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INTERNATIONAL STANDARD



456

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ ORGANISATION INTERNATIONALE DE NORMALISATION

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 :

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

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

India

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