International Standard 6835

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

Surface active agents — Washing powders — Determination of total boron content — Titrimetric method

Agents de surface — Poudres à laver — Dosage du bore total — Méthode titrimétrique

First edition — 1981-04-01

UDC 661.185 : 543.24 : 546.27 **Ref. No. ISO 6835-1981 (E)**

Descriptors : surfactants, tests, determination of content, boron, chemical analysis, titration.

Price based on 3 pages

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 6835 was developed by Technical Committee ISO/TC 91, *Surface active agents*, and was circulated to the member bodies in January 1980.

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

Australia	Germany, F.R.	South Africa, Rep. of
Austria	Hungary	Spain
Belgium	India	Switzerland
China	Japan	USA
Egypt, Arab Rep. of	Korea, Rep. of	USSR
France	Romania	

The member body of the following country expressed disapproval of the document on technical grounds :

Netherlands

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC)

Printed in Switzerland

Surface active agents — Washing powders — Determination of total boron content — Titrimetric method

1 Scope and field of application

This International Standard specifies a titrimetric method for the rapid determination of the total boron content of commercial washing powders, without interference from other compounds usually present.

The method may be used in the presence of sequestering agents.

2 References

ISO/R 385, *Burettes.*

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

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

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

3 Principle

Removal of phosphates from an aqueous solution of a washing powder by passing over an ion-exchange resin, followed by formation and titration of the boric acid-mannitol complex.

4 Reagents

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

4.1 *D*-**mannitol.**

4.2 Hydrochloric acid, 100 g/l solution.

Place 25 ml of hydrochloric acid (ϱ_{20} approximately 1,18 g/ml) in a 100 ml volumetric flask, dilute to volume and mix.

4.3 Hydrochloric acid, 10 g/l solution.

Place 10 ml of the hydrochloric acid solution (4.2) in a 100 ml volumetric flask, dilute to volume and mix.

4.4 Sodium hydroxide, 100 g/l solution.

Dissolve 10 g of sodium hydroxide in water in a 100 ml volumetric flask, dilute to volume and mix.

4.5 Sodium hydroxide, 10 g/l solution.

Place 10 ml of the sodium hydroxide solution (4.4) in a 100 ml volumetric flask, dilute to volume and mix.

4.6 Boric acid, solution containing 0,5 g of boron oxide (B_2O_3) per litre.

Weigh, to the nearest 0,1 mg, 0,888 g of boric acid (H_3BO_3), dissolve it in water in a 1 000 ml volumetric flask and dilute to volume.

1 ml of this solution corresponds to 0,5 mg of boron oxide.

4.7 Sodium hydroxide, standard volumetric solution, c(NaOH) ≈ 0,05 mol/l.

4.7.1 Standardization

By means of a pipette (5.3), place 25,0 ml of the boric acid solution (4.6) in a 150 ml beaker, add 75 ml of water, and proceed as specified in 7.2.3.

4.7.2 Calculation of concentration

The concentration, c, expressed in moles of NaOH per litre, is given by the formula

$$c = \frac{0{,}012\,5}{V_0 \times 0{,}034\,8}$$

where V_0 is the volume, in millilitres, of sodium hydroxide solution used for the titration of 0,012 5 g of boron oxide.

4.8 Anion-exchange resin, strongly basic, (quaternary ammonium group), (Cl^-) form, with a rate of reticulation of 4 % of divinylbenzene, and of particle size range 150 to 300 µm.[1)]

5 Apparatus

Ordinary laboratory apparatus, and in particular :

5.1 Beaker, made of polyethylene, of capacity 2 000 ml.

5.2 Volumetric flasks, of capacity 250 and 1 000 ml, complying with the requirements of ISO 1042.

5.3 Pipettes, of capacity 25 and 200 ml, complying with the requirements of ISO 648.

5.4 Burette, of capacity 10 ml, complying with the requirements of ISO/R 385.

5.5 pH-meter, provided with an extended scale, accurate to 0,05 pH unit.

5.6 Glass electrode.

5.7 Calomel electrode.

5.8 Ion-exchange column, of length 30 cm, internal diameter 1,5 cm, with a reservoir of capacity 75 ml.

The column shall be filled with the resin (4.8) to a length of 22 cm.

5.9 Vacuum filter, of capacity 250 ml.[2)]

5.10 Filter medium, high retention filter paper or equivalent.[2)]

5.11 Mechanical stirrer.

5.12 Magnetic stirrer.

6 Sampling

The washing powder laboratory sample shall be prepared and stored in accordance with ISO 607.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,01 g, about 10 g of the laboratory sample.

