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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 895

SURFACE ACTIVE AGENTS

TECHNICAL SODIUM SECONDARY 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 895, *Surface active agents – Technical sodium secondary 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. 1036) 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.

CONTENTS

ISO Recommendation R 895 December 1968

SURFACE ACTIVE AGENTS

TECHNICAL SODIUM SECONDARY ALKYLSULPHATES

METHODS OF ANALYSIS

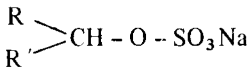
INTRODUCTION

The word "secondary", 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 primary alkylsulphates. As shown in the general formula given below, the former may be considered as derived from *secondary* alcohols, whereas the latter are derived from *primary* alcohols.

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

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

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



$$\begin{matrix} R \\ R' \end{matrix} \!\!> CH - O - SO_3Na$$

where R and R' are aliphatic radicals.

PART I

GENERAL

1. SCOPE

This ISO Recommendation describes the methods of analysis of technical sodium secondary 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 the content of sodium secondary alkylsulphates.

(7) Determination of the organic sulphur content arising from the sulphate group (Wurzschmitt method).

(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 the content of organic sulphur arising from the sulphate group of technical sodium alkylsulphates, (isolated in accordance with Part VII of this ISO Recommendation) (the Grote-Krekeler method).

2. FIELD OF APPLICATION

This ISO Recommendation is applicable only to technical sodium secondary alkylsulphates in liquid form, free from other products extraneous to their manufacture.

It is not at present applicable to powders or pastes.

3. PREPARATION OF SAMPLE

As the material for analysis is a liquid (see section 2) and is thus homogeneous at a temperature of 20 °C, it is not necessary to carry out the procedure for the preparation of a representative sample.

Storage

Liquid products 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.

Separation of the sodium alkylsulphates from an aliquot portion of the residual aqueous alcoholic liquid, after repeated evaporation to dryness in the presence of propan-2-ol and final extraction of the anhydrous residue by means of propan-2-ol.

Determination of the organic sulphur content, after complete evaporation of the solvent.

On separate test portions 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 Appendix Y for general scheme of the method.

PART II

DETERMINATION OF WATER CONTENT

1. PRINCIPLE

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

2. REAGENT

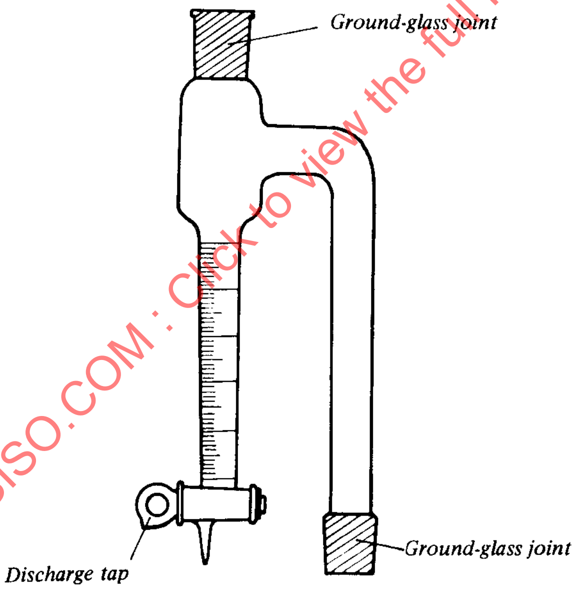
Xylene, commercial quality (any isomers or mixture of isomers in any proportion) saturated with water.

3. APPARATUS

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 diagrammatically below, with or without discharge tap;
- *c*) *a straight reflux condenser,* connected to the tube by means of a ground-glass joint;

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.

4. PROCEDURE

4.1 Test portion

Weigh, to the nearest 0.01 g, about 5 g of the raw material, into the round-bottomed flask.

4.2 Determination

Add to the test portion, 250 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 point (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.

5. EXPRESSION OF RESULTS

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.

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

PROCEDURE

Using distilled or deionized water of pH between 6.5 and 7, prepare a solution of 5 % (m/m) of the raw material. 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.

