{--} ions in solution L_2, resulting from the acid hydrolysis (see Part VII of this ISO Recommendation), by precipitation as barium sulphate. Calculation of the content of combined sulphur arising from the sodium alkylsulphate, after deduction of the content, as sodium sulphate (see Part IX of this ISO Recommendation), of SO_4^{--} ions in the test portion.

2. REAGENT

Barium chloride dihydrate ($BaCl_2.2H_2O$), 100 g/l solution in distilled water.

3. APPARATUS

Ordinary laboratory apparatus.

4. PROCEDURE

4.1 Test portion

Use solution L_2 resulting from the acid hydrolysis (see Part VII of this ISO Recommendation).

4.2 Determination

On an aliquot portion, determine gravimetrically the SO_4^{--} ions as barium sulphate.

5. EXPRESSION OF RESULTS

5.1 Method of calculation and formula

The percentage of combined sulphur arising from the sodium alkylsulphate is equal to

$$0.1374 \times M_1 \times \frac{250}{V} \times \frac{100}{M_0} - 0.2257 \times P = \frac{3435\, M_1}{V \times M_0} - 0.2257\, P$$

where

M_0 is the mass, in grammes, of the test portion (see Part VI of this ISO Recommendation);

M_1 is the mass, in grammes, of the precipitate;

V is the volume, in millilitres, of the aliquot portion;

P is the percentage of sodium sulphate (see Part IX of this ISO Recommendation).

5.2 Reproducibility

The difference between results obtained on the same sample, in two different laboratories, should not exceed 0.4 g of combined sulphur for 100 g of sample.

PART IX

DETERMINATION OF SODIUM SULPHATE CONTENT

1. PRINCIPLE

Precipitation of insoluble mineral salts from a hot test portion of the representative sample of the raw material by means of propan-2-ol, followed by filtration. Dissolution of these salts in distilled water and determination in this solution of the content of SO_4^{--} ions by precipitation as barium sulphate.

2. REAGENTS

2.1 *Propan-2-ol*, 98 % solution.

2.2 *Dilute hydrochloric acid* (ρ_{20} = 1.06 g/ml), obtained by addition of 1 volume of hydrochloric acid (ρ_{20} = 1.19 g/ml), to 2 volumes of distilled water.

2.3 *Barium chloride dihydrate* ($BaCl_2.2H_2O$), 100 g/l solution in distilled water.

2.4 *Congo red paper.*

3. APPARATUS

Ordinary laboratory apparatus not otherwise specified, and the following items:

(*a*) *beakers*, 250 ml;

(*b*) *filter crucible* of sintered glass, porosity 5 to 15 µm;

(*c*) *analytical balance.*

4. PROCEDURE

4.1 Test portion

Into a 250 ml beaker, weigh to the nearest 0.01 g, a mass of the representative sample of the raw material corresponding to approximately 0.05 g of insoluble product.

Let M_0 be the mass weighed.

4.2 Determination

Dilute or dissolve the test portion with 200 ml of propan-2-ol (2.1) and allow to boil gently for 30 minutes.

Allow to settle at room temperature until separation is complete. Pour off the liquid phase through the sintered glass filter. Treat the residue in the beaker in the same way with three successive 30 ml portions of boiling propan-2-ol (2.1), then dissolve the residue remaining in the beaker and any residue on the sintered glass filter with 75 to 100 ml of boiling water. Evaporate this solution to dryness in a 250 ml beaker, then dissolve the residue in 100 ml of hot water.

While hot, render just acid to congo red indicator with hydrochloric acid (2.2), taking special care at the start (because of foaming and splashing due to alkaline carbonates). Stir, bring to the boil and determine the SO_4^{--} ion content gravimetrically as barium sulphate.

5. EXPRESSION OF RESULTS

5.1 Method of calculation and formula

The percentage of sodium sulphate in the raw material is equal to

$$0.6085 \times M_1 \times \frac{100}{M_0} = \frac{60.85\,M_1}{M_0}$$

where

M_0 is the mass, in grammes, of the test portion;

M_1 is the mass, in grammes, of the precipitate.

5.2 Reproducibility

The difference between results obtained on the same sample, in two different laboratories, should not exceed 0.4 g of sodium sulphate for 100 g of sample.

NOTE. – If the sodium sulphate content is less than 0.1 %, state "traces".