

Analysis of soaps —

Part 1: General introduction, sampling, and test for presence of synthetic anionic-active surface active agents

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Committees responsible for this British Standard

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Chemical Industries Association
 Chemical Industries Association (GOSIP)
 Consumer Policy Committee of BSI
 Department of Trade and Industry (Laboratory of the Government Chemist)
 Ministry of Defence
 Royal Society of Chemistry
 Soap and Detergent Industry Association
 Society of Dyers and Colourists

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Foreword

This Part of BS 1715 has been prepared under the direction of the Chemicals Standards Policy Committee. It is a revision of BS 1715-1:1989, which is withdrawn. BS 1715 provides a series of analytical methods for soaps, other than those with added inorganic materials (e.g. sodium silicate, sodium carbonate, sodium phosphates, sodium perborate and abrasives), which may be analysed by other methods in the Parts and Sections of BS 3762.

BS 1715 is published in two Parts, the analytical methods in Part 2 being published separately as Sections.

In preparing this British Standard, the opportunity was taken, if appropriate, of implementing, preferably without alteration, international standards describing test methods for soaps. These were prepared, with the active participation of the UK, by Technical Committee 91, "Surface active agents", of the International Organization for Standardization (ISO). In this Part of BS 1715, Table 1 lists the Parts and Sections of BS 1715 and the analytical methods included therein and gives, if appropriate, their relationships to the corresponding international standards.

It is intended that work should continue with the object of improving existing test methods and preparing others for publication as further Sections of BS 1715.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 and 2, 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

This Part of BS 1715 outlines, in Table 1, the present structure of BS 1715 and describes methods of sample preparation and a qualitative test for presence of synthetic anionic-active surface active agents.

NOTE The titles of the publications referred to in this standard are listed on the inside back cover.

2 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and water complying with grade 3 of BS 3978.

3 Sampling

NOTE 1 It is not possible to give instructions which will adequately cover all instances of sampling of soap, and frequently the procedure adopted will be dictated by the experience and judgement of the authority responsible for the sampling.

Place the samples immediately in an air-tight container, taking care to ensure that the sample remains in its original condition as far as possible.

NOTE 2 Lever-lid tins and screw-capped glass bottles are considered to be satisfactory containers.

NOTE 3 Analysis should follow sampling as soon as possible.

4 Preparation of samples

4.1 Bars, cakes or tablets

Cut the bar, cake or tablet into eight parts by three cuts at right angles to each other and intersecting at the centre. Slice or grate finely the whole of two diagonally opposite eighths, mix thoroughly and place in an air-tight container.

4.2 Soft soap

Mix the sample well by kneading and weigh all portions for analysis at one time, preserving the remainder in an air-tight container.

Table 1 — Methods of analysis of soaps: structure and relationship between BS 1715 and international standards

BS 1715		Subject	Corresponding international standard	Relationship of international standard to BS test method
Part	Section number			
1. General introduction, sampling, and test for presence of synthetic anionic-active surface active agents	—	Introduction, sample preparation, qualitative test	—	—
2. Quantitative test methods	2.1	Total alkali and total fatty matter content	685	Identical
	2.2	Total free alkali content	684	Identical
	2.3	Free caustic alkali content	456	Identical
	2.4	Free fatty acids content	—	—
	2.5	Unsaponifiable, unsaponified, and unsaponified saponifiable matter contents	1067	Identical
	2.6	Moisture and volatile matter content	672	Identical
	2.7	Chloride content	4323	Identical
	2.8	Glycerol content	1066	Identical
	2.9	EDTA content	4325	Related
	2.10	Not allotted	—	—
	2.11	Ethanol-insoluble matter content	673	Identical

4.3 Other samples

Take portions for analysis direct from the well-mixed sample.

5 Test for presence of synthetic anionic-active surface active agents

5.1 Reagents

5.1.1 Chloroform

5.1.2 Sulphuric acid solution, $c(\text{H}_2\text{SO}_4) = 2.5 \text{ mol/L}$.

5.1.3 Mixed indicator

5.1.3.1 *Dimidium bromide-acid blue 1 stock solution*. This solution is prepared from dimidium bromide (3,8-diamino-5-methyl-6-phenyl-phenanthridinium bromide), and acid blue 1 (CI 42045) (sodium α -[4-(diethylamino)phenyl]- α -[4-(diethylamino)cyclohexa-2,5-dienylidene] toluene-2,4-disulphonate).

NOTE This solution is available commercially under the name "Dimidium bromide-disulphine blue indicator stock solution".

Weigh $0.5 \pm 0.005 \text{ g}$ of dimidium bromide into a 50 mL beaker and $0.25 \pm 0.005 \text{ g}$ of acid blue 1 into a second 50 mL beaker. Add between 20 mL and 30 mL of hot 10 % (V/V) ethanol solution in water to each beaker. Stir until dissolved and transfer the solutions to a 250 mL one-mark volumetric flask. Rinse the beakers into the volumetric flask with the ethanol solution and dilute to the mark with 10 % (V/V) ethanol solution.

5.1.3.2 *Mixed acid indicator solution*. Add 200 mL of water to 20 mL of the stock solution (5.1.3.1) in a 500 mL one-mark volumetric flask. Add 20 mL of the sulphuric acid solution (5.1.2), mix and dilute to the mark with water. Store out of direct sunlight.

5.2 Procedure

Dissolve approximately 0.3 g of the sample (see clause 4) in 20 mL of water. Add 1 mL of the sulphuric acid solution (5.1.2), 5 mL of the mixed acid indicator solution (5.1.3.2) and 5 mL of the chloroform (5.1.1). Shake well.

A distinct pink colour in the chloroform layer denotes the presence of a synthetic anionic-active surface active agent.

6 Other methods

In addition to the methods listed in Table 1, other British Standards appropriate for the analysis of soaps include BS 684-2.34 and BS 684-2.35.

Publication(s) referred to

BS 684, *Methods of analysis of fats and fatty oils.*

BS 684-2.34, *Preparation of methyl esters of fatty acids.*

BS 684-2.35, *Analysis by gas-liquid chromatography of methyl esters of fatty acids.*

BS 3762, *Analysis of formulated detergents¹⁾.*

BS 3978, *Specification for water for laboratory use.*

ISO 456, *Surface active agents — Analysis of soaps — Determination of free caustic alkali.*

ISO 672, *Soaps — Determination of moisture and volatile matter content — Oven method.*

ISO 673, *Soaps — Determination of content of ethanol—insoluble matter.*

ISO 684, *Analysis of soaps — Determination of total free alkali.*

ISO 685, *Analysis of soaps — Determination of total alkali content and total fatty matter content.*

ISO 1066, *Analysis of soaps — Determination of glycerol content — Titrimetric method.*

ISO 1067, *Analysis of soaps — Determination of unsaponifiable, unsaponified and unsaponified saponifiable matter.*

ISO 4323, *Soaps — Determination of chlorides content — Potentiometric method.*

ISO 4325, *Soaps and detergents — Determination of chelating agent content — Titrimetric method.*

¹⁾ Referred to in the foreword only.

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