INTERNATIONAL STANDARD 6121

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Surface active agents — Technical alkylsulphonates — Determination of alkylmonosulphonates content (Direct two-phase titration method)

Agents de surface — Alcanesulfonates techniques — Détermination de la teneur en alcanemonosulfonates (Méthode par titrage direct dans deux phases)

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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 6121 was developed by Technical Committee ISO/TC 91, *Surface active agents*, and was circulated to the member bodies in September 1977.

It has been approved by the member bodies of the following countries :

Australia	India	Romania
Austria	Iran	South Africa, Rep. of
Belgium	Italy	Spain
Brazil	Japan	Switzerland
Bulgaria	Kenya	Turkey
Chile	Mexico	United Kingdom
France	Netherlands	U.S.A.
Germany, F. R.	New Zealand	U.S.S.R.
Hungary	Poland	

No member body expressed disapproval of the document.

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Surface active agents — Technical alkylsulphonates — Determination of alkylmonosulphonates content (Direct two-phase titration method)

0 INTRODUCTION

The analysis based on selective light petroleum extraction of alkylmonosulphonates has been applied successfully in ISO 3206.

It has been considered desirable to standardize a direct two-phase titration procedure which is faster than that of ISO 3206 and which minimizes interference from disulphonates.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the alkylmonosulphonates content of technical alkylsulphonates containing small quantities of paraffins.

It is applicable to all alkali metal salts of the products of sulphochlorination and sulphoxidation of paraffins.

2 REFERENCES

ISO/R 385, *Burettes.*

ISO 607, *Surface active agents and detergents — Methods of sample division.*[1]

ISO 648, *Laboratory glassware — One-mark pipettes.*

ISO 1402, *Laboratory glassware — One-mark volumetric flasks.*

ISO 2271, *Surface active agents — Detergents — Determination of anionic active matter (Direct two-phase titration procedure).*

ISO 3206, *Surface active agents — Analysis of technical alkane sulphonates — Determination of alkane monosulphonates content.*

3 DEFINITION

For the purpose of this International Standard, the following definition applies :

alkylmonosulphonate : Alkali metal salt of the monosulphonic acids present in the technical products of sulphochlorination and sulphoxidation of pure straight-chain paraffins of which the chain consists of between 12 and 20 carbon atoms.

4 PRINCIPLE

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

NOTE — A description of the chemical process is given in ISO 2271.

5 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Chloroform, ρ_{20} 1,48 g/ml, distilling between 59,5 and 61,5 °C.

5.2 Sodium sulphate, anhydrous, 200 g/l solution.

5.3 Benzethonium chloride,[2] 0,004 mol/l standard volumetric solution[3].

Benzyl dimethyl-2-[2-4(1,1,3,3-tetramethylbutyl)phenoxyethoxy]ethyl ammonium chloride, monohydrate :

$$[(CH_3)_3\text{-}C\text{-}CH_2\text{-}C\text{-}(CH_3)_2\text{-}C_6H_4OCH_2\text{-}CH_2\text{-}OCH_2\text{-}CH_2\text{-}N\text{-}(CH_3)_2\text{-}CH_2\text{-}C_6H_5] + Cl^-.H_2O$$

5.3.1 *Preparation of the solution*

Weigh, to the nearest 0,001 g, between 1,75 and 1,85 g of benzethonium chloride and dissolve in water.

Transfer the solution quantitatively to a ground glass stoppered 1 litre one-mark volumetric flask and dilute to the mark with water.

NOTE — In order to prepare a 0,004 mol/l solution, dry the benzethonium chloride at 105 °C, allow to cool in a desiccator, weigh 1,792 g to the nearest 0,001 g, dissolve in water and dilute to 1 litre.

1) At present at the stage of draft. (Revision of ISO/R 607.)

2) One of the commercial products is also known under the name of "Hyamine 1622".

3) Previously expressed as 0,004 M standard volumetric solution.

5.3.2 *Standardization of the solution*

By means of a pipette, transfer 25 ml of a 0,004 mol/l standard volumetric sodium lauryl sulphate solution to a bottle or measuring cylinder of 100 ml capacity, add 10 ml of water, 15 ml of the chloroform (5.1) and 10 ml of the mixed indicator solution (5.4).

Titrate with the benzethonium chloride solution (5.3.1); stopper the bottle or measuring cylinder after each addition and shake well. The lower layer will be coloured pink. Continue the titration, with repeated vigorous shaking. As the end point approaches, the emulsions formed during shaking tend to break easily. Continue the titration drop by drop, shaking after each addition of titrant, until the end point is reached. This is at the moment when the pink colour is completely discharged from the chloroform layer, which becomes a faint greyish blue.

The molecular concentration, c, of the benzethonium chloride solution is given by the formula

$$c = \frac{c_0 \times 25}{V_0}$$

where

c_0 is the exact molecular concentration, in moles per litre, of the sodium lauryl sulphate solution;

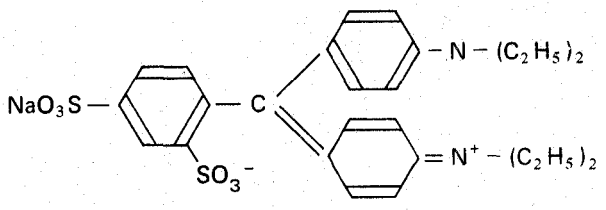
V_0 is the volume, in millilitres, of the benzethonium chloride solution used for the titration.

