BRITISH STANDARD

BS 3762-3.22:
1986
ISO 8214:1985

Analysis of formulated detergents —

Part 3: Quantitative test methods —

Section 3.22 Method for determination of inorganic sulphate content

[ISO title: Surface active agents — Washing powders — Determination of inorganic sulfates — Gravimetric method]

NOTE It is recommended that this Section be read in conjunction with the information in the "*General Introduction*", published separately as BS 3762-0.

UDC 661.185:543

This British Standard, having been prepared under the direction of the Chemicals Standards Committee, was published under the authority of the Board of BSI and comes into effect on 30 September 1986

The committees responsible for this British Standard are shown in Part 0.

The following BSI references relate to the work on this standard:
Committee reference CIC/34
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Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

National foreword

This Section of BS 3762 has been prepared under the direction of the Chemicals Standards Committee. It is identical with ISO 8214:1985 "*Surface active agents — Washing powders — Determination of inorganic sulfates — Gravimetric method*", published by the International Organization for Standardization (ISO).

This method supersedes method D13 of BS 3762:1964, which has been deleted by amendment.

Terminology and conventions. The text of the International Standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards, it is current practice to use a full point on the baseline as the decimal marker.

In British Standards it is current practice to use the symbol "L" for litre (and in its submultiples) rather than "l", and to use the spelling "sulphur", etc., instead of "sulfur", etc.

Wherever the words "International Standard" appear, referring to this standard, they should be read as "Section of this British Standard".

Cross-references

International Standard	Corresponding British Standard
ISO 607:1980	BS 3762 *Analysis of formulated detergents* Part 1:1983 *Methods of sample division* (Identical)
ISO 1042:1981	BS 1792:1982 *Specification for one-mark volumetric flasks* (Identical)
ISO 8215:1985	BS 3762 *Analysis of formulated detergents* Section 3.21:1986 *Method for determination of total silica content* (Identical)

Additional information

Reagents. With reference to clause **4**, water complying with BS 3978 "*Water for laboratory use*" is suitable.

This standard describes a method of test only and should not be referred to as a specification defining limits of purity. Reference to the standard should indicate that the method of test used is in conformity with BS 3762-3.22.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 4, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope and field of application

This International Standard specifies a gravimetric method for the determination of inorganic sulfates content of all types of commercial washing powders, without interference from other compounds usually present.

2 Reference

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

3 Principle

Removal of all the ethanol-soluble matter from a test portion by extraction with ethanol.

In the presence of silicates, filtration after dehydration then precipitation of sulfates present in the filtrate with barium chloride. Filtration of the precipitate, washing, heating at 900 °C and weighing.

NOTE The residue obtained after removal of silicates may be used for the determination of total silica according to ISO 8215.

4 Reagents

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

4.1 *Ethanol*, anhydrous or denatured.

4.2 *Hydrochloric acid*, ϱ 1,16 to 1,19 g/ml.

4.3 *Ammonia*, concentrated solution.

4.4 *Barium chloride dihydrate* ($BaCl_2,2H_2O$), 100 g/l solution.

4.5 *Silver nitrate*, 5 g/l solution.

4.6 *Methyl orange*, 1 g/l solution.

4.7 *Pumice stones*, particle size 2 to 4 mm, or equivalent as boiling aid.

5 Apparatus

Ordinary laboratory apparatus and

5.1 *One-mark volumetric flasks*, of capacity 1 000 ml, complying with the requirements of ISO 1042.

5.2 *Soxhlet extractor*, with flask of capacity 500 ml, and extractor tube of capacity 200 ml (see the Figure).

