INTERNATIONAL STANDARD ISO 2270

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Surface active agents — Ethylene oxide adducts — Iodometric determination of oxyethylene groups

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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 2270 was drawn up by Technical Committee ISO/TC 91, *Surface active agents*.

It was approved in August 1971 by the Member Bodies of the following countries :

Austria	Japan	Sweden
Belgium	New Zealand	Switzerland
Chile	Poland	Thailand
Egypt, Arab Rep. of	Portugal	Turkey
France	Romania	United Kingdom
Germany	South Africa, Rep. of	U.S.A.
Hungary	Spain	U.S.S.R.

No Member Body expressed disapproval of the document.

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Surface active agents — Ethylene oxide adducts — Iodometric determination of oxyethylene groups

1 SCOPE

This International Standard specifies a method for the determination of oxyethylene groups in ethylene oxide adducts.

2 FIELD OF APPLICATION

This method is applicable to the analysis of adducts of ethylene oxide with

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

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

This method is not applicable in the presence of

- compounds containing sulphur 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 REFERENCES

ISO/R 383, *Interchangeable conical ground glass joints.*

ISO/R 385, *Burettes.*

4 PRINCIPLE

Hydrolysis of the oxyethylene groups by nascent hydriodic acid.

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

5 REACTIONS

The basic reaction is the conversion of each (CH_2-CH_2O) group to ethylene di-iodide ICH_2-CH_2I:

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

The unstable ethylene di-iodide is then decomposed at 200 °C :

$$ICH_2-CH_2I \rightarrow C_2H_4 + I_2$$

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.

6 REAGENTS

The water used shall be distilled water or water of at least equivalent purity. The reagents used shall have the following properties :

6.1 Nitrogen or carbon dioxide.

6.2 Potassium iodide, crystals, minimum purity 99.5 %.

6.3 Phosphoric acid, $\rho_{20} = 1.70$ g/ml, approximately.

6.4 Methanol, $\rho_{20} = 0.79$ g/ml.

6.5 Potassium iodide, 100 g/l solution.

6.6 Sodium thiosulphate ($Na_2S_2O_3$), 0.1 N standard volumetric solution.

6.7 Starch solution.

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

7 APPARATUS

Ordinary laboratory apparatus and (see Figures 1 and 2) :

7.1 Round bottom flask, 50 ml, with a central neck fitted with a 19/26 conical ground glass cone and a side neck fitted with a 14/23 ground glass conical cone. The conical ground glass joints shall comply with ISO/R 383.

7.2 Gas inlet tube, with a 14/23 conical ground glass cone terminating in a length of at least 20 mm of tube of 1.5 mm internal diameter. When fitted to the flask, the tube should end about 10 mm above the bottom of the flask (7.1).

7.3 Reflux water-cooled condenser, with a 200 to 500 mm jacket, 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/R 383.

7.4 Bubbler, of capacity 7 to 10 ml (see Figure 1).

7.5 Boiling rod (see Figure 1), comprising a solid glass rod with a wide-mouthed cone at one of the ends.

7.6 Oil bath, capable of being thermostatically controlled at 165 ± 1 °C.

7.7 Flow meter, to indicate flows in the region of 0.5 ml/s.

7.8 Burette, 50 ml, complying with Class A of ISO/R 385.

8 PROCEDURE

8.1 Test portion

Weigh, to the nearest 0.1 mg, into the boiling rod (7.5) :

- 60 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 to 100 mg of ethylene oxide in the case of a product containing more than 33 % (m/m) of ethylene oxide.

8.2 Determination

Invert the flask (7.1) over the boiling rod containing the test portion (see 8.1), 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 (6.2).

Attach the gas inlet tube (7.2) and the condenser (7.3), using a little of the phosphoric acid (6.3) as lubricant.

Pass a stream of the nitrogen or carbon dioxide (6.1) 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 (7.6) maintained at 165 ± 1 °C. Reduce the gas flow rate to between 0.1 and 0.5 ml/s. Add 5 ml of the phosphoric acid (6.3) via the condenser into the flask. Fill the bubbler (7.4) with a suitable quantity (7 to 10 ml) of the potassium iodide solution (6.5) and attach it 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 bubbler about the axis of the joint so that its contents flow down the condenser into the flask. Turn the bubbler back to its original position and repeat the washing with another 7 to 10 ml of the potassium iodide solution (6.5). Rinse the condenser and its lower joint with a few millilitres of the potassium iodide solution (6.5), collecting the rinsings in the flask, and remove the condenser.

Similarly rinse and remove the gas inlet tube.

Transfer the contents of the flask to a 250 ml conical flask, rinsing with water and a few millilitres of the potassium iodide solution (6.5). If a sparingly soluble residue is seen, dissolve this in a few millilitres of the methanol (6.4).

