

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION MEXALHAPODHAS OPTAHUSALUS TO CTAHDAPTHSALUS ORGANISATION INTERNATIONALE DE NORMALISATION

Surface active agents - Detergents - Determination of anionic-active matter (direct two-phase titration procedure) 

First edition - 1972-09-01

UDC 661.185 : 543

Ref. No. ISO 2271-1972 (E)

Descriptors : surfactants, detergents, chemical analysis, determination of content, volumetric analysis, anions.

## FOREWORD

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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 2271 was drawn up by Technical Committee ISO/TC 91, *Surface active agents*.

It was approved in August 1971 by the Member Bodies of the following countries :

Austria Belgium Egypt, Arab Rep. of France Germany Hungary India Japan New Zealand Poland Portugal Romania South Africa, Rep. of Spain Sweden Switzerland Thailand Turkey United Kingdom U.S.S.R.

The Member Body of the following country expressed disapproval of the document :

U.S.A.

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Printed in Switzerland

# Surface active agents — Detergents — Determination of anionic-active matter (direct two-phase titration procedure)

## 1 SCOPE

This International Standard specifies a method for the determination of anionic-active matter present in detergents.

#### 2 FIELD OF APPLICATION

This method is applicable to the analysis of alkylbenzene sulphonates, alkyl sulphonates, sulphates and hydroxy-sulphates, alkylphenol and fatty alcohol ethoxysulphates and dialkylsulphosuccinates and to the determination of active materials containing one hydrophilic group per molecule.

NOTE – Low relative molar mass sulphonates present as hydrotropes (toluene, xylene) do not interfere when present in concentations up to 15 % (m/m) on active material. At higher levels, their influence must be evaluated in each particular case.

Soap, urea and ethylenediaminetetra-acetic acid salts do not interfere.

Typical inorganic components of detergent formulations such as sodium chloride, sulphate, borate, tripolyphosphate, perborate, silicate, etc., do not interfere but bleaching agents other than perborate should be destroyed before the analysis.

#### **3 REFERENCES**

ISO/R 385, Burettes.

ISO/R 648, One-mark pipettes.

ISO/R 1042, One-mark volumetric flasks.

#### **4 PRINCIPLE**

Determination of anionic-active matter in a medium consisting of an aqueous and a chloroform phase, by volumetric titration with a standard cationic-active solution (benzethonium chloride), in the presence of an indicator which consists of a mixture of a cationic dye (dimidium bromide) and an anionic dye (acid blue 1).

NOTE – The chemical process is as follows: the anionic-active matter forms a salt with the cationic dye which dissolves in the chloroform to give this layer a red-pink colour.

In the course of the titration, the benzethonium chloride displaces the dimidium bromide from this salt and the pink colour leaves the chloroform layer as the dye passes to the aqueous phase. The benzethonium chloride added in excess forms a salt with the anionic dye, which dissolves in the chloroform layer and colours it blue.

## **5 REAGENTS**

The water used shall be distilled water or water of at least equivalent purity.

The reagents used shall have the following properties :

5.1 Chloroform,  $\rho_{20} = 1.48$  g/ml, distilling between 59.5 and 61.5 °C.

5.2 Sulphuric acid, 5 N solution.

Carefully add 134 ml of sulphuric acid,  $\rho_{20} = 1.83$  g/ml, to 300 ml of water and dilute to 1 l.

5.3 Sulphuric acid, 1.0 N solution.

5.4 Sodium hydroxide, 1.0 N standard volumetric solution.

5.5 Sodium lauryl sulphate (sodium dodecyl sulphate) [CH<sub>3</sub> (CH<sub>2</sub>)<sub>11</sub> OSO<sub>3</sub>Na], 0.004 M standard volumetric solution.

Check the purity of the sodium lauryl sulphate and simultaneously prepare the standard solution.

5.5.1 Determination of purity of sodium lauryl sulphate

Weigh, to the nearest 1 mg,  $5 \pm 0.2$  g of the product into a 250 ml round bottom flask with ground glass neck. Add exactly 25 ml of the sulphuric acid solution (5.3) and reflux under a water condenser. During the first 5 to 10 min, the solution will thicken and tend to foam strongly; control this by removing the source of heat and swirling the contents of the flask.

In order to avoid excessive foaming, instead of refluxing, the solution may be left on a boiling water bath for 60 min.

After a further 10 min, the solution clarifies and foaming ceases. Reflux for a further 90 min.

Remove the source of heat, cool the flask and carefully rinse the condenser with 30 ml of ethanol followed by water.