

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 895

SURFACE ACTIVE AGENTS
TECHNICAL SODIUM SECONDARY ALKYL SULPHATES
METHODS OF ANALYSIS

1st EDITION
December 1968

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Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

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

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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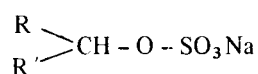
INTRODUCTION

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



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 (Wurzschnitt 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.

* 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

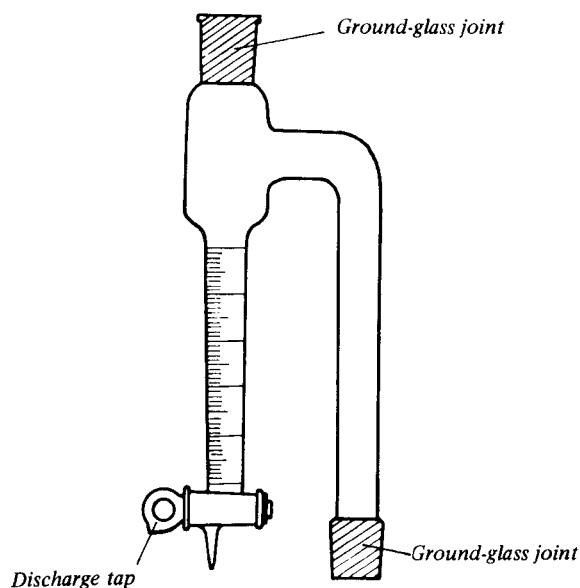
Xylene, commercial quality (any isomers or mixture of isomers in any proportion) saturated with water.

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.