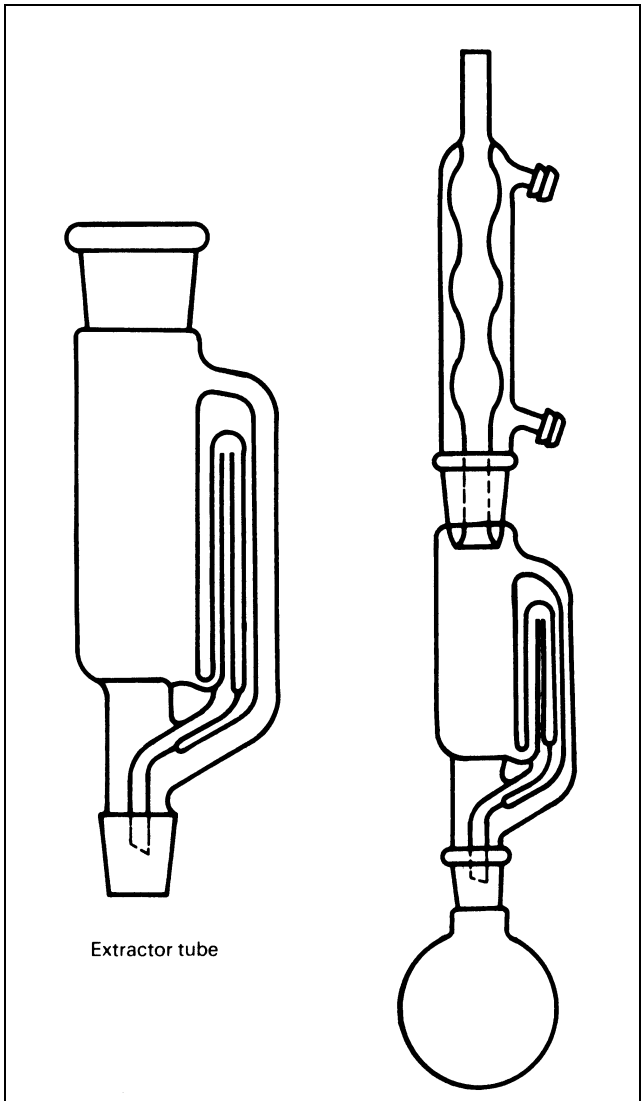

Figure — Soxhlet extractor

5.3 *Glass thimble extractor*, of porosity P 1,6 (1,6 µm), diameter about 36 mm, length about 95 mm; when a silicate determination is not required, an equivalent paper extraction thimble can be used.

5.4 *Oven*, capable of being controlled at 105 ± 2 °C.

5.5 *Filtering crucible*, in porcelain, porosity P 4 (1,6 to 4 µm).

5.6 *Platinum crucible*

5.7 *Furnace*, capable of being controlled at 900 °C.

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 in a 600 ml beaker or in the extraction thimble (**5.3**).

7.2 Removal of organic materials

One of the two following procedures may be used:

7.2.1 *Soxhlet extraction*

Introduce 300 ml of the ethanol (**4.1**) into the 500 ml round-bottom flask of the Soxhlet extractor (**5.2**) and a few pumice stones (**4.7**).

Place the thimble (**5.3**) with the test portion (**7.1**) in the extractor tube of the Soxhlet extractor and assemble the equipment (flask, extractor tube, condenser).

Start the extraction and continue with a fairly rapid rate of extraction for 2 h 30 min after the initial siphoning.

Allow to cool, and transfer the remaining ethanol of the extractor to the flask and discard the ethanol-soluble fraction.

7.2.2 *Extraction by treatment in beaker*

Add approximately 250 ml of ethanol (**4.1**) to the test portion (**7.1**).

Cover with a watch-glass, heat and stir with a mechanical or magnetic stirrer until the ethanol is boiling.

Continue boiling and stirring for 5 min.

Allow the beaker to cool and the insoluble matter to settle. Filter the ethanolic phase through a medium-grade filter paper. Repeat this extraction twice more with new portions of the ethanol (**4.1**) using the same filter paper.

Add approximately 75 ml of the hot ethanol (50 to 60 °C) to the beaker containing the insoluble matter and break any remaining hard lumps with a glass rod. Allow the insoluble matter to settle and filter through the same filter paper.

Repeat this operation twice more.

Puncture the bottom of the filter paper and wash with about 50 ml of hot water to transfer any residue to the beaker containing the insoluble matter.

7.3 Removal of silicates

After extraction (**7.2.1**), remove the thimble from the Soxhlet extractor (**5.2**) and, using hot water (50 to 75 ml), quantitatively transfer the contents to a 400 ml beaker; or use the 600 ml beaker and alcohol-insoluble matter obtained as specified in **7.2.2**.