Titrate with the 0.1 N sodium thiosulphate solution (6.6), adding the starch indicator solution (6.7) when the iodine colour has nearly disappeared.

8.3 Blank test

Carry out a blank test, following the same procedure as described in 8.2.

9 EXPRESSION OF RESULTS

9.1 Calculation

The oxyethylene content, as a percentage by mass, is equal to

$$\frac{(V_1 - V_0) \times T \times 2.2}{m}$$

where

V_0 is the volume, in millilitres, of the sodium thiosulphate solution used for the blank test;

V_1 is the volume, in millilitres, of the sodium thiosulphate solution used for the sample;

T is the normality of the sodium thiosulphate solution;

m is the mass, in grams, of the test portion.

9.2 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 shall not exceed 1.5 % of the average value for adducts containing more than 10 % of ethylene oxide.

9.3 Reproducibility

The difference between the results obtained with the same sample in two different laboratories shall not exceed 3 % of the average value for adducts containing more than 10 % of ethylene oxide.

10 TEST REPORT

The test report shall include the following information :

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

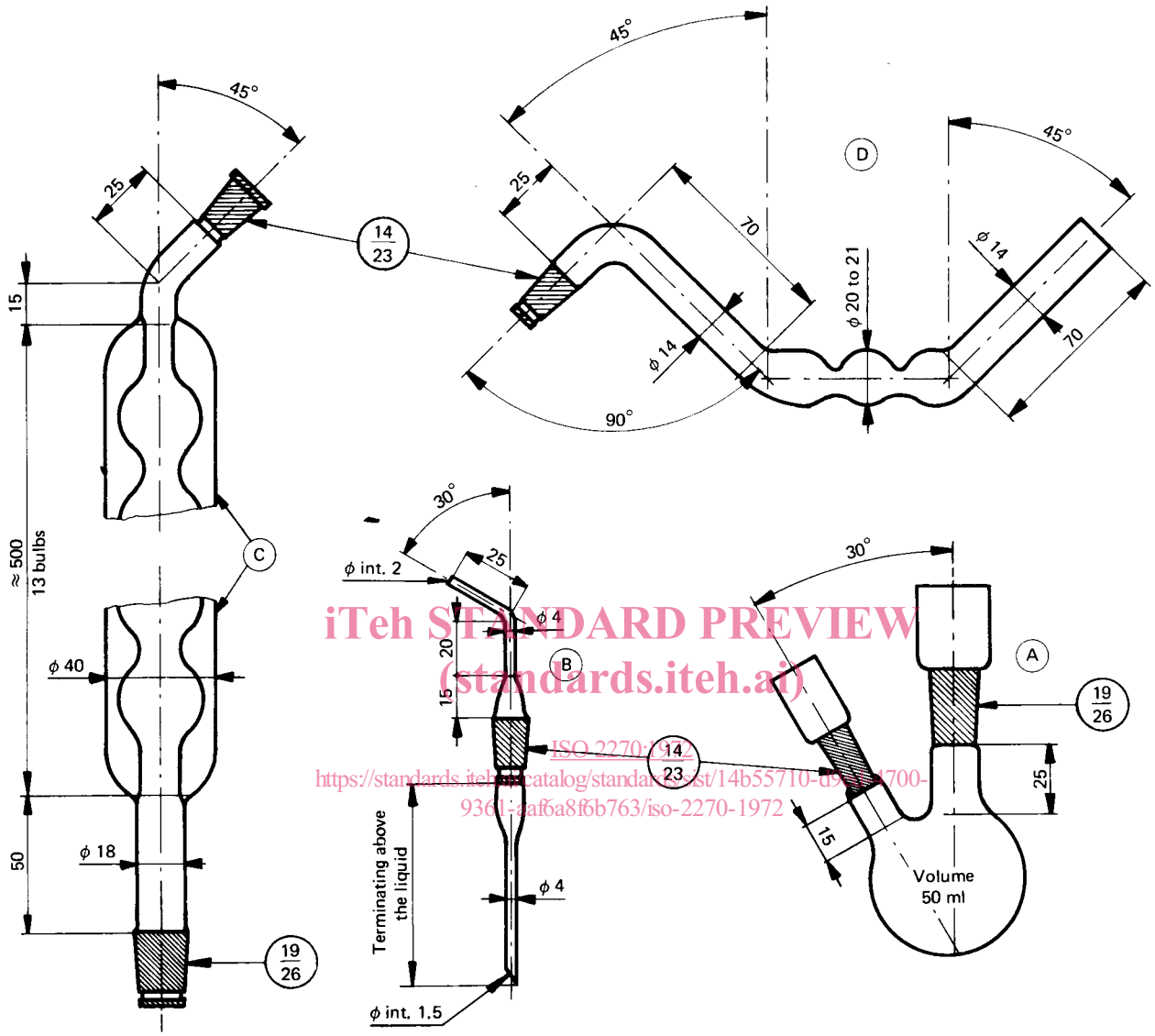
b) the method used;

c) the results obtained;

d) the test conditions;

e) any operational details not specified in this International Standard, or optional, as well as all incidents likely to have influenced the results.

