BRITISH STANDARD

BS EN ISO
4320:1998

Non-ionic surface active agents — Determination of cloud point index — Volumetric method —

(ISO 4320:1977, including Erratum: 1980)

The European Standard EN ISO 4320:1997 has the status of a British Standard

ICS 71.100.40

Confirmed
June 2009

National foreword

This British Standard is the English language version of EN ISO 4320:1997. It is derived from ISO 4320:1977, incorporating Erratum: 1980. It supersedes BS 6829-4.4:1989 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CII/34, Methods of test for surface active agents, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

Cross-references

Attention is drawn to the fact that CEN and CENELEC standards normally include an annex which lists normative references to international publications with their corresponding European publications. The British Standards which implement these international or European publications may be found in the BSI Standards Catalogue under the section entitled "International Standards Correspondence Index", or by using the "Find" facility of the BSI Standards Electronic Catalogue.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

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 ISO title page, page 2, the ISO title page, page ii, pages 1 and 2 and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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 March 1998

ISBN 0 580 29123 5

Amendments issued since publication

Amd. No.	Date	Comments

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 4320

May 1997

ICS 71.100.40

Descriptors: See ISO document

English version

Non-ionic surface active agents — Determination of cloud point index — Volumetric method

(ISO 4320:1977, Including Erratum:1980)

Agents de surface non ioniques — Détermination de l'indice de trouble — Méthode volumétrique
(ISO 4320:1977, Erratum: 1980 inclus)

Nichtionische Tenside — Bestimmung der Trübungstitrationszahl — Volumetrisches Verfahren
(ISO 4320:1977, einschließlich Erratum: 1980)

This European Standard was approved by CEN on 1997-04-06. CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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CEN members are the national standards bodies of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

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

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

Foreword

The text of the International Standard from Technical Committee ISO/TC 91 "Surface active agents" of the International Organization for Standardization (ISO) has been taken over as an European Standard 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 month of November 1997, and conflicting national standards shall be withdrawn at the latest by November 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.

Endorsement notice

The text of the International Standard ISO 4320:1977, Including Erratum: 1980 has been approved by CEN as a European Standard without any modification.

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INTERNATIONAL STANDARD ISO 4320

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

Non-ionic surface active agents — Determination of cloud point index — Volumetric method

Agents de surface non ioniques — Détermination de l'indice de trouble — Méthode volumétrique

First edition — 1977-02-01

UDC 661.185 : 543 **Ref. No. ISO 4320-1977 (E)**

Descriptors : surfactants, non-ionic surfactants, tests, determination, cloud point, volumetric analysis.

Price based on 3 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

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 4320 was drawn up by Technical Committee ISO/TC 91, *Surface active agents*, and was circulated to the member bodies in October 1975.

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

Austria	Iran	Portugal
Belgium	Italy	Romania
Brazil	Japan	South Africa, Rep. of
Canada	Korea, Dem. P. Rep. of	Spain
France	Mexico	Switzerland
Germany	Netherlands	Turkey
Hungary	New Zealand	United Kingdom
India	Poland	U.S.A.

No member body expressed disapproval of the document.

Descriptors: Surfactants, non-ionic surfactants, tests, determination, cloud point, volumetric analysis.

0 Introduction

Determination of the cloud point index provides a way of characterizing weakly alkoxylated derivatives and their hydrocarbon hydrophobic groups.

This simple and rapid measurement is a valuable means for checking the level of alkoxylation of non-ionic derivatives intended, for example, for sulphonation.

1 Scope

This International Standard specifies a volumetric method for the determination of the cloud point index of non-ionic surface active agents.

2 Field of application

The method is applicable to weakly alkoxylated non-ionic surface active agents (1 to 5 oxyethylene groups) the hydrophobic group of which is provided by an alcohol, an alkylphenol or a fatty acid (provided that the last has a melting point lower than 30 °C), subject to the product being soluble in propan-1-ol to the extent of 1 g in 10 ml at 30 °C.

It is equally applicable to lipophilic bases derived from alcohols, alkylphenols and fatty acids.

3 Reference

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

4 Definition

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

cloud point index

the number of millilitres of distilled water necessary to render cloudy, at a specified temperature, a solution containing a given mass of surface active agent in a given volume of solvent

5 Principle

Addition, at 30 °C, of distilled water to a propanol solution of the surface active agent until the appearance of cloudiness.

6 Reagents

6.1 *Distilled water.*

6.2 *Propan-1-ol*, complying with the following requirements:

— assay (by gas chromatography) 99 % (*m*/*m*);

— density ρ_{20} 0,804 to 0,805 g/ml;

— refractive index n_D^{20} 1,384 to 1,385;

— non-volatile matter < 0,005 % (*m*/*m*);

— free acidity (expressed as C_2H_5COOH) < 0,01 % (*m*/*m*);

— water (determined by the Karl Fischer method; see ISO 760) < 0,2 % (*m*/*m*).

