BRITISH STANDARD

BS EN
1791 : 1997

Surface active agents — Fatty alkyl dimethyl amine oxides — Determination of the amine oxide content

The European Standard EN 1791 : 1997 has the status of a British Standard

ICS 71.100.40

Committees responsible for this British Standard

The preparation of this British Standard was entrusted to Technical Committee CII/34, Methods of test for surface active agents, upon which the following bodies were represented:

British Association for Chemical Specialities
Chemical Industries Association
Chemical Industries Association (GOSIP)
Laboratory of the Government Chemist
Ministry of Defence (DRA)
Soap and Detergent Industry Association

This British Standard, having been prepared under the direction of the Sector Board for Materials and Chemicals, was published under the authority of the Standards Board and comes into effect on
15 July 1997

Amendments issued since publication

Amd. No.	Date	Text affected

The following BSI references relate to the work on this standard:
Committee reference CII/34
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Contents

National foreword

This British Standard has been prepared by Technical Committee CII/34, and is the English language version of EN 1791 : 1997, *Surface active agents — Fatty alkyl dimethyl amine oxides — Determinaiton of the amine oxide content*, published by the European Committee for Standardization (CEN).

Cross-references

Publication referred to	Corresponding British Standard
ISO 607 : 1980	BS 3762 *Analysis of formulated detergents* Part 1 : 1983 *Methods of sample division*
ISO 1042 : 1983	BS 1792 : 1982 *Specification for one-mark volumetric flasks*

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN title page, pages 2 to 6, an inside back cover and a back cover.

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 1791

February 1997

ICS 71.100.40

Descriptors: Surfactants, chemical analysis, determination of content, oxides, tertiary amines, titration, potentiometric methods

English version

Surface active agents — Fatty alkyl dimethyl amine oxides — Determination of the amine oxide content

Agents de surface — Oxydes d'alkyldiméthylamine grasses — Détermination de la teneur en oxyde d'amine

Grenzflächenaktive Stoffe — Fettalkyldimethylaminoxide — Bestimmung des Aminoxidgehalts

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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

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.

Contents

1 Scope

This 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 carbon sulfide.

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 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).

$$R - N(CH_3)_2 0 + (CH_3CO)_2 0 \rightarrow R - N(CH_3)COCH_3 + HCHO + CH_3COOH \quad (1)$$

$$R - N(CH_3)_2 + HClO_4 \rightarrow R - NH(CH_3)_2^+ ClO_4^- \quad (2)$$

$$R - N(CH_3)_2 0 + R - N(CH_3)_2 + 2HClO_4 \rightarrow R - N(CH_3)_2\ 0H^+ ClO_4^- + R - NH(CH_3)_2^+ ClO_4^- \quad (3)$$

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.

7 Procedure

7.1 Titration of the sum : Amine oxide and tertiary amine

Weigh (m_1), to the nearest 0,1 mg, a portion of the laboratory sample containing approximately 1,5 milli-equivalent of amine oxide into a 150 ml beaker (see **5.4**).

Add 50 ml acetic acid (see **4.2**) with a measuring cylinder (see **5.5**) and dissolve the sample.

Place the beaker on the potentiometer (see **5.1**) and insert a stirring bar.

Insert the combined glass electrode (see **5.2**) and start stirring.

Titrate with the perchloric acid standard volumetric solution (see **4.1**) until slightly beyond the potential jump and record the volume of perchloric acid solution necessary to reach equivalent point (V_1) (see annex B).

7.2 Polonovski reaction and titration of tertiary amine

Weigh (m_2), approximately 5 g to the nearest 10 mg of the laboratory sample into a conical flask (see **5.6**).

Add 25 ml of acetic anhydride (see **4.3**) with a measuring cylinder and dissolve the sample.

Insert a stirring bar, place the flask on the hot plate (see **5.3**) and connect the reflux condenser (see **5.7**).

Heat to the boiling point and maintain a moderate reflux for 10 min while stirring.

