BRITISH STANDARD

BS 1715-2.7:
1989
ISO 4323:1977

Analysis of soaps —

Part 2: Quantitative test methods —

Section 2.7 Method for determination of chloride content

[ISO title: Soaps — Determination of chloride content — Potentiometric method]

UDC 661.18:543

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Chemicals Standards Policy Committee (CIC/-) to Technical Committee CIC/34, upon which the following bodies were represented:

Chemical Industries Association
Consumer Policy Committee of BSI
Department of the Environment
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

This British Standard, having been prepared under the direction of the Chemicals Standards Policy Committee, was published under the authority of the Board of BSI and comes into effect on 31 July 1989

The following BSI references relate to the work on this standard:
Committee reference CIC/34
Draft for comment 88/53275 DC

ISBN 0 580 17323 2

Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

National foreword

This Section of BS 1715 has been prepared under the direction of the Chemicals Standards Policy Committee. It is identical with ISO 4323:1977 "*Soaps — Determination of chloride content — Potentiometric method*" published by the International Organization for Standardization (ISO).

This method supersedes the method "*Determination of chlorides*" given in BS 1715:1963, which is 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.

The symbol "l" has been used to denote litre (and in its submultiples). In British Standards it is current practice to use the symbol "L".

Wherever the words "International Standard" appear, referring to this standard, they should be read as "Section of BS 1715".

Cross references. The International Standard for sampling of soaps (see clause **2** and clause **6**) is not yet published. Relevant information is included in BS 1715 "*Analysis of soaps*", Part 1:1989 "*General introduction, sampling, and test for presence of synthetic anionic-active surface active agents*".

ISO/R 385 (see **5.5**) has been revised as ISO 385-1:1984, ISO 385-2:1984 and ISO 385-3:1984; a related British Standard is BS 846:1985 "*Specification for burettes*".

Additional information. With reference to clause **4**, water complying with grade 3 of BS 3978 "*Specification for water for laboratory use*" is suitable.

With reference to **4.2**, the nitric acid solution, expressed as an amount-of-substance concentration, is $c(HNO_3) = 6$ mol/L. With reference to **4.3** and **4.4**, the silver nitrate solutions, expressed as amount-of-substance concentrations, are $c(AgNO_3) = 0.1$ mol/L and $c(AgNO_3) = 0.01$ mol/L respectively. With reference to **4.5** and **4.6**, the potassium chloride standard reference solutions, expressed as amount-of-substance concentrations, are $c(KCl) = 0.1$ mol/L and $c(KCl) = 0.01$ mol/L respectively.

In **7.3.3** and clause **8**, "normality" should be read as "amount-of-substance concentration (mol/L)".

This Section describes a method of test only, and should not be used or quoted as a specification defining limits of purity. Reference to this Section should indicate that the method of test used is in accordance with BS 1715-2.7.

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 potentiometric method for the determination of the chloride content of commercial soaps, containing or not containing other surface active agents, and also of compounded products.

2 Reference

ISO ..., *Soaps — Sampling*[1].

3 Principle

Potentiometric titration of the chloride (Cl^-) ions with standard volumetric silver nitrate solution in a nitric acid medium, using a silver-silver chloride measurement electrode and a calomel reference electrode.

4 Reagents

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

4.1 *Potassium nitrate,* solution saturated at 20 °C.

4.2 *Nitric acid,* approximately 6 N solution.

4.3 *Silver nitrate,* approximately 0,1 N standard volumetric solution.

Dissolve 8,5 g of silver nitrate in water in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Store this solution in a dark amber-coloured flask.

4.4 *Silver nitrate,* approximately 0,01 N solution.

Prepare this solution immediately before use by appropriate dilution of the standard volumetric silver nitrate solution (**4.3**).

4.5 *Potassium chloride,* 0,1 N standard reference solution.