PART IV

DETERMINATION OF FREE ALKALINITY OF 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 10 g of the raw material 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 standard volumetric solution of strength 0.1 N (0.1 N potassium hydroxide or 0.1 N hydrochloric or sulphuric acid).

3. EXPRESSION OF RESULTS

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

The acid value (or the alkali value) is equal to

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

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 in accordance with Part III of this ISO Recommendation, a pH which is significantly greater that 7 is observed, and that on determing 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 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) 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 alkali value, is equal to

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

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, 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 I in the aqueous alcoholic solution of the test portion, taking into consideration the volatility of the products in question.

3. REAGENTS

3.1 *Propan-2-ol,* 98 % (v/v) solution.

3.2 *Hexane,* not less than 95 %.*

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

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

3.5 *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 ml and 400 ml;

(*f*) *volumetric flask,* 500 ml;

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

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

(*i*) *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 raw material containing approximately 4 g of sodium secondary alkylsulphates, into the 100 ml beaker.

Let M_0 be the mass weighed.

5.2 Determination

Introduce the test portion into one of the 500 ml separating funnels (A) and wash the beaker with distilled water so as to obtain a final volume of approximately 125 ml; add 50 ml of propan-2-ol (3.1).

Check that liquid is faintly alkaline to phenolphthalein, and if necessary make it so with sodium hydroxide solution (3.3) until a pale pink colour is obtained with the indicator (3.5).

Shake to render the mixture homogeneous.

Add 50 ml of hexane (3.2).

Shake *vigorously* for 30 seconds and allow to separate. Add the minimum quantity of propan-2-ol (3.1) 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 (3.2).

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

Extract the aqueous alcoholic phase three more times, each time using 25 ml of hexane (3.2).

Combine the hydrocarbon extracts in the separating funnel (A) and transfer the aqueous alcoholic layer to the 400 ml beaker after the last extraction.

Wash both the separating funnels (B) and (C) three times, each time using 20 ml of distilled water. Alternatively, a 5 to 10 % solution of propan-2-ol in distilled water may be used.

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

Wash the hydrocarbon extract with successive portions of 15 ml of distilled water until the washings are no longer alkaline; add the washings to the aqueous alcoholic phase.

Heat the aqueous alcoholic phase on a boiling water bath for 10 to 15 minutes to evaporate the hexane and allow to cool.

Ensure that the solution is still alkaline to phenolphthalein, and if necessary render alkaline with sodium hydroxide solution (3.3).

Transfer the solution to the 500 ml volumetric flask, rinsing the beaker with water and adding the washings to the volumetric flask. Dilute to the mark.

This solution, L_1, will be used for the determination of the content of sodium alkylsulphates.

Transfer the hydrocarbon layer quantitatively into a 300 ml conical flask, containing sodium sulphate (3.4). Shake the liquid and allow it to stand for 30 minutes. Then filter through filter paper into the distillation flask, previously tared, containing a few glass beads. Wash the conical flask, the sodium sulphate and the filter five times, each time using 10 ml of hexane (3.2). Pay particular attention to the edges of the filter paper, which should not show greasy marks.

Fit the fractionating column to the distillation flask, place the assembly on the hotplate or in the water bath and distil until nearly 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 distillation flask by hand, in an inclined position, out of 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.

To avoid loss, care is necessary in evaporating the solvent, particularly 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 fall, 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 two last 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 raw material, is equal to

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

where

M_0 is the masse, 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 THE SODIUM SECONDARY ALKYLSULPHATES CONTENT

1. PRINCIPLE

Evaporation of an aliquot portion of the aqueous alcoholic liquid, L_1, from the previous determination to one fifth of its volume, addition of propan-2-ol, then evaporation to dryness. Further addition of propan-2-ol and evaporation to dryness (these successive evaporations are made to remove completely the water in the aqueous alcoholic solution). Extraction of the sodium secondary alkylsulphates by means of hot propan-2-ol, from the dry residue thus obtained.

Isolation of the sodium secondary alkylsulphates by evaporation of the solvent. The residue may include part of the sodium chloride and sodium carbonate present, the masses of which are determined and subtracted from the total mass of the residue of the extract.

