2268-72 4851903 0010248 0

INTERNATIONAL STANDARD ISO 2268

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

Surface active agents (non-ionic) — Determination of polyethylene glycols and non-ionic active matter (adducts) — Weibull method

First edition — 1972-11-15

UDC 661.185.4 : 547.422 Ref. No. ISO 2268-1972 (E)

Descriptors : surfactants, chemical analysis, determination of content, by-products, polyoxyethylene, condensates.

Price based on 5 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 2268 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	New Zealand	Sweden
Belgium	Poland	Switzerland
Egypt, Arab Rep. of	Portugal	Turkey
Germany	Romania	United Kingdom
Hungary	South Africa, Rep. of	U.S.A.
Japan	Spain	U.S.S.R.

The Member Body of the following country expressed disapproval of the document on technical grounds :

France

Printed in Switzerland

2268-72 4851903 0010250 9

Surface active agents (non-ionic) — Determination of polyethylene glycols and non-ionic active matter (adducts) — Weibull method

1 SCOPE

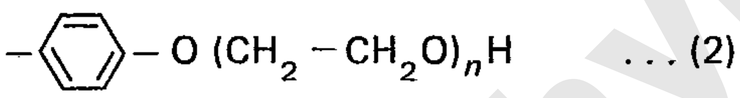
This International Standard specifies a method for the determination of polyethylene glycols and non-ionic active matter (adducts) in fatty alcohol and alkylphenol polyoxyethylate derivatives.

Generally the commercial products contain polyethylene glycols as a by-product. The Weibull method allows the determination of both the non-ionic active matter (adducts) and the polyethylene glycols impurity.

2 FIELD OF APPLICATION

Alkyl and alkylphenol polyoxyethylates correspond to the formulae (1) and (2) given below :

$$RO\,(CH_2-CH_2O)_nH \qquad \ldots (1)$$



$$R-\langle\bigcirc\rangle-O\,(CH_2-CH_2O)_nH \qquad \ldots (2)$$

where n is a mean number of oxyethylene (CH_2—CH_2O) groups per molecule of hydrophobe and R is, in formula (1), a straight or branched chain alkyl group, usually comprising C_{10} to C_{18}, and, in formula (2), a branched chain alkyl group, usually nonyl or tertiary octyl.

The method is applicable to all commercial non-ionic surface active agents containing from 2 to 80 oxyethylene groups per molecule.

3 PRINCIPLE

The method relies on the facts that both non-ionic adducts and polyethylene glycols are soluble in sodium chloride solution, but one is soluble in ethyl acetate whereas the other is not.

Separation of the polyethylene glycols and the adducts is, therefore, possible by the following method :

Dissolution of the sample in ethyl acetate and extraction, at $35 \pm 1\ ^{\circ}C$, of polyethylene glycols by sodium chloride solution and successive washes of the sodium chloride solution by ethyl acetate, and of the ethyl acetate by the sodium chloride solution.

Extraction of the polyethylene glycols isolated in the sodium chloride solution with chloroform, removal of the solvent and weighing of the residue.

Evaporation of the ethyl acetate solution which contains the adducts and weighing of the residue.

4 REAGENTS

The water used shall be distilled water or water of at least equivalent purity.

The reagents used shall have the following properties.

4.1 Ethyl acetate, $\rho_{20} = 0{,}90$ g/ml, distilling between 75,5 and 77,5 °C.

4.2 Sodium chloride solution

Dissolve 300 g of sodium chloride in 1 000 ml of distilled water.

4.3 Chloroform, $\rho_{20} = 1{,}48$ g/ml, distilling between 59,5 and 61,2 °C.

4.4 Acetone, anhydrous, $\rho_{20} = 0{,}79$ g/ml, distilling between 55 and 57 °C.

4.5 Light petroleum, distilling between 40 and 60 °C.

5 APPARATUS

Ordinary laboratory apparatus and :

5.1 Separating funnels

5.1.1 *three separating funnels*, capacity 250 ml, with ground glass stoppers, for method 1 (see Annex).

5.1.2 *three separating funnels*, capacity 250 ml, with ground glass stoppers and jackets, according to Figure, for method 2 (see Annex).

5.2 One separating funnel, capacity 500 ml, with ground glass stopper.

5.3 Two flasks, capacity 250 ml, wide-necked, with ground glass stoppers.

5.4 Conical flask, capacity 500 ml, wide-necked flat-bottomed.

6 PROCEDURE

6.1 Preparation of the sample

6.1.1 *Liquid products*

If the sample is a clear liquid, stir it with a glass rod or spoon to ensure that the sample is homogeneous.

If the liquid is cloudy or contains a solid deposit, heat the product slowly to 45 °C maximum, leaving the cover or the stopper of the container in position until the liquid is clear. Then stir with a glass rod or spoon to obtain a homogeneous sample.