7.2 Determination

7.2.1 Preparation of the test solution

Transfer the test portion (7.1) to the beaker (5.1). Fill the 1 000 ml volumetric flask (5.2) to volume with water at 35 to 40 °C, and pour the water on to the test portion, allowing a few seconds for drainage.

Stir vigorously, by means of the stirrer (5.11), for about 3 min in order to dissolve the test portion. Small quantities of insoluble silicates (and other salts) may remain undissolved.

7.2.2 Separation by anionic exchange resin

By means of a pipette (5.3), transfer 200 ml of the test solution (7.2.1) to a 250 ml beaker and, using the hydrochloric acid solutions (4.2 and 4.3), adjust the pH to 3 ± 0,1, checking by means of the pH-meter (5.5) fitted with the glass electrode (5.6) and the calomel electrode (5.7). Transfer this solution quantitatively to the 250 ml volumetric flask (5.2) and dilute to volume.

Transfer 50 ml of this solution to the vacuum filter (5.9) and filter through the filter medium (5.10); the filtrate shall be as clear as possible to avoid any subsequent blocking.

Wash the filled ion-exchange column (5.8) with water until the washings are neutral. By means of a pipette (5.3), transfer 25 ml of the filtrate to the reservoir of the column and allow it to flow through the column at a rate of 1 to 1,5 ml/min, collecting the eluate in a 150 ml tall-form beaker. Wash the column three times with 25 ml portions of water, allowing the water to flow at a rate of 1 to 1,5 ml/min, and collect the washings in the beaker.

Regenerate the resin after each passage of the test solution by passing 100 ml of hydrochloric acid solution diluted (1 + 4), [1 volume of hydrochloric acid (ϱ_{20} approximately 1,18 g/ml) + 4 volumes of water], at a rate of 1 to 1,5 ml/min, and washing with water, at the same rate, until the washings have a pH equal to or greater than 5.

When not in use, the column should be filled with hydrochloric acid solution diluted (1 + 4).

7.2.3 Titration

By means of the magnetic stirrer (5.10), stir the eluate in the beaker and adjust the pH to 5,80 ± 0,05, checking by means of the pH-meter (5.5) fitted with the electrodes (5.6 and 5.7), using, as appropriate, the hydrochloric acid solutions (4.2 and 4.3), or the sodium hydroxide solutions (4.4 and 4.5). Add 12 g of the *D*-mannitol (4.1).

After stirring for 1 min, titrate with the sodium hydroxide solution (4.7), using the burette (5.4), until a pH of 5,80 ± 0,05 is obtained, measured using the normal scale, then the extended scale, of the pH-meter.

1) A suitable resin is available commercially. Details may be obtained from the Secretariat of ISO/TC 91 (AFNOR) or ISO Central Secretariat.

2) Suitable apparatus is available commercially. Details may be obtained from the Secretariat of ISO/TC 91 (AFNOR) or ISO Central Secretariat.

The volume of the sodium hydroxide solution used to return to the initial pH of 5,8 corresponds to the quantity of boric acid present in the aliquot portion.

8 Expression of results

8.1 Method of calculation

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

$$\frac{V \times c \times 0{,}034\,8}{m} \times \frac{1\,000}{200} \times \frac{250}{25} \times 100$$

$$= \frac{V \times c \times 174}{m}$$

where

V is the volume, in millilitres, of the sodium hydroxide solution (4.7), used for the titration of the 25 ml aliquot portion of the test solution (7.2.1);

c is the exact concentration, in moles of NaOH per litre, of the sodium hydroxide solution (4.7);

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

0,034 8 is the mass, in grams, of boron oxide corresponding to 1 ml of exactly 1 mol/l sodium hydroxide solution.

By replacing c in the above formula by the expression given in 4.7.2, one obtains the formula

$$\frac{V \times 62{,}5}{V_0 \times m}$$

8.2 Precision

Comparative analyses, on samples of two washing powders containing respectively 10 % (m/m) of perborate [about 2 % (m/m) of B_2O_3] and 25 % (m/m) of perborate [about 5,7 % (m/m) of B_2O_3] carried out in eight laboratories, have given the statistical results given in the following table.

Table

Boron oxide content	2 % (m/m)	5,7 % (m/m)
Standard deviation of repeatability, σ_r	0,05	0,08
Standard deviation of reproducibility, σ_R	0,14	0,08

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

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