

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

Revised

ISO RECOMMENDATION

R 894

SURFACE ACTIVE AGENTS
TECHNICAL SODIUM PRIMARY ALKYL SULPHATES
METHODS OF ANALYSIS

1st EDITION

December 1968

COPYRIGHT RESERVED

The copyright of ISO Recommendations and ISO Standards belongs to ISO Member Bodies. Reproduction of these documents, in any country, may be authorized therefore only by the national standards organization of that country, being a member of ISO.

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

Printed in Switzerland

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

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	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.

CONTENTS

	Page
Introduction	5
Part I — General	6
1. Scope	6
2. Field of application	6
3. Preparation of sample	6
4. General principle	7
Part II — Determination of water content	8
1. Karl Fischer method	8
2. Azeotropic method	9
Part III — Measurement of pH	11
Part IV — Determination of free alkalinity or free acidity	12
Part V — Determination of total alkalinity	13
Part VI — Determination of matter extractable by hexane	14
Part VII — Determination of matter extractable by diethyl ether after acid hydrolysis (combined technical fatty alcohols)	17
Part VIII — Determination of the combined sulphur content arising from the sodium alkylsulphate	20
Part IX — Determination of sodium sulphate content	21
Part X — Determination of sodium chloride content	23
Part XI — Report of the analysis	24
Appendix Y — General scheme of analysis	25
Appendix Z — Determination of water content (Karl Fischer method using two solutions)	26

SURFACE ACTIVE AGENTS
TECHNICAL SODIUM PRIMARY ALKYL SULPHATES
METHODS OF ANALYSIS

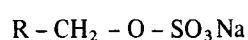
INTRODUCTION

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 unnecessary 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



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.

* 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

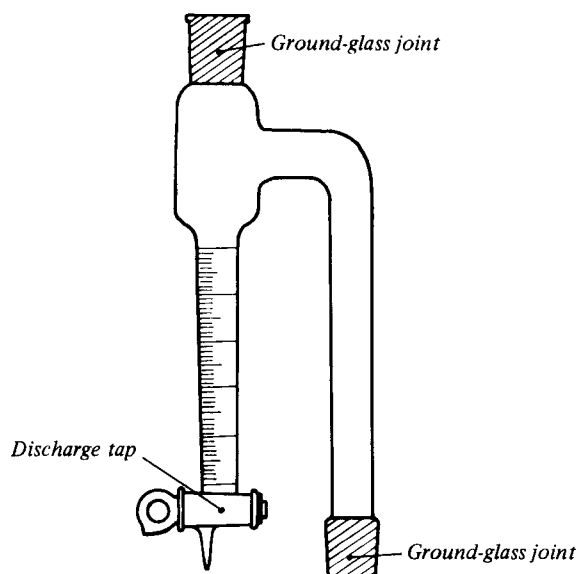
Xylene, commercial quality (any isomer or mixture of isomers in any proportion) saturated with water.

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 diagrammatically 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.