INTERNATIONAL STANDARD

ISO 2270

Second edition
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Non-ionic surface active agents — Polyethoxylated derivatives — Iodometric determination of oxyethylene groups

Agents de surface non ioniques — Dérivés polyéthoxylés — Dosage iodométrique des groupes oxyéthylène

Reference number
ISO 2270 : 1989 (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 Commission (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 2270 was prepared by Technical Committee ISO/TC 91, *Surface active agents.*

This second edition cancels and replaces the first edition (ISO 2270 : 1972), of which it constitutes a minor revision.

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Non-ionic surface active agents — Polyethoxylated derivatives — Iodometric determination of oxyethylene groups

1 Scope

This International Standard specifies a method for the iodometric determination of oxyethylene groups in polyethoxylated non-ionic surface active agents.

The method is applicable to the analysis of polyethoxylated derivatives of

- primary saturated fatty alcohols;
- oleyl alcohol;
- saturated fatty acids;
- straight and branched chain alkylphenols.

It is also applicable in the presence of unreacted alcohols, fatty acids or alkylphenols of the types mentioned above.

The method is not applicable in the presence of

- compounds containing sulfur or nitrogen;
- compounds containing oxygen or halogen atoms on adjacent carbon atoms other than as oxyethylene groups, for example compounds containing oxypropylene groups;
- aldehydes or acetals;
- sterols and derivatives.

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 383 : 1976, *Laboratory glassware — Interchangeable conical ground glass joints.*

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

3 Principle

Hydrolysis of the oxyethylene groups by nascent hydriodic acid.

Liberation of the iodine and titration with a standard volumetric sodium thiosulfate solution.

4 Reactions

The basic reaction is the conversion of each (CH_2-CH_2O) group to ethylene di-iodide (ICH_2-CH_2I), in accordance with the equation

$$RO\text{-}(CH_2\text{-}CH_2O)_nH + 2nHI \rightarrow nICH_2\text{-}CH_2I + ROH + nH_2O$$

The unstable ethylene di-iodide is then decomposed on heating:

$$ICH_2\text{-}CH_2I \rightarrow C_2H_4 + I_2$$

NOTE — If **R** is an alkyl radical, **ROH** will be converted to **RI**. Some of the ethylene groups (and all of any oleyl alcohol present) will react with HI to form a stable iodide.

5 Reagents

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

5.1 Nitrogen or **carbon dioxide.**

5.2 Potassium iodide, crystals, minimum purity 99,5 %.

5.3 Phosphoric acid, ϱ_{20} approximately 1,70 g/ml.

5.4 Methanol, ϱ_{20} 0,79 g/ml.

5.5 Potassium iodide, 100 g/l solution.

5.6 Sodium thiosulfate, standard volumetric solution, $c(Na_2S_2O_3)$ = 0,1 mol/l.

5.7 Starch, indicator solution.

Mix 5 g of starch and 10 g of mercury(II) iodide with 30 ml of water; add this mixture to 1 litre of boiling water and boil for 3 min.

6 Apparatus

Ordinary laboratory apparatus and:

6.1 Round-bottom flask (see figures 1 and 2), capacity 50 ml, with a central neck fitted with a 19/26 conical ground-glass socket and a side neck fitted with a 14/23 conical ground-glass socket. The conical ground-glass joints shall comply with ISO 383.

6.2 Gas inlet tube (see figures 1 and 2), with a 14/23 conical ground-glass cone terminating in a length of at least 20 mm of tubing of 1,5 mm internal diameter. When fitted to the flask (6.1), the tube shall end about 10 mm above the bottom of the flask.

6.3 Water-cooled reflux condenser (see figures 1 and 2), with a nominal length of 200 mm to 500 mm, a 19/26 conical ground-glass cone at the bottom and a 14/23 conical ground-glass socket, attached at an angle of 45°, at the top (see figure 1). The conical ground-glass joints shall comply with ISO 383.