7 Apparatus

Ordinary laboratory apparatus, and in particular:

7.1 *Beaker* (see the figure) of height 80 mm and diameter 50 mm, with double walls permitting temperature stability, and having a mass less than 200 g, fitted with a polyethylene, polytetrafluoroethylene or an aluminium foil cover pierced with two holes allowing entry of the thermometer (**7.2**) and the burette (**7.4**).

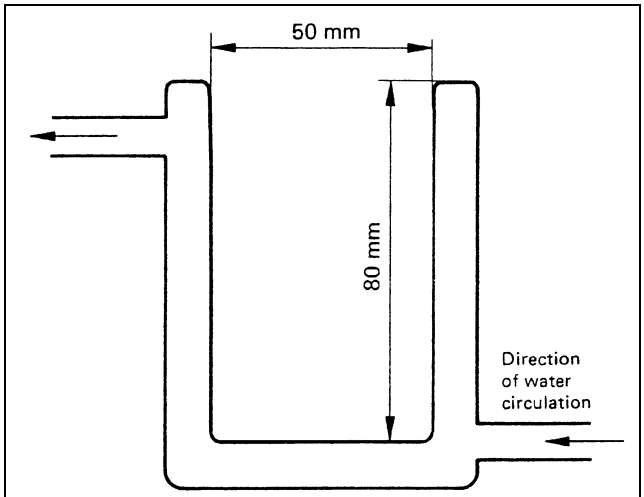

Figure — Double-walled beaker

7.2 *Thermometer,* STC/0,1/29/41, complying with the requirements of ISO/R 654.

7.3 *One-mark pipette*, capacity 10 ml, complying with the requirements of ISO 648.

7.4 *Burette,* capacity 50 ml, complying with the requirements of class A of ISO 385.

7.5 *Magnetic stirrer.*

7.6 *Bar magnet*, coated with polytetrafluoroethylene.

7.7 *Water bath,* with circulation, capable of being controlled to within ± 0,1 °C.

8 Sampling

The laboratory sample of the non-ionic surface active agent shall be prepared and stored according to the instructions in ISO 607.

[1] In preparation, (Revision of ISO/R 607.)

9 Procedure

9.1 Test portion

Weigh 1,0 ± 0,1 g of the laboratory sample into the beaker (**7.1**), previously weighed to the nearest 0,01 g.

9.2 Determination

Add 10 ml of the propan-1-ol (**6.2**) to the beaker (**7.1**) containing the test portion (**9.1**). Introduce the bar magnet (**7.6**), place the beaker fitted with its cover on the magnetic stirrer (**7.5**) and insert the thermometer (**7.2**).

Fit the beaker (**7.1**) onto the water bath (**7.7**) and control the temperature at 30,0 ± 0,1 °C.

Start the water circulation and the stirrer (stir gently at first in order to avoid splashing the liquid on the walls of the beaker).

Ensure that the test portion is completely dissolved (the solution should be clear) and add, drop by drop, the water (**6.1**) from the burette (**7.4**) until the liquid remains cloudy.

Check that the temperature in the beaker is maintained at 30,0 ± 0,5 °C during the complete operation.

The results of the test depend upon the speed of introduction of the water. Hence, the period during which it is introduced should be between 20 and 30 min, according to the quantity of water introduced.

Immediately after the cloud point is reached, allow the solution to equilibrate for 5 min so as to verify that the turbidity does not disappear.

10 Expression of results

10.1 Method of calculation

The cloud point index of the product is expressed as the volume, in millilitres, of water added by the procedure specified in clause **9** to render opaque the solution of non-ionic surface active agent.

Take as the result the mean of at least three determinations, expressed to the nearest 0,05 ml (1 drop of water).

10.2 Repeatability

The maximum difference between the results of two determinations carried out in rapid succession on the same sample, by the same analyst using the same apparatus, should not exceed 2 % of the mean volume found.

10.3 Reproducibility

The difference between results obtained on the same sample in two different laboratories should not exceed 5 % of the mean volume found.

11 Test report

The test report shall include the following particulars:

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

b) the reference to the method used;

c) the results obtained and the form in which they are expressed;

d) the conditions of test:

— the temperature of the water bath,

— the temperature of the solution of the product at the moment the turbidity appears,

— the time taken to dissolve the product,

— the exact length of time during which distilled water is introduced,

— the length of the determination,

— the nature of the change from clear to cloudy, namely if the change is sharp or if there is a state of opalescence before cloudiness,

— the mass of the test portion;

e) any operation not included in this International Standard or regarded as optional, as well as any incidents which may have affected the results.

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