NOTE. – It is very important that the solution remain alkaline throughout this determination.

2. REAGENTS

2.1 *Propan-2-ol,* 98 % solution rendered just alkaline to phenolphthalein by sodium hydroxide 0.1 N solution.

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

2.3 *Sulphuric acid,* 0.1 N standard volumetric solution in distilled water.

2.4 *Phenolphthalein,* 0.2 % solution in ethanol.

2.5 *Acetone,* analytical reagent grade. The residue on evaporation of the acetone must be less than 5 mg per 100 ml.

3. APPARATUS

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

(*a*) *pipette,* 100 ml;

(*b*) *water bath;*

(*c*) *glass beads;*

(*d*) *oven* regulated at 85 to 90 °C;

(*e*) *vacuum desiccator;*

(*f*) *two beakers,* 250 ml;

(*g*) *two round-bottomed flasks,* 250 ml;

(*h*) *analytical balance.*

4. PROCEDURE

4.1 Test portion

Using the aqueous alcoholic solution, L_1, remaining from the determination of matter extractable by hexane (see Part VI of this ISO Recommendation), and corresponding to the test portion, M_0, introduce by means of the 100 ml pipette an aliquot of 100 ml of the test portion into each of the two 250 ml beakers.

4.2 Determination

Treat the contents of each beaker as indicated below.

Reduce the volume to about 10 ml by evaporation on the water-bath using a current of air. Add 20 ml of propan-2-ol (2.1) and evaporate again to dryness. Then add 50 ml of propan-2-ol (2.1), break up the residue thoroughly with a glass stirrer and bring to the boil on the water bath. Allow to boil for 3 minutes and stir from time to time.

Allow the matter insoluble in the alcohol to settle and decant the hot supernatant liquor, filtering it through a filter paper into one of the 250 ml flasks, which has been previously tared, containing a few glass beads.

Place the flask on the boiling water bath so that the filtrate may evaporate while the solvent vapour is drawn off through a tube inserted into the neck of the flask.

Add 25 ml of propan-2-ol (2.1) to the beaker, bring to the boil and allow to boil gently for 2 minutes. Then allow to settle and filter the supernatant liquid through the same filter paper into the flask.

Repeat this extraction another two times, each time with 25 ml of propan-2-ol (2.1), transferring most of the insoluble matter to the filter paper with the last addition of propan-2-ol (2.1). Wash the beaker, the filter and its contents with hot propan-2-ol and pour through the filter, paying particular attention to the edges of the filter paper,which should not show any greasy marks.

Continue to evaporate the contents of the flask to dryness, with a current of air. Add 10 ml of acetone (2.5).

Evaporate the solvent. To do this, rotate the flask by hand in an inclined position, while blowing air through it. In this way the liquid in the flask spreads in a thin film over the walls of the flask, which facilitates the removal of the last traces of solvent.

Then put the flask in the oven at 90 °C for 5 minutes. Allow it to cool in the vacuum desiccator. Repeat the drying in the oven until constant weight is attained, i.e. so that two consecutive weighings made at an interval of 15 minutes do not differ by more than 0.005 g.

In this way two residues are obtained. One of them (R_1) can be used in the determination of the content of organic sulphur from the sulphate group (see Part VIII of this ISO Recommendation). Dissolve the other (R_2) in distilled water, with gentle heating if necessary, until dissolution is complete. Ensure that this solution is alkaline to phenolphthalein and determine the content of any sodium carbonate carried over, during the extraction, by means of sulphuric acid (2.3) using phenolphthalein as indicator (2.4). Then in the remaining aqueous liquor, determine the content of Cl^- ions, from any sodium chloride which may have been carried over, using the argentimetric method described in Part X of this ISO Recommendation.

