INTERNATIONAL STANDARD 456

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Surface active agents — Analysis of soaps — Determination of free caustic alkali

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, International Standard ISO 456 replaces ISO Recommendation R 456-1965 drawn up by Technical Committee ISO/TC 91, *Surface active agents.*

The Member Bodies of the following countries approved the Recommendation :

Argentina	Hungary	Poland
Austria	Italy	Portugal
Canada	Japan	Romania
Chile	Korea, Rep. of	Spain
Colombia	Morocco	Switzerland
Czechoslovakia	Netherlands	United Kingdom
France	New Zealand	Yugoslavia
Germany	Norway	

The Member Body of the following country expressed disapproval of the Recommendation on technical grounds :

India

Printed in Switzerland

Surface active agents — Analysis of soaps — Determination of free caustic alkali

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 ($BaCl_2.2H_2O$) 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.

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