6.4 Absorption bulbs (see figures 1 and 2), of capacity 7 ml to 10 ml.

6.5 Sample introduction rod (see figure 1), comprising a solid glass rod with a wide-mouthed hollow cone at one end.

6.6 Oil bath, capable of being maintained at a temperature of 165 °C ± 1 °C.

6.7 Flow meter, capable of measuring flow rates in the region of 0,5 ml/s.

6.8 Burette, capacity 50 ml, complying with class A of ISO 385-1.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,1 mg, into the cone of the sample introduction rod (6.5)

— 60 mg to 300 mg of the sample in the case of a product containing less than 33 % (m/m) of ethylene oxide;

— sufficient of the sample to contain 20 mg to 100 mg of ethylene oxide in the case of a product containing more than 33 % (m/m) of ethylene oxide.

7.2 Determination

Invert the flask (6.1) over the sample introduction rod containing the test portion (see 7.1), then turn the flask upright, allowing the rod to slide into the flask so that its cavity end is at the bottom of the flask.

Add 3 g of the potassium iodide (5.2).

Attach the inlet tube (6.2) and the condenser (6.3) to the flask, using a little of the phosphoric acid (5.3) to lubricate the ground-glass joints.

Pass a stream of the nitrogen or carbon dioxide (5.1) into the flask at a flow rate greater than 0,5 ml/s for at least 20 min.

Without interrupting the gas flow or removing the condenser, place the flask in the oil bath (6.6) maintained at 165 °C ± 1 °C. Reduce the gas flow rate to between 0,1 ml/s and 0,5 ml/s. Add, via the condenser, 5 ml of the phosphoric acid (5.3) to the flask. Fill the absorption bulbs (6.4) with a suitable quantity (7 ml to 10 ml) of the potassium iodide solution (5.5) and attach them to the condenser.

Allow the reaction to proceed for 30 min, ensuring that the oil bath does not exceed the specified temperature during the last 20 min.

Remove the flask from the source of heat, without interrupting the gas flow, and allow to cool to below 80 °C.

Stop the flow of nitrogen or carbon dioxide.

Quickly turn the absorption bulbs about the axis of the joint so that their contents flow down the condenser into the flask. Turn the bulbs back to their original position. Introduce another 7 ml to 10 ml of the potassium iodide solution into the absorption bulbs and again turn them so that the contents flow down the condenser into the flask. Remove the absorption bulbs. Rinse the condenser and its lower joint with a few millilitres of the potassium iodide solution, collecting the rinsings in the flask, and remove the condenser.

Similarly, rinse and remove the gas inlet tube.

Transfer quantitatively the contents of the round-bottom flask to a 250 ml conical flask, rinsing with water and a few millilitres of the potassium iodide solution. If a sparingly soluble residue is seen, dissolve this in a few millilitres of the methanol (5.4).

Titrate with the standard volumetric sodium thiosulfate solution (5.6), adding the starch indicator solution (5.7) when the iodine colour has nearly disappeared.

7.3 Blank test

Carry out a blank test in parallel with the determination, using the same reagents and following the same procedure as for the determination, but omitting the test portion.

8 Expression of results

8.1 Calculation

The oxyethylene content, expressed as a percentage by mass, is given by the formula

$$\frac{(V_1 - V_0)\, c \times 0{,}022 \times 100}{m}$$

$$= \frac{2{,}2\,(V_1 - V_0)\, c}{m}$$

where

V_0 is the volume, in millilitres, of the sodium thiosulfate solution (5.6), used for the blank test (see 7.3);

V_1 is the volume, in millilitres, of the sodium thiosulfate solution (5.6), used for the determination (7.2);

c is the exact concentration, in moles of $Na_2S_2O_3$ per litre, of the sodium thiosulfate solution (5.6);

m is the mass, in grams, of the test portion (7.1);

0,022 is the mass, in grams, of oxyethylene corresponding to 1,00 ml of sodium thiosulfate solution, $c(Na_2S_2O_3) = 1{,}000$ mol/l.

8.2 Precision

8.2.1 Repeatability

The maximum difference found between the results of two determinations carried out on the same product simultaneously by the same analyst using the same apparatus should not exceed 1,5 % of the average value for derivatives containing more than 10 % (m/m) of ethylene oxide.

8.2.2 Reproducibility

The difference between the results obtained with the same sample in two different laboratories should not exceed 3 % of the average value for derivatives containing more than 10 % (m/m) of ethylene oxide.