5. EXPRESSION OF RESULTS

5.1 Method of calculation and formula

The percentage of sodium secondary alkylsulphates in the raw material is equal to

$$\frac{M_1+M_2}{2}\times\left(1-\frac{0.0106\,V_0+0.00585\,V_1}{M_2}\right)\times\frac{500}{100}\times\frac{100}{M_0}=\frac{M_1+M_2}{2}\times\left(1-\frac{0.0106\,V_0+0.00585\,V_1}{M_2}\right)\times\frac{500}{M_0}$$

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, (R_1), used for the determination of organic sulphur;

M_2 is the mass, in grammes, of the residue, (R_2), which was used for the determination of the content of sodium carbonate and Cl^- ions,

V_0 is the volume, in millilitres, of sulphuric acid (2.3) used for the determination of sodium carbonate content,

V_1 is the volume, in millilitres, of silver nitrate 0.1 N used for the determination of Cl^- ion content.

NOTE. – 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.

5.2 Reproducibility

The difference between results obtained on the same sample, in two different laboratories, should not exceed 1.2 g of sodium secondary alkylsulphates for 100 g of sample.

PART VIII

DETERMINATION OF THE ORGANIC SULPHUR CONTENT ARISING FROM THE SULPHATE GROUP

(Wurzschmitt method)

1. PRINCIPLE

Conversion of the organic sulphate from the sulphate group of the sodium alkylsulphates (isolated as described in Part VII of this ISO Recommendation) by oxidative ashing to sulphate in which is estimated gravimetrically as barium sulphate. The organic sulphur content gives a measure of the number of sulphate groups attached to the aliphatic chain.

The oxidative ashing is carried out in a nickel bomb (the Wurzschmitt method).

NOTE. – See Appendix Z in which is described a method for the determination of organic sulphur (sulphur from the sulphate group) by combustion in a current of air (the Grote-Krekeler method). This method is capable of giving additional analytical information.

2. REAGENTS

2.1 *Sodium peroxide.*

2.2 *Ethanediol.*

2.3 *Bromine.*

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

2.5 *Ammonia solution* (ρ_{20} = 0.925 g/ml), analytical reagent grade.

2.6 *Methyl orange*, 0.2 % solution in distilled water.

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

3. APPARATUS

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

(*a*) *squat-form beaker*, 600 ml;
(*b*) *bomb for sodium peroxide combustion*, * pure nickel, 22 ml capacity;
(*c*) *protective shield with micro-burner;*
(*d*) *analytical balance.*

4. PROCEDURE

4.1 Test portion

Weigh, to the nearest 0.1 mg, about 0.3 g of the sodium alkylsulphates, isolated as described in Part VII of this ISO Recommendation (residue R_1), into the nickel crucible of the bomb.

Let M'_0 be the mass weighed.

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* For example, the Parr bomb.

4.2 Determination

Add several drops (about 0.15 to 0.20 g) of ethanediol (2.2) to the test portion from a pipette, then cover with 6 to 8 g of sodium peroxide (2.1). Assemble the bomb and place it in the protective shield. Regulate the micro-burner so the flame just plays on the bottom of the bomb. Combustion starts in 10 to 20 seconds. Leave the micro-burner under the bomb for a further 50 seconds.

Allow the bomb to cool, then open it and wash the bomb and lid quantitatively with hot distilled water in the 600 ml beaker. Place the nickel crucible in the 600 ml beaker and cover with 230 ml of hot distilled water.

Withdraw the crucible when the contents have dissolved, rinsing it with distilled water.

Add 5 to 6 drops of bromine (2.3) to the solution. Boil for 10 minutes. Cool, acidify with hydrochloric acid (2.4) and boil until bromine vapour is no longer evolved.

Filter through paper into a second 600 ml beaker. Wash the filter paper quantitatively with distilled water.

Render the filtrate slightly alkaline with ammonia solution (2.5), then acidify the methyl orange (2.6) with hydrochloric acid (2.4) adding an excess of 1 ml.

Bring to the boil, then precipitate the $SO_4^{=}$ ion with barium chloride (2.7), determining it gravimetrically as barium sulphate.

