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SURFACE ACTIVE AGENTS - FATTY ALKYL DIMETHYL AMINE OXIDES - DETERMINATION OF THE AMINE OXIDE CONTENT

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Contents

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 276 "Surface active agents", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 1997, and conflicting national standards shall be withdrawn at the latest by August 1997.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Annexes A and B are informative.

1 Scope

This draft European Standard specifies a method for the determination of up to approximately 2 milli-equivalents of tertiary amine oxide. Components which lose their basicity in reaction with acetic anhydride by forming an amide (e.g. primary and secondary amines) interfere. If present, these primary and secondary amines should be determined by a different procedure e.g. reaction with carbonsulfide.

The method is applicable to solids or to aqueous solutions of the active material. The molecular mass of the amine oxide shall be known if its content is expressed as a percentage of mass.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

ISO 607	1980	Surface active agents and detergents - Methods of sample division
ISO 1042	1983	Laboratory glassware - One-mark volumetric flasks

3 Principle

This draft European Standard is based on the Polonovski reaction in which the fatty alkyldimethylamine oxide reacts with acetic anhydride to form a N,N-disubstituted acetamide and an aldehyde (see equation 1). Free tertiary amine, residual raw material of technical amine oxide, remains unreacted and after dissolution of the sample in mixture of acetic acid and acetic anhydride 2:1 (as a volume fraction) is titrated with a perchloric acid solution (see equation 2). In this medium the acetamide is not titrated.

In a second titration the sum of amine oxide and tertiary amine is determined by potentiometric titration in acetic acid medium with a perchloric acid solution (see equation 3).

$$R-N(CH_3)_20 \ + \ (CH_3CO)_20 \rightarrow R-N(CH_3)COCH_3 + HCHO + CH_3COOH \qquad ...(1)$$

$$R-N(CH_3)_2 + HClO_4 \rightarrow R-NH(CH_3)_2^+Cl0_4^- \qquad ...(2)$$

$$R-N(CH_3)_20 + R-N(CH_3)_2 + 2HCl0_4 \rightarrow R\text{-}N\ (CH_3)_20H^+ClO_4^- + R-NH(CH_3)_2^+Cl0_4^- \qquad ...(3)$$

The difference between these two titrations (3 - 2) gives the amine oxide content.

4 Reagents

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

4.1 Perchloric acid standard volumetric solution of known titre, c = 0,1 mol/l in acetic acid.

4.2 Acetic acid, glacial (purity 99 % as a mass fraction (minimum), density 1,05 g/ml at 20 °C).

4.3 Acetic anhydride (purity 99 % as a mass fraction (minimum), density 1,08 g/ml at 20 °C).

5 Apparatus

Ordinary laboratory apparatus and the following :

5.1 Recording potentiometer equipped with a 20 ml plunger burette and stirrer.

NOTE : An example of instrument settings is given in annex A.

5.2 Combined glass electrode.

5.3 Magnetic stirrer with electrically heated hot plate, capable of maintaining a temperature of (100 ± 5) °C.

5.4 Beaker, 150 ml capacity.

5.5 Measuring cylinder, 100 ml capacity.

5.6 Conical flask, 100 ml capacity.

5.7 Reflux condenser.

5.8 Analytical balance.

6 Sampling

The laboratory sample of the detergent shall be prepared and stored in accordance with instructions given in ISO 607.