Dimensions in millimetres



(A) Round bottom flask with two ground necks

(B) Gas inlet tube

(C) Reflux water-cooled condenser

(D) Bubbler

(E) Boiling rod

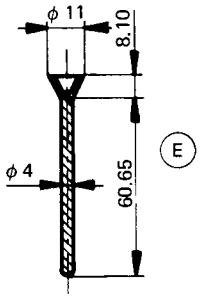


FIGURE 1 — **Details of glassware**

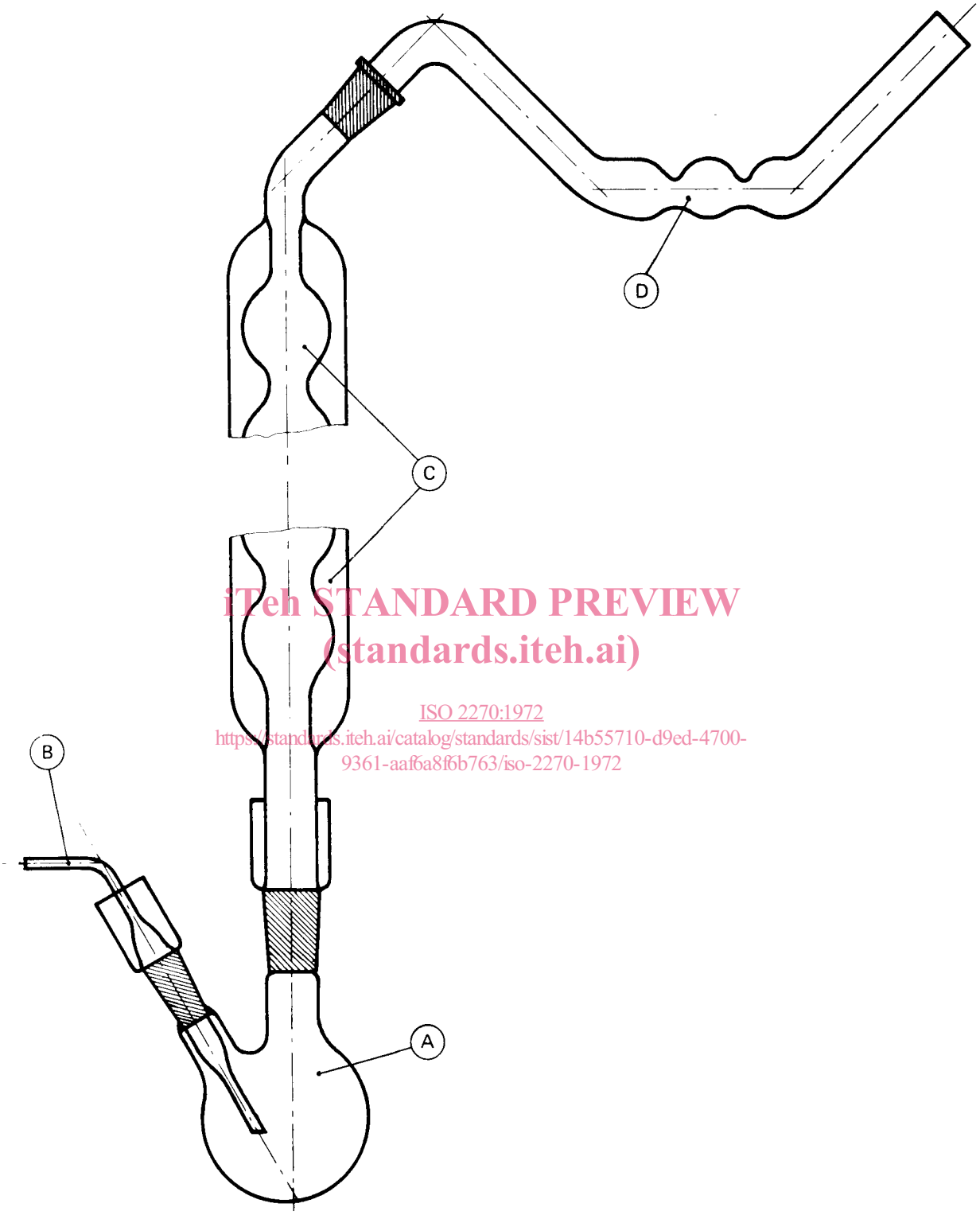


FIGURE 2 – **Assembled apparatus**