6.1.2 *Solid products*

If the sample is in a solid form, heat it slowly to 45 °C maximum, as described above, in a dry oven until the sample has just melted.

Remove from the oven and stir until solidification is well advanced and stirring is impossible. Allow the sample to cool to room temperature.

6.2 Test portion

Weigh, to the nearest 0,01 g, 5 ± 0,05 g of a sample prepared as described in 6.1.

6.3 Determination

6.3.1 *Separation of the polyethylene glycols and adducts*

During this separation, all the operations, including the rest phases, shall be carried out at 35 ± 1 °C, the reagents and glassware having beforehand been raised to this temperature (see Annex).

Dissolve the test portion in the ethyl acetate (4.1) and transfer it quantitatively to the 250 ml separating funnel (5.1) (A), bringing the final volume to 75 ml. Add 50 ml of the sodium chloride solution (4.2), shake to mix well, leave standing for about 30 min or until the two phases are well separated and then run the sodium chloride solution into the second 250 ml separating funnel (5.1) (B).

Again add 50 ml of the sodium chloride solution (4.2) to the ethyl acetate solution contained in funnel (A) and begin the extraction operation again under the same conditions as before, collecting the sodium chloride solution in funnel (B).

Repeat the extraction for a third time, again with 50 ml of the sodium chloride solution (4.2).

Add 25 ml of the ethyl acetate (4.1) to funnel (B), which now contains the three portions of sodium chloride solution, shake, leave standing for about 30 min, and then run the sodium chloride solution into the third 250 ml separating funnel (5.1) (C). Add 25 ml of the ethyl acetate (4.1) to funnel (C) and start the washing process again, under the same conditions as above, collecting the sodium chloride solution in the 500 ml separating funnel (5.2) (D). Transfer the ethyl acetate from funnel (C) to funnel (B).

Add 25 ml of the sodium chloride solution (4.2) to funnel (B), which now contains the two portions of ethyl acetate used for washing the sodium chloride solution. Shake, allow the phases to separate and run the sodium chloride solution into funnel (C). Use this solution to rinse funnel (C) and transfer this rinse to funnel (D). Rinse funnel (C) with 10 ml of sodium chloride solution (4.2) and transfer this rinse to funnel (D).

Funnel (D) now contains the sodium chloride solution in which the polyethylene glycols are to be found, while the adducts are in solution in the ethyl acetate contained in funnels (A) and (B).

6.3.2 *Determination of adducts*

Transfer the ethyl acetate solution in funnel (A) to the 250 ml wide-neck flask (5.3) and evaporate the solvent. Rinse funnel (A) with the ethyl acetate solution contained in funnel (B), then transfer this solution to the 250 ml flask. Pour 25 ml of the ethyl acetate (4.1) into funnel (B) to rinse it, transfer this rinse to funnel (A) to rinse it also, then run into the 250 ml flask. Repeat this operation with 25 ml of the ethyl acetate (4.1) then evaporate if off completely.

Dissolve the residue in 75 ml of the ethyl acetate (4.1), heat at 45 °C and filter immediately through filter paper (Whatman No. 541, 9 cm diameter or equivalent) collecting the filtrate in the tared 250 ml flask (5.3) fitted with a ground glass stopper. Rinse the first flask and the filter paper six times with 10 ml portions of warm ethyl acetate (4.1), always collecting the filtrate in the tared flask. Completely evaporate the solvent on a steam bath.

Dry the residue by adding 10 ml of the acetone (4.4), evaporate it off, then remove as much solvent vapour as possible by passing a stream of cold dry air through the flask. Repeat these operations with a further 10 ml of the acetone (4.4).

Then add 10 ml of the light petroleum (4.5), evaporate off and remove the last traces of solvent vapour with a stream of cold dry air.

Stopper the flask, cool in a desiccator and weigh after releasing and immediately replacing the stopper. Heat the flask in an oven at 100 °C for 10 min, blow out with a stream of cold dry air, replace the stopper, allow to cool and weigh again. Repeat these heating and weighing operations until the difference between two consecutive weighings does not exceed 0,1 % of the mass of active matter.

6.3.3 *Determination of polyethylene glycols*

Add 100 ml of the chloroform (4.3) to separating funnel (D) containing the sodium chloride solution, shake, leave to stand for at least 15 min and run the chloroform phase into the 500 ml conical flask (5.4).

Repeat this operation twice more, each time with 100 ml of the chloroform (4.3).

Evaporate off the chloroform. Dissolve the residue in 50 ml of the chloroform (4.3), filter through filter paper (Whatman No. 541, 9 cm diameter or equivalent) and collect the filtrate in a tared, wide-neck flask with a ground glass stopper. Rinse the original flask and filter paper six times with 10 ml portions of warm chloroform (4.3) always collecting the filtrate in the tared flask. Evaporate off the solvent on a steam bath.

Dry the residue twice with 30 ml of the acetone (4.4) and once with 10 ml of the light petroleum (4.5) using the same procedure as described in 6.3.2. Then weigh as before.