Cool to room temperature and add 50 ml of acetic acid.

Place the flask on the potentiometer (see **5.1**).

Insert the combined glass electrode (see **5.2**) and start stirring.

Titrate with the perchloric acid standard volumetric solution (see **4.1**) until slightly beyond the potential jump and record the volume of perchloric acid solution necessary to reach the equivalent point (V_2) (see annex B).

8 Expression of results

8.1 Calculation

The amine oxide content (A) expressed as a percentage by mass is given by the following equation :

$$A = \left(\frac{V_1}{m_1} - \frac{V_2}{m_2}\right) \times C \times \frac{M \times 100}{1000} \% \qquad (4)$$

where:

V_1 is the volume of the perchloric acid standard volumetric solution (see **4.1**) necessary to reach the equivalence point in **7.1**, in millilitres;

V_2 is the volume of the perchloric acid standard volumetric solution necessary to reach the equivalence point in **7.2**, in millilitres;

m_1 is the mass of the laboratory sample, weighed in **7.1**, in grams;

m_2 is the mass of the laboratory sample, weighed in **7.2**, in grams;

C is the concentration of the perchloric acid standard volumetric solution;

M is the mean molecular mass of the amine oxide.

Express the result to one decimal place.

8.2 Precision

8.2.1 *Repeatability*

0,8 % at a level of approximately 31 % tertiary amine oxide.

In the normal and correct operation of the method, the difference between two individual results obtained within the shortest feasible time interval by the same operator using the same apparatus and identical test material will exceed the repeatability value on average not more than once in 20 cases.

8.2.2 *Reproducibility*

0,9 % at a level of approximately 31 % tertiary amine oxide.

In the normal and correct operation of the method, individual results obtained by two different laboratories using identical test material will differ by more than reproducibility value on average not more than once in 20 cases.

9 Test report

The test report shall include the following particulars:

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

b) the method used (a reference to this European Standard);

c) the results obtained and the way in which they have been expressed;

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

Annex A (informative)
Instrument settings

Data given are settings for the Metrohm Potentiograph type E 536 and are intended to act as guide only :

Stop % U	off
Autocontrol	10
Min/100 % vol	5
mV x 100	
mV scale	500
Recording	mV/pH
mV/pH	100 %
Temperature	25 °C
Scan speed	400 mm/100 % vol.

Annex B (informative)

