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International Standard ISO 6844

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Surface active agents — Determination of mineral sulfate content — Titrimetric method

Agents de surface — Détermination de la teneur en sulfate minéral — Méthode titrimétrique

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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 6844 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
Austria
China
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France
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South Africa, Rep. of
Spain
Switzerland
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The member body of the following country expressed disapproval of the document on technical grounds :

Belgium

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INTERNATIONAL STANDARD ISO 6844-1983 (E)

Surface active agents — Determination of mineral sulfate content — Titrimetric method

1 Scope

This International Standard specifies a titrimetric method for the determination of the mineral sulfate content of surface active agents.

2 Field of application

This International Standard applies to anionic surface active agents, containing sodium, ammonium and alkanolamine sulfates.

It is not applicable to products containing other compounds such as phosphates or large amounts of chloride, which also give a sparingly soluble lead salt under the conditions of the test, nor is it applicable in the presence of large amounts of salts of weak acids (for example, soaps or monoesters of sulfosuccinic acid) which prevent the correct adjustment of the pH.

NOTE — It is essential to conduct the determination at the apparent pH specified in note 1 to 8.2. At apparent pH values below 4, the reaction is not stoichiometric.

This International Standard is applicable only if so indicated in the relevant International Standard for each product.

3 References

ISO 385/2, *Laboratory glassware — Burettes — Part 2 : Burettes for which no waiting time is specified.*[1]

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

ISO 648, *Laboratory glassware — One-mark pipettes.*

ISO 835/2, *Laboratory glassware — Graduated pipettes — Part 2 : Pipettes for which no waiting time is specified.*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks.*

4 Principle

Titration of a buffered acetonic solution of a test portion with a standard volumetric lead(II) nitrate solution in the presence of dithizone as indicator.

5 Reagents

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

5.1 Acetone.

5.2 Nitric acid, solution $c(HNO_3) \approx 1$ mol/l.

5.3 Sodium hydroxide, 40 g/l solution.

5.4 Lead(II) nitrate, standard volumetric solution, $c[Pb(NO_3)_2] = 0{,}01$ mol/l.

Dissolve 3,312 ± 0,005 g of lead(II) nitrate in 200 ml of water. Transfer to a 1 000 ml one-mark volumetric flask fitted with a ground glass stopper, dilute to the mark with water and mix thoroughly.

5.5 Ammonium dichloroacetate, buffer solution, pH 1,5 to 1,6, giving in acetonic medium, 70 to 85 % (*V*/*V*), an apparent pH value of 4,1 ± 0,2.

Add 67 ml of dichloroacetic acid to about 250 ml water and carefully neutralize with 18 % (*m*/*m*) ammonia solution (about 80 ml) to pH 7 as indicated by a pH-meter or a precise indicator paper. Allow to cool, add a further 33 ml of dichloroacetic acid and dilute to 600 ml.

5.6 1,5-Diphenyl thiocarbazone (Dithizone) (C_6H_5—NH—NHCSN=NC_6H_5), 0,5 g/l solution in acetone.

Store in a dark bottle and discard after 1 week.

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[1] At present at the stage of draft. (Revision, in part, of ISO/R 385-1964.)

6 Apparatus

Ordinary laboratory apparatus and

6.1 Beaker, of capacity 150 ml.

6.2 One-mark volumetric flask, of capacity 100 ml, complying with the requirements of ISO 1042.

6.3 Conical flask, of capacity 250 ml.

6.4 Pipettes, of capacities 5 — 10 — 15 and 20 ml, complying with the requirements of ISO 648.

6.5 Graduated pipette, of capacity 1 or 2 ml, complying with the requirements of ISO 835/2.

6.6 Burette, of capacity 25 ml, complying with the requirements of class A of ISO 385/2.

6.7 Graduated measuring cylinder, of capacity 100 ml.

7 Sampling

The laboratory sample of the surface active agent shall be prepared and stored according to the instructions given in ISO 607.

8 Procedure

8.1 Test portion

Depending on the expected sodium sulfate content, weigh, to the nearest 0,001 g, the amount of laboratory sample given in table 1, into the beaker (6.1).

Table 1 — Mass of test portion

Expected sodium sulfate content in sample	Mass of test portion
% (*m*/*m*)	g
< 0,5	10 to 14
0,5 to 6	5
> 6*	< 5

* For a product containing more than 6 % (*m*/*m*) of sodium sulfate, weigh a test portion containing 0,1 to 0,3 g of sodium sulfate.

8.2 Determination

Dissolve the test portion (8.1) in about 50 ml of water by warming, if necessary, to not more than 50 °C. Transfer quantitatively to the one-mark volumetric flask (6.2), dilute to the mark with water and mix thoroughly.

By means of one of pipettes (6.4), take an appropriate aliquot portion, depending on the expected sodium sulfate content as given in table 2.

Table 2 — Volume of aliquot portions

Expected sodium sulfate content	Volume of aliquot portions
% (*m*/*m*)	ml
> 3	5,0
1 to 3	10,0
0,5 to 1,5	15,0
< 0,5	20,0

Transfer the aliquot portion to the conical flask (6.3) and make up to 20 ml with water. Add 1 ml of dithizone solution (5.6).

If the solution is green, add the sodium hydroxide solution (5.3) until the appearance of a red colour.

Add the nitric acid solution (5.2), drop by drop, until the appearance of a green colour and then add 2,0 ml of the ammonium dichloroacetate solution (5.5) and 80 ml of the acetone (5.1). Immediately after the addition of the acetone, titrate the buffered acetonic solution with the lead(II) nitrate solution (5.4) until a brick-red colour that is stable for 15 s is obtained.

NOTES

1 The initial apparent pH value is 4,1 ± 0,2 and decreases considerably during the determination.

2 At the end-point the acetone content shall be between 70 and 85 % (*V*/*V*).

9 Expression of results

9.1 Method of calculation

The mineral sulfate content, expressed as a percentage by mass of sodium sulfate, is given by the formula

$$\frac{0{,}142 \times V_1 \times c \times 100}{\dfrac{m_0 V_0}{100}}$$

$$= \frac{1\,420 \times V_1 \times c}{m_0 V_0}$$

where

m_0 is the mass, in grams, of the test portion (8.1);

V_0 is the volume, in millilitres, of the aliquot portion taken for the determination (8.2);

V_1 is the volume, in millilitres, of the lead(II) nitrate solution (5.4) used in the determination (8.2);

c is the actual concentration, expressed in moles of $Pb(NO_3)_2$ per litre, of the lead(II) nitrate solution (5.4);

0,142 is the mass, in grams, of sodium sulfate corresponding to 1,00 ml of lead(II) nitrate solution, $c[Pb(NO_3)_2] = 1{,}000$ mol/l.

9.2 Precision

Comparative analyses, on samples of two ammonium laurylethersulfates containing respectively about 0,15 and 1,15 % (*m/m*) of mineral sulfate carried out in seven laboratories, have given the statistical results given in table 3.

Table 3 — Precision

Mineral sulfate content Mean value	0,16 % (*m/m*)	1,10 % (*m/m*)
Standard deviation of repeatability, σ_r	0,02	0,04
Standard deviation of reproducibility, σ_R	0,04	0,16

10 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 ISO 607 to which reference is made, or regarded as optional, as well as any incidents likely to have affected the results.