Add 10 ml of the hydrochloric acid (**4.2**) to the beaker. Stir with a glass rod.

Evaporate to dryness on a steam bath.

Add 35 to 40 ml of water. Heat, with occasional stirring, for 10 min. If silica and insoluble matter are absent, proceed as specified in **7.4**; otherwise, continue as follows.

Again add 10 ml of the hydrochloric acid (**4.2**), stir and evaporate to dryness as before. Dissolve the residue, add 10 ml of hydrochloric acid (**4.2**), stir, and evaporate to dryness a third time. Place the beaker and residue in the oven (**5.4**), maintained at 105 ± 2 °C, for 1 h. Add 50 ml of hot water and 10 ml of the hydrochloric acid (**4.2**). Heat for 10 min on a steam bath, with occasional stirring.

Filter through the tared porcelain filtering crucible (**5.5**) under suction or through a fast-running hardened filter paper.

Wash the residue four times with 30 ml portions of hot water.

NOTE The insoluble residue may be used for the determination of total silica according to ISO 8215; in this case, change the filtrate receiver at this point and continue the transference and washing of the residue as specified in ISO 8215.

7.4 Determination

Quantitatively transfer the filtrate and first four washings (from **7.3**) to a 1 000 ml one-mark volumetric flask; or transfer the solution if silica and insoluble matter are absent.

Dilute to volume and mix.

By means of a pipette, transfer an aliquot volume of the solution to a beaker, taking 200 ml for sulfate contents of less than 6 % (*m*/*m*) (calculated as Na_2SO_4) and for higher contents taking a volume corresponding to a mass of barium sulfate of between 0,15 and 0,30 g.

Dilute to 200 ml if necessary. Add four drops of the methyl orange solution (**4.6**) and neutralize with the ammonia solution (**4.3**).

Add the hydrochloric acid (**4.2**) until just acid and then add 5,0 ml in excess.

Heat to boiling and slowly add 5 ml of the barium chloride solution (**4.4**) while boiling. Cover with a watch-glass and boil gently for 5 min.

Place on a steam bath for a minimum 1 h at 70 to 80 °C.

Test for completeness of precipitation by adding a few drops of the barium chloride solution (**4.4**).

Filter through the tared porcelain filtering crucible (**5.5**) under vacuum or through an ashless grade medium or fine filter paper. Before taring, heat the porcelain crucible in the furnace (**5.7**), controlled at 900 °C, and allow to cool in a desiccator.

Wash the precipitate on to the filter with hot water and continue washing until free of chlorides as shown by testing with a few drops of the silver nitrate solution (**4.5**).

In the case of a filter paper, place it in the platinum crucible (**5.6**), previously tared after heating in the furnace (**5.7**), controlled at 900 °C, and allowing to cool in a desiccator.

Gradually heat the crucible and contents to 900 °C, then leave in the furnace (**5.7**), controlled at 900 °C, for 30 min. Allow to cool in a desiccator and weigh to the nearest 0,001 g.

8 Expression of results

8.1 Method of calculation

The inorganic sulfate content, expressed as a percentage by mass of sodium sulfate (Na_2SO_4), is given by the formula

$$\frac{m_1 \times 1\,000 \times 0{,}608\,6}{m_0 \times V} \times 100 = \frac{60\,860\ m_1}{m_0 V}$$

where

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

m_1 is the mass, in grams, of the barium sulfate precipitate;

V is the volume, in millilitres, of the aliquot portion taken;

0,608 6 is the conversion factor for $BaSO_4$ to Na_2SO_4.

8.2 Precision

Comparative analysis on three samples ranging from 6 % to 15 % Na_2SO_4, carried out in 11 laboratories, has given the statistical results shown in the following table.

Sulfate content (Na_2SO_4), x	6 to 15 % (m/m)
Repeatability	$0{,}05\ \sqrt{x}$
Reproducibility	$0{,}20\ \sqrt{x}$

9 Test report

The test report shall include the following particulars:

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 operational details not included 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.

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Publications referred to

See national foreword.

BS 3762-3.22: 1986
ISO 8214:1985

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London
W4 4AL