
International Standard



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Surface active agents — Polyethoxylated alcohol and alkylphenol sulfates — Determination of total active matter content

Agents de surface — Sulfates d'alcools et d'alkylphénols polyéthoxylés — Détermination de la teneur en matière active totale

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (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 authorized 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 6842 was developed by Technical Committee ISO/TC 91, *Surface active agents*, and was circulated to the member bodies in March 1982.

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

Australia	Hungary	Romania
Austria	Iran	South Africa, Rep. of
Belgium	Ireland	Spain
China	Italy	Switzerland
Czechoslovakia	Japan	United Kingdom
Egypt, Arab Rep. of	Mexico	USA
France	Netherlands	USSR
Germany, F. R.	Poland	

No member body expressed disapproval of the document.

Surface active agents — Polyethoxylated alcohol and alkylphenol sulfates — Determination of total active matter content

1 Scope and field of application

This International Standard specifies a method for the determination of the total active matter content in ordinary commercial neutralized products of sulfation of ethoxylated alcohols or alkylphenols [alkyl polyoxyethylene sulfates (polyethoxylated alcohol sulfates) or alkylphenyl polyoxyethylene sulfates (polyethoxylated alkylphenol sulfates)].

The total active matter comprises the organic material soluble in ethanol (alkylether sulphates, alkylphenylether sulfates, polyglycol sulphates, and non-ionic fractions).

2 Reference

ISO 607, *Surface active agents and detergents — Methods of sample division.*

3 Principle

Boiling under reflux, of a test portion with ethanol in the presence of sodium sulfate. Filtration, evaporation of the filtrate and weighing of the residue. Determination of any sodium chloride present, by dissolution of the residue in aqueous acetone and titration with standard volumetric silver nitrate solution. Correction of the mass of the residue for the sodium chloride content.

4 Reagents

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

4.1 Ethanol, 99 % (V/V).

4.2 Dichloromethane.

4.3 Sodium sulfate, anhydrous.

4.4 Acetone, 50 % (V/V) aqueous solution.

4.5 Silver nitrate, standard volumetric solution, $c(\text{AgNO}_3) = 0,1 \text{ mol/l}$.

4.6 Potassium chromate, 100 g/l indicator solution.

5 Apparatus

Ordinary laboratory apparatus and :

5.1 Conical flask, of capacity 250 ml, having a ground glass neck.

5.2 Rotary evaporator, with round-bottomed flasks of capacity 250 ml.

5.3 Condenser, to fit the conical flask (5.1).

6 Sampling

The laboratory sample shall be prepared and stored in accordance with the requirements of ISO 607.

7 Procedure

7.1 Test portion

From the laboratory sample, rendered homogeneous (if necessary) by the addition of a known, appropriate quantity of water, weigh, to the nearest 0,001 g, into the conical flask (5.1) a quantity of homogeneous material containing about 0,5 to 1,5 g of total active matter.

7.2 Determination

Introduce into the conical flask containing the test portion (7.1) 100 ml of the ethanol (4.1) and 100 mg of the sodium sulfate (4.3), fit the condenser (5.3), and boil under reflux for 30 min.

Disconnect the condenser. Rinse the inner wall of the condenser and the neck of the flask with ethanol, collect the washings in the flask and allow to settle.

Filter the contents of the conical flask whilst still hot through a fast filter paper into one of the round bottomed flasks (5.2), previously dried and tared to the nearest 1 mg, rinse the conical flask with about 50 ml of hot ethanol, filter and collect the washings in the round bottomed flask.

Evaporate the ethanolic solution by means of the rotary evaporator (5.2) controlled at approximately 40 °C. Add 10 ml of the dichloromethane (4.2) and evaporate. Repeat this step using a further 10 ml of the dichloromethane. Remove the last traces of water by evaporation and leave the flask for a further 15 min on the rotary evaporator.

Remove the flask from the rotary evaporator, allow to stand in a desiccator for 15 min and weigh the flask and contents.

Leave the flask for a further 15 min on the rotary evaporator, then allow it to stand in the desiccator for 15 min and again weigh the flask and contents. Repeat the process of drying and weighing until the difference between two consecutive weighings does not exceed 3 mg.

Dissolve the residue in 60 to 80 ml of the aqueous acetone (4.4). Add 1 ml of the potassium chromate indicator solution (4.6) and titrate with the silver nitrate solution (4.5) until a permanent brown coloration.

7.3 Blank test

Carry out a blank test at the same time as the determination using the same reagents and following the same procedure, but omitting the test portion.

8 Expression of results

8.1 Method of calculation

The total active matter content, expressed as a percentage by mass, is given by the formula

$$\frac{m_1 - 0,058\ 5\ c(V_1 - V_0)}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion (7.1);¹⁾

m_1 is the mass, in grams, of the residue obtained;

V_0 is the volume, in millilitres, of the silver nitrate solution (4.5) used for the blank test (7.3);

V_1 is the volume, in millilitres, of the silver nitrate solution (4.5) used for the determination (7.2) of any sodium chloride present;

c is the concentration, in moles of AgNO_3 per litre, of the silver nitrate solution (4.5);

0,058 5 is the mass, in grams, of sodium chloride corresponding to 1,00 ml of silver nitrate solution $c(\text{AgNO}_3) = 0,100$ mol/l.

8.2 Precision

Comparative analysis, carried out in 15 laboratories, have given the following statistical results :

— mean [total active matter, % (m/m)] :	58,67
— standard deviation of repeatability :	0,33
— standard deviation of reproducibility :	0,94

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9 Test report

The test report shall include the following information :

- a) all information necessary for the complete identification of the sample;
- b) the reference of the method used (reference to this International Standard);
- c) the results and the method of expression used;
- d) the test conditions;
- e) any details not specified in this International Standard or in the International Standard to which reference is made, or regarded as optional, as well as any incidents likely to have affected the results.

1) Corrected for dilution in the case of heterogeneous products.