INTERNATIONAL STANDARD

ISO
6121

Second edition
1988-06-01

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION
ORGANISATION INTERNATIONALE DE NORMALISATION
МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Surface active agents — Technical alkane sulfonates — Determination of alkane monosulfonates content by direct two-phase titration

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

Reference number
ISO 6121 : 1988 (E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commision (IEC) on all matters of electrotechnical standardization.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 6121 was prepared by Technical Committee ISO/TC 91, *Surface active agents*.

This second edition cancels and replaces the first edition (ISO 6121 : 1979), of which it constitutes a minor revision.

Printed in Switzerland

Introduction

The analytical method based on selective solvent extraction which has been applied successfully for the analysis of technical sodium alkane sulfonates in ISO 893 leads to only approximate results during the analytical examination of the products of sulfochlorination and sulfoxidation of paraffins, owing to the presence of alkane disulfonates, in addition to alkane monosulfonates, formed during the synthesis. However, because of the different properties and applications of mixtures with different relative proportions of mono- and disulfonates, the knowledge of the contents of such mixtures is of paramount importance.

Therefore, it seemed advisable to establish this method to allow the determination specifically of the alkane monosulfonates content. The method is applicable to all synthetic alkane sulfonates and minimizes interference due to disulfonates.

This page intentionally left blank

Surface active agents — Technical alkane sulfonates — Determination of alkane monosulfonates content by direct two-phase titration

1 Scope

This International Standard specifies a method for the determination of the alkane monosulfonates content of technical alkane sulfonates containing small quantities of paraffins.

It is applicable to all alkali metal salts of the products of sulfochlorination and sulfoxidation of paraffins.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1 : 1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 607 : 1980, *Surface active agents and detergents — Methods of sample division.*

ISO 648 : 1977, *Laboratory glassware — One-mark pipettes.*

ISO 1042 : 1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 2271 : 1972, *Surface active agents — Detergents — Determination of anionic-active matter (Direct two-phase titration procedure).*

ISO 6845 : 1982, *Surface active agents — Technical alkane sulfonates — Determination of mean relative molecular mass of alkane monosulfonates.*

3 Definition

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

alkane monosulfonate: Alkali metal salt of the monosulfonic acids present in the technical products of sulfochlorination and sulfoxidation of pure straight-chain paraffins of which the chain consists of between 12 and 20 carbon atoms.

4 Principle

Determination of alkane monosulfonates content in a medium consisting of an aqueous phase and a chloroform phase, in the presence of sodium sulfate, by titration against 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 sulfate, anhydrous, 200 g/l solution.

5.3 Benzethonium chloride[1], standard volumetric solution, $c(C_{27}H_{42}ClNO_2) = 0{,}004$ mol/l.

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

$$[(CH_3)_3\text{-}C\text{-}CH_2\text{-}C\text{-}(CH_3)_2\text{-}C_6H_4OCH_2\text{-}CH_2OCH_2\text{-}CH_2\text{-}N\text{-}(CH_3)_2\text{-}CH_2\text{-}C_6H_5]^+ Cl^- . H_2O$$

1) Hyamine 1622 is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

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 1 000 ml one-mark volumetric flask fitted with a ground glass stopper, and make up 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 1 000 ml.

5.3.2 Standardization of the solution

By means of a pipette, transfer 25 ml of a standard volumetric sodium lauryl sulfate solution, $c(C_{12}H_{25}NaO_4S) = 0{,}004$ mol/l, to a bottle or measuring cylinder of 100 ml capacity, and add 10 ml of water, 15 ml of chloroform (5.1) and 10 ml of the mixed indicator solution (5.4).

Titrate against the benzethonium chloride solution (5.3); 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 emulsion formed during shaking tends 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.

5.3.3 Calculation of the concentration

The exact concentration, c, of the benzethonium chloride solution, expressed in moles of $C_{27}H_{42}ClNO_2$ per litre, is given by the formula

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

where

c_0 is the exact concentration, in moles of $C_{12}H_{25}NaO_4S$ per litre, of the sodium lauryl sulfate solution used for the titration in 5.3.2;

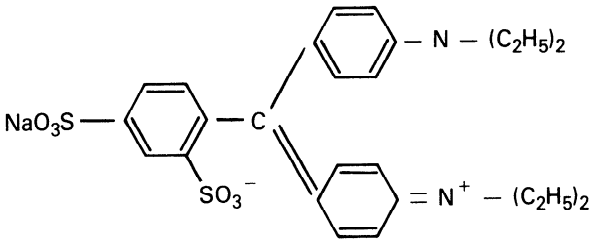
V_0 is the volume, in millilitres, of the benzethonium chloride solution used for the titration in 5.3.2.

5.4 Mixed indicator solution.[1]

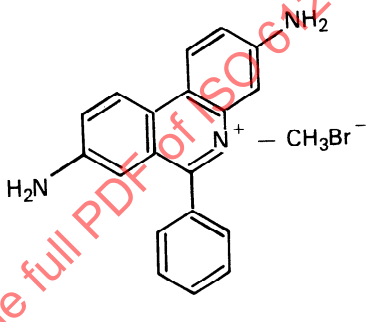
5.4.1 Stock solution

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

5.4.1.1 Acid blue 1[2] (Colour Index 42045) (disodium-4', 4''-dinitrilodiethyltriphenylmethane-2,4-disulfonate):



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 g ± 0,005 g of dimidium bromide (5.4.1.2) into a 50 ml beaker, and 0,25 g ± 0,005 g of 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 sulfuric acid solution (245 g/l), 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.

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

2) Acid blue 1, VS blue and disulfine blue VN150 are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.