5. EXPRESSION OF RESULTS

5.1 Method of calculation and formula

The percentage of sulphur (arising from the sulphate group) in the sodium alkylsulphates, isolated as described in Part VII of this ISO Recommendation is equal to

$$M_4 \times 0.1374 \times \frac{100}{M'_0\left(1 - \dfrac{0.0106\,V_0 + 0.00585\,V_1}{M_3}\right)} = \frac{13.74\,M_4}{M'_0\left(1 - \dfrac{0.0106\,V_0 + 0.00585\,V_1}{M_3}\right)}$$

where

M'_0 is the mass, in grammes, of the test portion (sodium alkylsulphates, residue R_1, see Part VII of this ISO Recommendation);

M_3 is the mass, in grammes, of residue R_2, obtained as described in Part VII of this ISO Recommendation;

M_4 is the mass, in grammes, of barium sulphate;

V_0 is the volume, in millilitres, of 0.1 N sulphuric acid used for the determination of sodium carbonate in the residue R_2 (see Part VII, section 5 of this ISO Recommendation);

V_1 is the volume, in millilitres, of 0.1 N silver nitrate used in the determination of the content of Cl^- ions (see Part VII, section 5 of this ISO Recommendation).

NOTE. – If the standard volumetric solution used is not exactly of strength 0.7 N, a suitable correction factor should be used in calculating the results.

5.2 Reproducibility

The difference between results obtained on the same sample, in two different laboratories, should not exceed 0.4 g of organic 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 raw material by means of propan-2-ol, followed by filtration. Dissolution of these salts in distilled water and determination of the content of SO_4^{--}ions by precipitation as barium sulphate.

2. REAGENTS

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

2.2 *Hydrochloric acid*, dilute (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*) *beaker*, 250 ml;

(*b*) *filter crucible* of sintered glass, porosity 5-15 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 raw material containing about 0.05 g of insoluble matter.

Let M_0 be the mass weighed.

4.2 Determination

Dilute 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 in 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 (2.4) with hydrochloric acid (2.2), taking care particularly at the start (because of foaming and splashing due to alkaline carbonates). Stir, bring to the boil and determine the content in SO_4^{--}ions 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".

PART X

DETERMINATION OF SODIUM CHLORIDE CONTENT

1. PRINCIPLE

Argentimetric determination of the content of Cl^- ions in an aqueous solution of the raw material by potentiometry.

2. REAGENTS

2.1 *Nitric acid* (ρ_{20} = 1.33 g/ml), analytical reagent grade.

2.2 *Silver nitrate,* 0.1 N standard volumetric solution in distilled water.

3. APPARATUS

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

(*a*) *beaker,* 300 ml;

(*b*) *graduated measuring cylinder,* 10 ml;

(*c*) *potentiometric titration apparatus* of good construction, with a silver working electrode and a mercury/mercurous sulphate reference electrode (with potassium sulphate solution as bridge);

NOTE. – If a reference electrode of this type is not available, a calomel electrode may be used instead, but it should be connected by a potassium nitrate/agar bridge to the beaker in which the titration is carried out and into which the silver working electrode is immersed.

A bridge of this kind can be made easily as follows : saturate 100 ml of distilled water with approximately 32 g of potassium nitrate of analytical reagent quality and then add 4 g of agar. Heat at 70 °C until solution is complete. Fill a U-shaped capillary tube of the following dimensions : internal diameter 2 to 3 mm, length of legs 8 to 10 cm, distance between legs about 6 cm, at about 70 °C with the above solution and then let it cool. During cooling keep the open ends of the legs in the solution.

(*d*) *magnetic stirrer;*

(*e*) *analytical balance.*

4. PROCEDURE

4.1 Test portion

Weigh, to the nearest 0.001 g, into the 300 ml beaker, about 2 g of the raw material.

Let M be the mass weighed.

4.2 Determination

Dilute the test portion with 150 ml of distilled water, add 10 ml of nitric acid (2.1), measured with the graduated measuring cylinder, and introduce the magnetic stirrer. Stir for a few seconds, then titrate with the silver nitrate solution (2.2).

The volume, in millilitres, of silver nitrate (2.2) used is deduced from the curve mV/ml, or from the differential curve.