

Analysis of soaps —

Part 2: Quantitative test methods —

Section 2.3 Methods for determination of free caustic alkali content

[ISO title: Surface active agents — Analysis of soaps —
Determination of free caustic alkali]

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Committees responsible for this British Standard

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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

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National foreword

This Section of BS 1715 has been prepared under the direction of the Chemicals Standards Policy Committee. It is identical with ISO 456:1973 “*Surface active agents — Analysis of soaps — Determination of free caustic alkali*” published by the International Organization for Standardization (ISO).

These methods supersede the method for determination of free caustic alkali 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 “ml” has been used to denote millilitre. In British Standards it is current practice to use the symbol “mL”.

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

Additional information. With reference to 4.2 and 5.2, water complying with grade 3 of BS 3978 “*Specification for water for laboratory use*” is suitable, and other reagents used should be of a recognized analytical grade.

With reference to 4.2.2, the potassium hydroxide ethanolic solution, expressed as an amount-of-substance concentration, is $c(\text{KOH}) = 0.1 \text{ mol/L}$. With reference to 4.2.3 and 5.2.1, the hydrochloric acid standard volumetric solutions, expressed as amount-of-substance concentrations, are $c(\text{HCl}) = 0.1 \text{ mol/L}$. Similarly, in 5.2.2 and 5.2.3, the sodium hydroxide or potassium hydroxide solutions, expressed as amount-of-substance concentrations, are $c(\text{NaOH})$ or $c(\text{KOH}) = 1 \text{ mol/L}$ respectively. In the definitions of T in 4.5.1 and 5.5.1 “normality” should be read as “amount-of-substance concentration (mol/L)”. In the last paragraphs of 4.5.1 and 5.5.1, “milliequivalents” should be read as “millimoles”.

For the purposes of 4.2.2, 4.2.3, 4.2.4, 5.2.2 and 5.2.4, the ethanol may be replaced by industrial methylated spirits complying with BS 3591 “*Specification for industrial methylated spirits*” of appropriate strength. It should be noted that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations, 1983 (S.I. 1983 No. 252). It is not permissible to use duty-free ethanol, received under the provisions of the Alcoholic Liquors Duties Act 1972, Section 10, for purposes for which industrial methylated spirits is an acceptable alternative.

This Section describes methods 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 1715-2.3.

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 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.

0 Introduction

As soaps usually contain a small amount of unsaponified neutral fat, there is no perfect procedure for determining free caustic alkali because, when the sample of soap is dissolved, a neutral fat is more or less saponified by any free caustic alkali that is present.

Both methods described in this International Standard are therefore of a conventional nature.

Conventionally, free caustic alkali is expressed as

- sodium hydroxide (NaOH) for sodium soaps and
- potassium hydroxide (KOH) for potassium soaps.

1 Scope

This International Standard specifies two methods of determining free caustic alkali in commercial soaps, excluding compounded products:

- Method A, ethanol method;
- Method B, barium chloride method.

2 Field of application

2.1 Method A (ethanol method) should be applied only to sodium soaps of ordinary quality, as the presence of certain additives brings in sources of error. It is not applicable to potassium soaps, because of the solubility of potassium carbonate in ethanol.

2.2 Method B (barium chloride method) should be applied to all soft potassium soaps or mixed sodium and potassium soaps. The application of this method to sodium soaps of ordinary quality, which usually do not contain sufficient quantities of free caustic alkali to be determined by this method, is not recommended.

3 Definition

free caustic alkali in a soft soap

the quantity of hydroxyl ion, reported as potassium hydroxide (KOH), which is found in solution after precipitation with barium chloride under the operating conditions described

4 Method A (Ethanol method)

4.1 Principle

The soap is dissolved in neutralized ethanol, and the free caustic alkali is titrated with an ethanolic solution of hydrochloric acid.

4.2 Reagents

The water used shall be distilled water or water of at least equivalent purity.

The reagents shall have the following properties:

4.2.1 Ethanol, absolute, $\rho_{20} = 0,792$ g/ml.

4.2.2 Potassium hydroxide, ethanolic solution, approximately 0,1 N.

4.2.3 Hydrochloric acid, standard ethanolic solution, approximately 0,1 N.

4.2.4 Phenolphthalein solution, 1 g in 100 ml of 95 % (V/V) ethanol.

4.3 Apparatus

Ordinary laboratory apparatus, and in particular

4.3.1 Flask of approximately 500 ml capacity, that can be fitted to a reflux condenser.

4.3.2 Reflux condenser

4.3.3 Analytical balance

4.4 Procedure

4.4.1 Test portion

Weigh, to the nearest of 0,01 g, approximately 5 g of soap.