9 Test report

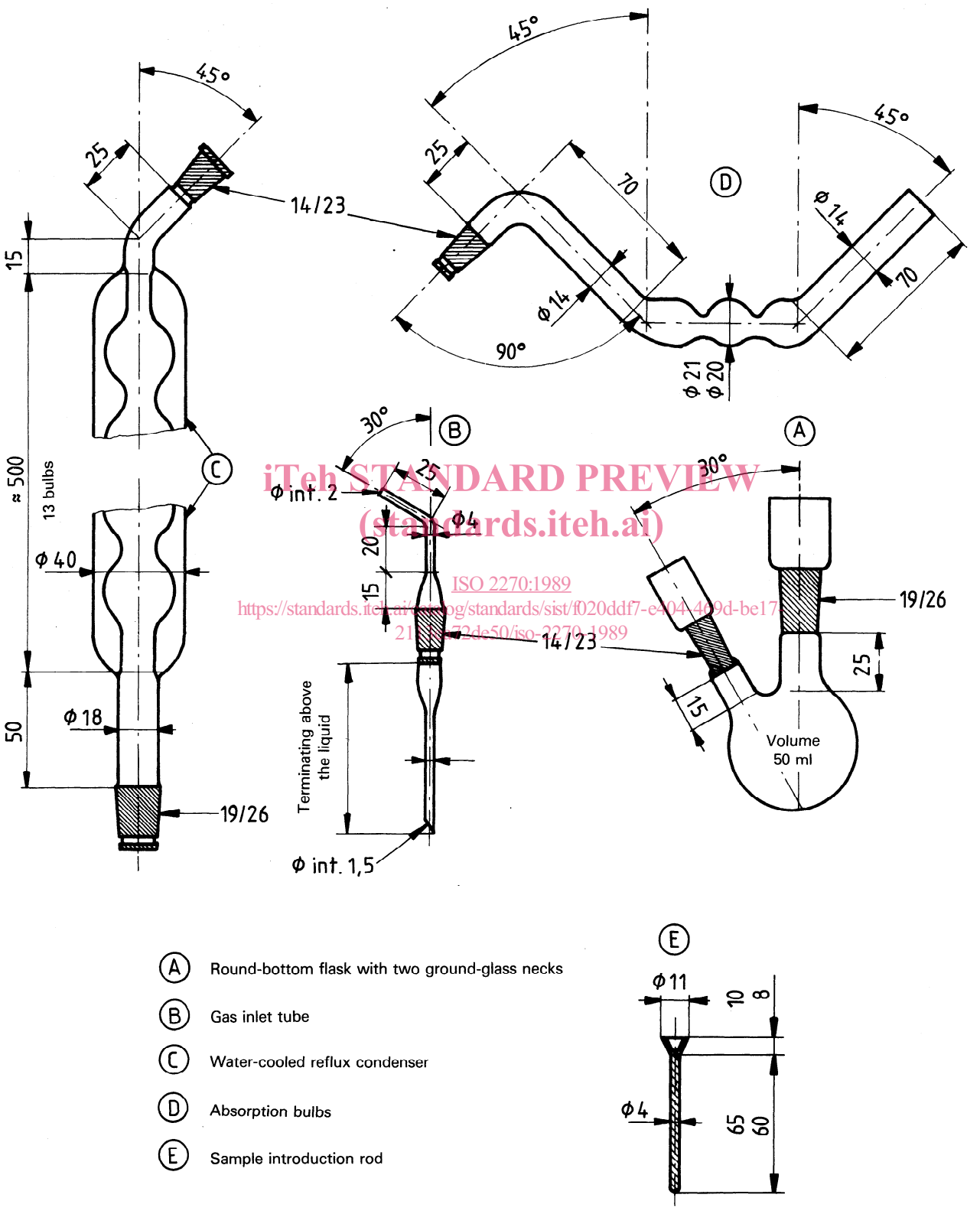
The test report shall include be following information:

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

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

c) the results obtained, as well as the units in which they have been expressed;