Weigh, to the nearest 0,001 g, 3,728 g of potassium chloride, previously dried for 2 h at 105 °C and cooled in a desiccator. Dissolve in a small quantity of water and transfer quantitatively to a 500 ml one-mark volumetric flask. Dilute to the mark and mix.

4.6 *Potassium chloride,* 0,01 N standard reference solution.

Prepare this solution immediately before use by appropriate dilution of the standard reference potassium chloride solution (**4.5**).

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

5 Apparatus

Ordinary laboratory apparatus, and

5.1 *Potentiometer,* sensitivity 2 mV, covering the range – 500 to + 500 mV.

5.2 *Electrodes*

5.2.1 *Calomel electrode — KCl saturated*

5.2.2 *Silver-silver chloride electrode*

5.2.3 *Bridge,* containing the saturated potassium nitrate solution (**4.1**), connected to the calomel electrode (**5.2.1**).

5.3 *Combined electrode,* as an alternative to the calomel electrode (**5.2.1**) and the silver electrode (**5.2.2**).

5.4 *Electromagnetic stirrer*

5.5 *Burette,* capacity 50 ml, complying with the requirements of ISO/R 385, Class A.

6 Sampling

The laboratory sample of soap shall be prepared and stored according to the procedure specified in ISO ...

7 Procedure

7.1 Measurement temperature

In order to reduce the effects of thermal and electric hysteresis, take care that the temperatures of the electrodes, the water used for washings, the standard solutions and the test solution are as close to each other as possible. The temperatures of the standard solutions and the test solution shall not differ by more than 1 °C. The measurement temperature should be 20 °C whenever possible.

7.2 Test portion and preparation of test solution

Select the reagent solutions and test portion according to the expected chloride content, as indicated in the following table:

Expected chloride content expressed as NaCl % (*m*/*m*)	Silver nitrate solution	Standard reference potassium chloride solution	Mass of test portion
Below 0,1	0,01 N (**4.4**)	0,01 N (**4.6**)	1 to 10 g
Above 0,1	0,1 N (**4.3**)	0,1 N (**4.5**)	1 to 3 g

[1] In preparation.

Weigh, to the nearest 0,001 g, the appropriate test portion and dissolve in 50 to 100 ml of hot water. Add 2 drops of the methyl orange solution (**4.7**), acidify with the nitric acid solution (**4.2**) and add several drops in excess. Filter through a wet filter paper and wash the fatty acids with small portions of hot water. Cool the filtrate, i.e. the test solution for titration, to 20 °C.

7.3 Calibration of the silver nitrate solution

7.3.1 *Preparation of the apparatus*

Assemble the apparatus and switch it on. Allow it to operate, according to the manufacturer's instructions, for a sufficient time to obtain a good electric stabilization before beginning the measurements. Take care that the interior liquid of the KCl-saturated calomel electrode (**5.2.1**) is in equilibrium with the atmospheric pressure, in order that its outflow across the bridge (**5.2.3**) is not obstructed.

Note the temperature of the standard reference solutions, make the corresponding adjustments in the circuit for correction of temperatures, and verify the zero of the apparatus. Do not alter the settings during the measurements.

7.3.2 *Titration*

Take 5,00 ml and 10,00 ml respectively of the appropriate standard reference potassium chloride solution (**4.5** or **4.6**) and place in two clean, dry vessels of convenient capacity (for example 150 ml). Carry out the following titration on the contents of each vessel.

After acidification by the nitric acid solution (**4.2**), add sufficient water to bring the total volume to about 100 ml.

Stir the resultant solution and immerse the combined electrode (**5.3**) or the silver-silver chloride electrode (**5.2.2**) and the free end of the bridge (**5.2.3**) in the solution, connect the electrode to the potentiometer (**5.1**) and, after having verified the zero of the apparatus, note the value of the starting potential.