4.4.2 Determination

Pour 200 ml of ethanol (4.2.1) into the flask. Connect to the reflux condenser. Bring to a gentle boil and keep at the boil for 5 min in order to remove carbon dioxide. Remove from the condenser and allow to cool to about 70 °C. Add 4 drops of phenolphthalein indicator (4.2.4). Neutralize exactly with the ethanolic solution of potassium hydroxide (4.2.2), until the indicator just turns pink. Place the test portion in the flask containing the neutralized ethanol. Connect the flask to the reflux condenser and boil gently until the soap has completely dissolved. Cool to about 70 °C. Titrate with the ethanolic solution of hydrochloric acid (4.2.3) until the colour is just perceptibly pink, identical with that obtained when the ethanol was neutralized.

4.5 Expression of results

4.5.1 Calculation and formula

The percentage of free caustic alkali in the soap, expressed as sodium hydroxide (NaOH), is

$$0,040 \times V \times T \times \frac{100}{m}$$

where

- m is the mass, in grams, of the test portion;
- V is the volume, in millilitres, of ethanolic hydrochloric acid solution (4.2.3) used;
- T is the normality of the ethanolic hydrochloric acid solution (4.2.3) used.

The free caustic alkali content can be also expressed in milliequivalents per kilogram by means of the following formula:

$$\frac{V \times T}{m}$$

4.5.2 Reproducibility

± 0,02 absolute.

5 Method B (Barium chloride method)

5.1 Principle

The soap and carbonate are precipitated with barium chloride and the residual alkalinity of the solution, representing the alkalinity of the free residual potassium hydroxide, is determined.

5.2 Reagents

The water used shall be distilled water or water of at least equivalent purity.

The reagents shall have the following properties:

5.2.1 Hydrochloric acid, standard volumetric solution, approximately 0,1 N.

5.2.2 Ethanol solution, 60 % (V/V), prepared as follows:

Mix 75 ml of carbon dioxide-free distilled water with 125 ml of carbon dioxide-free ethanol solution, 95 % (V/V) (which has been distilled over potassium hydroxide) and 1 ml of the indicator (5.2.4); neutralize to a violet colour with a 0,1 N solution of sodium hydroxide or potassium hydroxide, then heat under reflux for 10 min. Leave to cool to room temperature and neutralize with the hydrochloric acid solution (5.2.1) until the violet colour disappears.

5.2.3 Barium chloride solution, prepared by dissolving 10 g of barium chloride dihydrate ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in 90 ml of carbon dioxide-free distilled water and neutralizing to a violet colour with a 0,1 N solution of sodium hydroxide or potassium hydroxide in the presence of the indicator (5.2.4).

5.2.4 Indicator, prepared by dissolving 1 g of phenolphthalein and 0,5 g of thymol blue in 100 ml of hot ethanol solution, 95 % (V/V), and filtering the solution obtained.

5.3 Apparatus

Ordinary laboratory apparatus and in particular

5.3.1 Conical flask of 500 ml capacity, with wide neck.

5.3.2 Reflux condenser

5.3.3 Analytical balance

5.4 Procedure

5.4.1 Test portion

Weigh, to the nearest 0,01 g, approximately 4 g of soft soap into the flask (5.3.1).

5.4.2 Determination

Add 200 ml of the ethanol solution (5.2.2) and boil for 10 min under reflux. Add to this boiling solution 15 ml of the neutralized barium chloride solution (5.2.3) in small portions, mixing thoroughly. Then cool with running water to room temperature. Add 1 ml of the indicator (5.2.4) and immediately titrate with the hydrochloric acid solution (5.2.1) until the colour turns green.

5.5 Expression of results

5.5.1 Calculation and formula

The percentage of free caustic alkali in the soft soap, expressed as potassium hydroxide (KOH), is

$$0,056 \times V \times T \times \frac{100}{m}$$

where

- m is the mass, in grams, of the test portion;
- V is the volume, in millilitres, of hydrochloric acid solution (5.2.1) used;
- T is the normality of the hydrochloric acid solution (5.2.1) used.

The free caustic alkali content can be also expressed in milliequivalents per kilogram by means of the following formula:

$$\frac{V \times T}{m}$$

5.5.2 Reproducibility

± 0,05 absolute.

6 Test report

The test report shall include the following particulars:

- a) the reference of the method used;
- b) the results and the method of expression used;
- c) any unusual features noted during the determination;
- d) any operation not included in this International Standard, or regarded as optional.

Publications referred to

See national foreword.

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