NOTE — A general diagram of the separation procedures is given in an Appendix.

7 EXPRESSION OF RESULTS

The percentage by mass of polyethylene glycols in the sample is equal to

$$\frac{m_1 \times 100}{m_0}$$

The percentage by mass of non-ionic active matter (adducts) is equal to

$$\frac{m_2 \times 100}{m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of polyethylene glycols, weighed as described in 6.3.3;

m_2 is the mass, in grams, of adducts, weighed as described in 6.3.2.

8 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, reference being made to this International Standard;

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.

ANNEX

MAINTENANCE OF TEMPERATURE DURING THE SEPARATIONS

The two following techniques may be used for maintaining the temperature during the separations.

A.1 METHOD 1

The separations are carried out either in a small room or in a large cupboard, maintained at $35 \pm 1\ ^{\circ}C$ by means of a thermostat, in which the reagents and apparatus shall be kept permanently. Dissolving the sample and all the separations shall be carried out at this temperature and the separating funnels (5.1.1) used shall not, under any circumstances, be withdrawn from this room until all the separations with ethyl acetate have been completed. (The extraction of polyethylene glycols from the sodium chloride solution with chloroform may be carried out at ambient temperature.)

A.2 METHOD 2

The separations are carried out in separating funnels with ground glass stoppers and jackets (5.1.2). These jackets are fitted with inlets and outlets, like reflux condensers, which enable water, at a temperature controlled by a thermostat, to circulate continuously between the double walls so that the content of the funnels can be maintained at a suitable temperature. The dimensions and form of such a funnel are given in Figure 2.

Dimensions in millimetres

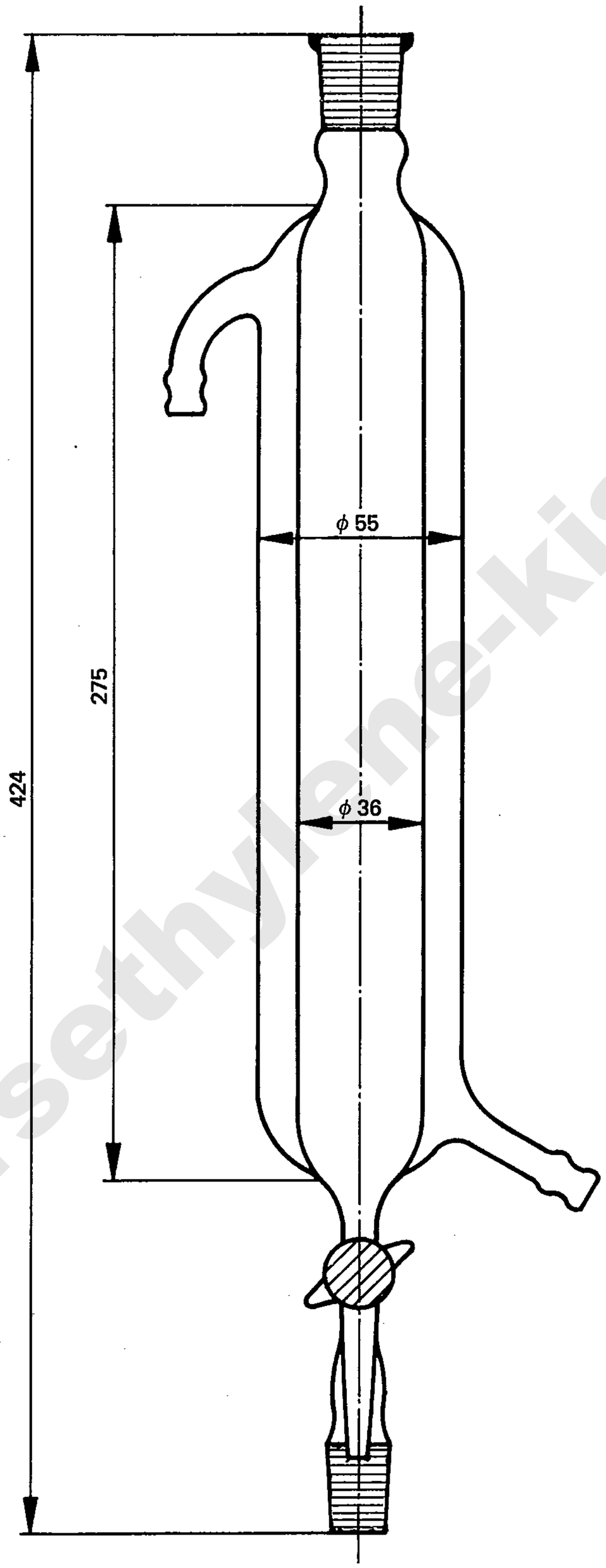


FIGURE — **Suitable type of separating funnel with jacket (5.1.2)**

APPENDIX

GENERAL DIAGRAM OF SEPARATIONS