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# INTERNATIONAL STANDARD



# 684

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

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## Analysis of soaps – Determination of total free alkali

*Analyse des savons – Détermination de la teneur en alcali libre total*

First edition – 1974-11-01

ITeH STANDARD PREVIEW  
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ISO 684:1974

<https://standards.iteh.ai/catalog/standards/sist/08e77612-4d88-4994-8dd4-3ddde515c1ce/iso-684-1974>

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UDC 661.185.1 : 543.241.2

Ref. No. ISO 684-1974 (E)

**Descriptors** : surfactants, soaps, chemical analysis, determination of content, alkalis, volumetric analysis.

## 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.

International Standard ISO 684 was drawn up by Technical Committee ISO/TC 91, *Surface active agents*. It was submitted directly to the ISO Council, in accordance with clause 6.12.1 of the Directives for the technical work of ISO.

This International Standard cancels and replaces ISO Recommendation R 684-1968, which had been approved by the Member Bodies of the following countries :

Argentina	Germany	Romania
Australia	Hungary	South Africa, Rep. of
Austria	Ireland	Spain
Belgium	Israel	Switzerland
Brazil	Japan	Turkey
Canada	Netherlands	United Kingdom
Chile	New Zealand	Yugoslavia
Egypt, Arab Rep. of	Poland	
France	Portugal	

No Member Body expressed disapproval of the document.

# Analysis of soaps – Determination of total free alkali

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the total free alkali content of commercial soaps, excluding compounded products.<sup>1)</sup>

The method is not applicable if the soap contains additives (alkali silicates, etc.) which can be decomposed by sulphuric acid by the procedure specified. The method is also not applicable to coloured soaps if the colour interferes with the phenolphthalein end point.

## 2 REFERENCES

ISO 685, *Analysis of soaps – Determination of total alkali.*<sup>2)</sup>

ISO . . . , *Soaps – Sampling.*<sup>3)</sup>

## 3 DEFINITION

For the purpose of this International Standard, the following definition applies :

**total free alkali** : The sum of the free caustic alkali and the free carbonate alkali contents.

The results are generally expressed as a percentage by mass as either sodium hydroxide (NaOH) for sodium soaps or potassium hydroxide (KOH) for potassium soaps.

They may also be expressed in milliequivalents per gram.

## 4 PRINCIPLE

Dissolution of the soap in an ethanolic solution, neutralization of the free alkali with a known excess of sulphuric acid solution and back titration of the excess acid with ethanolic potassium hydroxide solution.

## 5 REAGENTS

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

**5.1 Ethanol**, 95 % (V/V) solution, free from carbon dioxide.

Reflux this solution for 5 min, cool to ambient temperature and neutralize with the ethanolic potassium hydroxide solution (5.3) in the presence of 4 drops of the phenolphthalein solution (5.4) per 200 ml of ethanol.

**5.2 Sulphuric acid**, approximately N standard volumetric solution.

**5.3 Potassium hydroxide**, approximately 0,1 N ethanolic standard volumetric solution.

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

## 6 APPARATUS

Ordinary laboratory apparatus and

**6.1 Conical flask**, capacity 250 ml, with a conical ground glass joint.

**6.2 Reflux condenser**, water-cooled, with a conical ground glass joint at the bottom.

## 7 SAMPLING

Laboratory samples shall be prepared and stored according to the procedures specified in ISO . . .

1) See also ISO 685.

2) At present at the stage of draft. (Revision of ISO/R 685.)

3) In preparation.

**8 PROCEDURE**

**8.1 Test portion**

Weigh, to the nearest 0,001 g, about 5 g of the laboratory sample into the conical flask (6.1).

**8.2 Determination**

Add 100 ml of the ethanol solution (5.1) to the test portion (8.1).

Fit the conical flask (6.1) to the reflux condenser (6.2). Gently heat until the soap is completely dissolved, then add exactly 3,0 ml<sup>1)</sup> of the sulphuric acid solution (5.2) and boil gently for at least 10 min. Allow to cool to ambient temperature.

Titrate with the ethanolic potassium hydroxide solution (5.3) in the presence of the indicator (5.4).

Carry out two determinations on the same sample.

**9 EXPRESSION OF RESULTS**

**9.1 Method of calculation and formulae**

The total free alkali content, expressed as a percentage by mass of sodium hydroxide (NaOH) in the case of sodium soaps, is given by the formula :

$$0,040 \times \frac{V_0 T_0 - V_1 T_1}{m} \times 100$$

The total free alkali content, expressed as a percentage by mass of potassium hydroxide (KOH) in the case of potassium soaps, is given by the formula :

$$0,056 \times \frac{V_0 T_0 - V_1 T_1}{m} \times 100$$

where

- m* is the mass, in grams, of the test portion (8.1);
- V*<sub>0</sub> is the volume, in millilitres, of the sulphuric acid solution (5.2) used in the determination;

*V*<sub>1</sub> is the volume, in millilitres, of the potassium hydroxide solution (5.3) used in the titration;

*T*<sub>0</sub> is the exact normality of the sulphuric acid solution (5.2);

*T*<sub>1</sub> is the exact normality of the potassium hydroxide solution (5.3).

The total free alkali may also be expressed in milliequivalents per gram by the formula :

$$\frac{V_0 T_0 - V_1 T_1}{m}$$

Take as the result the arithmetic mean of the duplicate determinations.

**9.2 Reproducibility**

The results obtained on the same sample in two different laboratories, expressed either as sodium hydroxide (NaOH) or as potassium hydroxide (KOH), shall not differ by more than 0,05 %.

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**10 TEST REPORT**

The test report shall include the following particulars :

- a) all information necessary for the complete identification of the sample;
- b) the reference to method used, reference being made to this International Standard;
- c) the results obtained and the method of expression used;
- d) the test conditions;
- e) any operational details not specified in this International Standard, or regarded as optional, as well as all incidents likely to have affected the results.

1) This volume may be increased for certain soaps with high total free alkali contents.