Add, from the burette (**5.5**), in 1 ml portions, the silver nitrate solution (**4.3** or **4.4**) having the same normality as that of the standard reference potassium chloride solution (**4.5** or **4.6**) used. After each addition, await the stabilization of the potential.

Record the volumes added and the corresponding values of the potential in the first two columns of a table.

When approaching the end-point, continue the addition of the silver nitrate solution in portions of 0,1 ml for the 0,01 N solution or 0,05 ml for the 0,1 N solution.

In a third column of the table, record the successive increments $\Delta_1 E$ of the potential E. In a fourth column, record the differences $\Delta_2 E$, positive or negative, between the potential increments $\Delta_1 E$.

The end of the titration corresponds to the addition of the 0,1 ml or 0,05 ml portion of the silver nitrate solution which gives the maximum value of $\Delta_1 E$.

In order to calculate the exact volume V_{EQ} of the silver nitrate solution corresponding to the end of the reaction, use the formula:

$$V_{EQ} = V_0 + V_1 \times \frac{b}{B}$$

where

V_0 is the volume, in millilitres, of the silver nitrate solution (**4.3** or **4.4**), immediately lower than the volume which gives the maximum increment of $\Delta_1 E$;

V_1 is the volume, in millilitres, of the final portion of silver nitrate solution (**4.3** or **4.4**) added (0,05 or 0,1 ml respectively);

b is the last value of $\Delta_2 E$ which is positive;

B is the sum of the absolute values of the final positive value of $\Delta_2 E$ and the first negative value of $\Delta_2 E$ (see example in Annex).

7.3.3 *Calculation of normality of solution*

The normality T of the silver nitrate solution is given by the formula

$$T = T_0 \times \frac{5}{V_2 - V_3}$$

where

T_0 is the normality of the standard reference potassium chloride solution (**4.5** or **4.6**);

V_2 is the value, in millilitres, of V_{EQ}, corresponding to the titration of 10 ml of the standard reference potassium chloride solution (**4.5** or **4.6**);

V_3 is the value, in millilitres, of V_{EQ}, corresponding to the titration of 5 ml of the standard reference potassium chloride solution (**4.5** or **4.6**);

5 is the difference, in millilitres, between the two volumes of standard reference potassium chloride solution (**4.5** or **4.6**) used.

7.4 Blank test

Carry out a blank test without the test portion.

The value of the blank test on the reagents, V_4, is given, in millilitres, by the formula

$$V_4 = 2V_3 - V_2$$

where V_2 and V_3 are as defined in **7.3.3**.

7.5 Determination

Titrate the test solution (**7.2**) with the silver nitrate solution (**4.3** or **4.4**) corresponding to the expected chloride content and note the end-point of the reaction in accordance with the instructions given in **7.3**.

8 Expression of results

The chloride content is given, as a percentage by mass of sodium chloride (NaCl), by the formula

$$(V_5 - V_4) \times T \times 0{,}058\ 5 \times \frac{100}{m} = \frac{5{,}85\ T(V_5 - V_4)}{m}$$

where

T is the normality of the silver nitrate solution, calculated in accordance with **7.3.3**;

V_4 is the volume, in millilitres, corresponding to the blank test (**7.4**);

V_5 is the value, in millilitres, of V_{EQ}, corresponding to the determination (**7.5**);

m is the mass, in grams, of the test portion (see **7.2**).

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 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 specified in this International Standard or the International Standard to which reference is made, or regarded as optional, as well as all incidents likely to have affected the results.

Annex Example

Volume of silver nitrate solution (4.4) V	**Potential** E	$\Delta_1 E$	$\Delta_2 E$
ml	mV		
0,80	176		
0,90	211	35	
1,00	283	72	+ 37
1,10	306	23	− 49
1,20	319	13	− 10
$V_{EQ} = 0,9 + 0,1 \times \frac{37}{37 + 49} = 0,943$			

Publications referred to

See national foreword.

BS 1715-2.7: 1989
ISO 4323:1977

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