5.4 Mixed indicator solution.[1)]

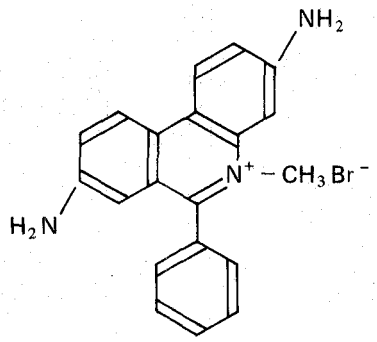
5.4.1 *Stock solution*

This solution shall be prepared from acid blue 1 and dimidium bromide.

5.4.1.1 Acid blue 1[2)] (Colour Index 42045) (*di*sodium-4′, 4″-dinitrilodiethyltriphenylmethane-2,4-disulphonate) :



5.4.1.2 Dimidium bromide (3,8-diamino-5-methyl-6-phenylphenanthridinium bromide) :



5.4.1.3 PREPARATION OF THE STOCK SOLUTION

Weigh, to the nearest 0,001 g, 0,5 ± 0,005 g of the dimidium bromide (5.4.1.2) into a 50 ml beaker, and 0,25 ± 0,005 g of the acid blue 1 (5.4.1.1) into a second 50 ml beaker.

Add between 20 and 30 ml of hot 10 % (*V/V*) ethanol to each beaker.

Stir until dissolved and transfer the solutions to a 250 ml one-mark volumetric flask. Rinse the beakers into the volumetric flask with the ethanol and dilute to the mark with the ethanol.

5.4.2 *Acid solution*

Add 200 ml of water to 20 ml of the stock solution (5.4.1) in a 500 ml one-mark volumetric flask. Add 20 ml of a 5 N sulphuric acid solution, mix and dilute to the mark with water. Store in the dark.

6 APPARATUS

Ordinary laboratory apparatus and :

6.1 One-mark volumetric flask, 250 ml capacity, complying with the requirements of ISO 1042.

6.2 Measuring cylinder or flask, 100 ml capacity, with ground glass stopper.

6.3 One-mark pipettes, 20 ml capacity, complying with requirements of class A of ISO 648.

6.4 Burette, 25 ml capacity, complying with the requirements of class A of ISO/R 385.

1) This mixed indicator is available commercially in the form of a basic solution, which should be acidified and diluted before use.

2) This material is known commercially by, for example, the names "Erioglaucine", or "Disulphine blue VN150".

7 SAMPLING

The laboratory sample shall be prepared and stored according to the instructions given in ISO 607.

8 PROCEDURE

WARNING — Comply with safety regulations relating to the handling of toxic solvents.

8.1 Test portion

Weigh, to the nearest 0,001 g, into a 150 ml beaker, a test portion which contains about 0,4 to 0,5 g of alkylmonosulphonate.

8.2 Determination

Dissolve the test portion (8.1) in 50 ml of water and transfer the solution quantitatively to the one-mark volumetric flask (6.1). Add the washing solutions, and dilute to the mark with water.

Mix thoroughly and then, by means of one of the pipettes (6.3), transfer 20,0 ml of this solution to the measuring cylinder or flask (6.2). Add successively, by means of measuring cylinders, 10 ml of the sodium sulphate solution (5.2), 10 ml of the mixed indicator solution (5.4), and 15 ml of the chloroform (5.1). Stopper the measuring cylinder or flask and shake well.

Titrate with the benzethonium chloride solution (5.3) contained in the burette (6.4), at first by successive additions of 2 to 3 ml. After each addition stopper the measuring cylinder or flask and shake well.

The lower layer of chloroform will be coloured pink. Continue the titration. As the end point approaches, the emulsions formed during shaking tend to break easily and the coloration of the chloroform layer becomes fainter.

From this point continue the titration by additions of 0,5 ml, then drop by drop and shaking after each addition of titrant, until the end point is reached. This is at the moment when the pink colour is completely discharged from the chloroform layer, which turns a faint greyish blue.

9 EXPRESSION OF RESULTS

9.1 Calculation

The alkylmonosulphonates content, expressed as a percentage by mass, is given by the formula

$$\frac{V \times c \times M_r \times 1{,}25}{m_0}$$

where

V is the volume, in millilitres, of the benzethonium chloride solution (5.3) used for the titration of a 20 ml aliquot portion of alkylsulphonate solution;

c is the exact molecular concentration, in moles per litre, of the benzethonium chloride solution (5.3);

M_r is the relative molecular mass of the alkylmonosulphonate;

m_0 is the mass, in grams, of the test portion (8.1).

9.2 Precision

Comparative analyses on a sample in the form of a homogeneous aqueous solution containing about 25 % (m/m) of total soluble matter comprising alkylmono-, di- and poly-sulphonates in the form of their sodium salts, sodium sulphate and small quantities of paraffins, carried out in 15 laboratories, have given the following statistical results :

- mean (percentage by mass) : 23,05
- standard deviation of repeatability (σ_r) : 0,55
- standard deviation of reproducibility (σ_R) : 1,57

10 TEST REPORT

The test report shall contain the following information :

a) all information necessary for the complete identification of the sample;

b) the reference of the method used (reference to this International Standard);

c) the results and the method of expression used;

d) the test conditions;

e) any operational details not specified in this International Standard or the International Standards to which reference is made, or regarded as optional, as well as any incidents likely to have affected the results.