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



(A) Round-bottom flask with two ground-glass necks

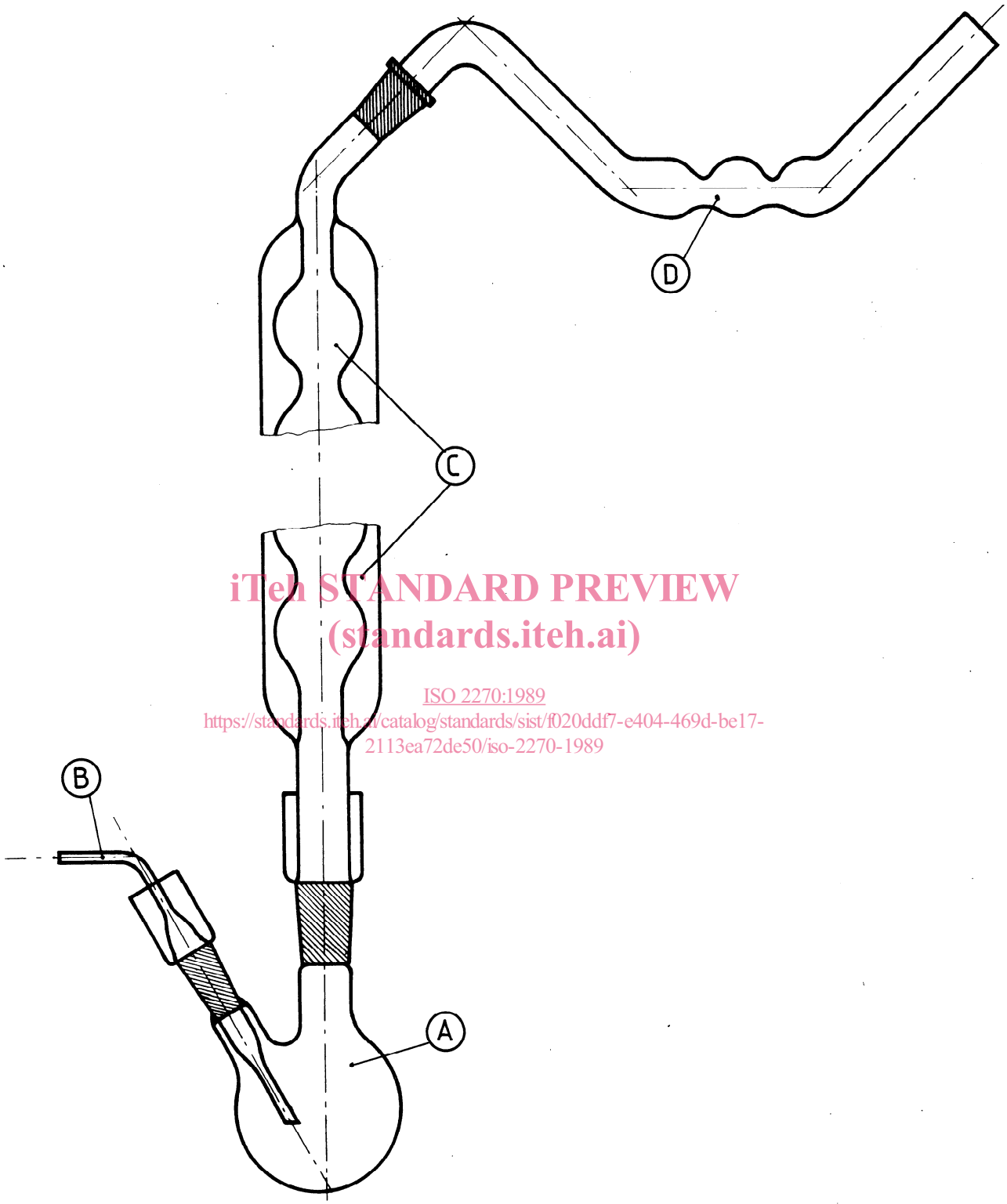
(B) Gas inlet tube

(C) Water-cooled reflux condenser

(D) Absorption bulbs

(E) Sample introduction rod

Figure 1 — Details of glassware



NOTE — For key, see figure 1.

Figure 2 — Assembled apparatus

UDC 661.185.4 : 543.242.3 : 547.422.22-31

Descriptors : surfactants, non-ionic surfactants, chemical analysis, determination of content, ethylene oxide, iodometry.

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