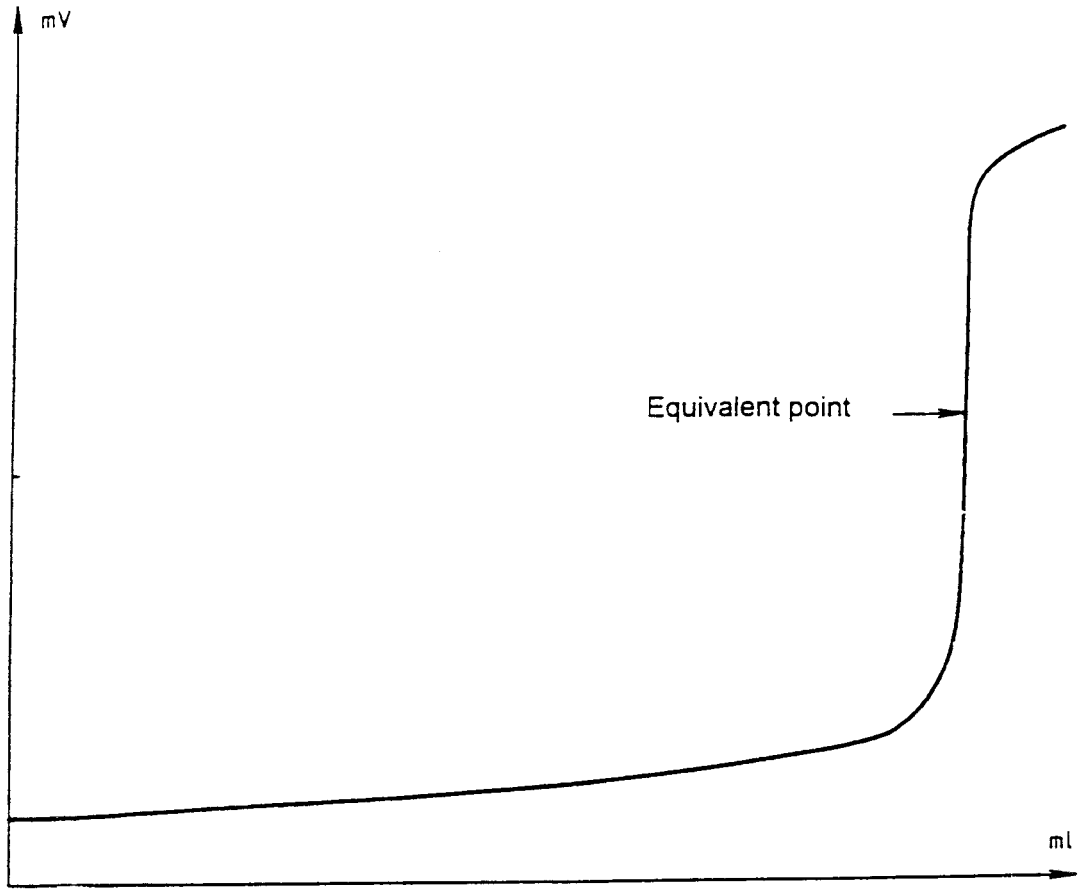

Figure B.1 Titration of the sum: Amine oxide and tertiary amine (see **7.1**)

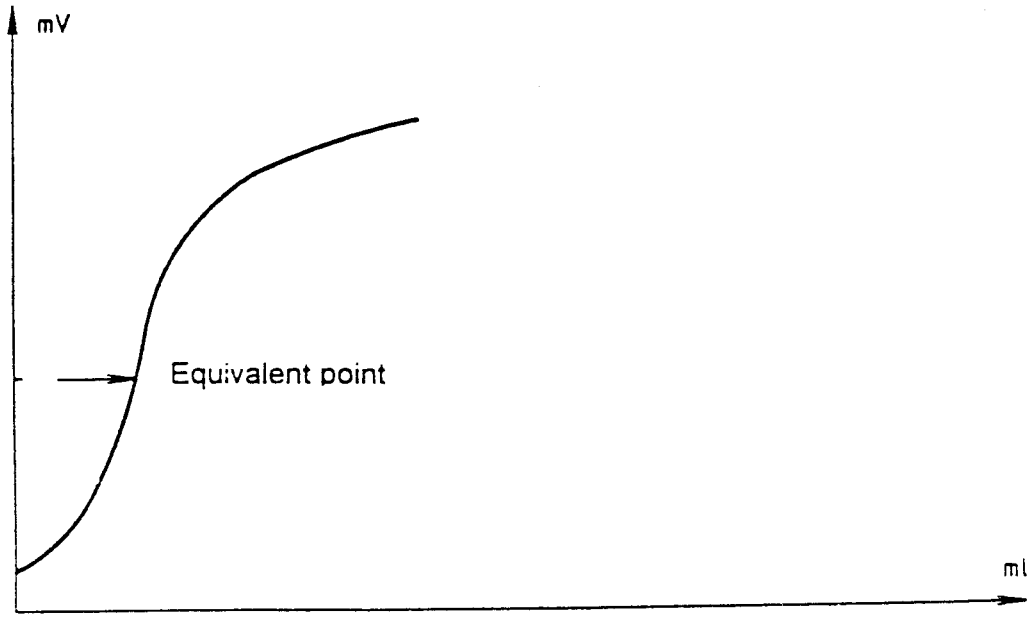

Figure B.2 Titration of tertiary amine after Polonovski reaction (see **7.2**)

List of references

See national foreword.

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