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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

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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 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.

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

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).

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

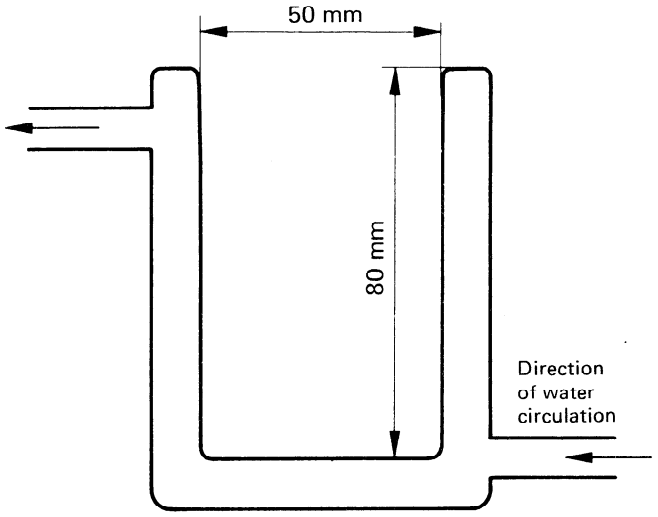

FIGURE — **Double-walled beaker**

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.

9 PROCEDURE

9.1 Test portion

Weigh, to the nearest 0,001 g, 1,0 ± 0,1 g of the laboratory sample into the beaker (7.1), previously weighed to the nearest 0,001 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 introduced to render cloudy 10 ml of a 100 g/l solution of non-ionic surface active agent in propan-1-ol.

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;

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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