

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 684

ANALYSIS OF SOAPS

DETERMINATION OF TOTAL FREE ALKALI

1st EDITION
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BRIEF HISTORY

The ISO Recommendation R 684, *Analysis of soaps – Determination of total free alkali*, was drawn up by Technical Committee ISO/TC 91, *Surface active agents*, the Secretariat of which is held by the Association Française de Normalisation (AFNOR).

Work on this question by the Technical Committee began in 1963 and led, in 1964, to the adoption of a Draft ISO Recommendation.

In December 1965, this Draft ISO Recommendation (No. 934) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided, in March 1968, to accept it as an ISO RECOMMENDATION.

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ANALYSIS OF SOAPS

DETERMINATION OF TOTAL FREE ALKALI

1. SCOPE

This ISO Recommendation describes the method for the determination of total free alkali in soaps, excluding compounded products.*

1.1 Field of application

The method is not applicable when the soap contains additives which can be decomposed by fatty acids under the operating conditions described (alkali silicates, etc.).

The method is not applicable to coloured soaps, if the colour interferes with the phenolphthalein end point.

2. DEFINITION

By *total free alkali* is understood the sum of the free caustic alkali and the free alkali as carbonate, expressed conventionally as sodium hydroxide (NaOH) for sodium soaps and as potassium hydroxide (KOH) for potassium soaps.

3. PRINCIPLE

Dissolution of the soap in an ethanolic solution of fatty acids, the excess of which is titrated with an ethanolic solution of potassium hydroxide.

4. REAGENTS

- 4.1 *Ethanol*, 80 % (v/v) solution freed from carbon dioxide by boiling for 5 minutes under a reflux condenser.

Cool to ambient temperature and neutralize by means of the ethanolic potassium hydroxide solution (4.2) in the presence of phenolphthalein (4.4) (four drops per 200 ml).

- 4.2 *Potassium hydroxide*, standard ethanolic volumetric solution, approximately 0.1 N.

- 4.3 *Liquid fatty acids* of any origin, freed from mineral acidity, and very light in colour.

- 4.4 *Phenolphthalein* solution, 1 g per 100 ml in ethanol 95 % (v/v).

* See also ISO Recommendation R 685, *Analysis of soaps – Determination of total alkali*.

5. APPARATUS

Usual laboratory apparatus, in particular

- 5.1 *Flask* of about 300 ml capacity, which can be fitted with a reflux condenser,
- 5.2 *Dish for weighing*,
- 5.3 *Analytical balance*.

6. PROCEDURE

6.1 Test portion

Weigh in the dish (5.2), to the nearest 0.001 g, about 5 g of soap.

6.2 Blank test

Carry out a blank test at the same time as the determination and under the same conditions, using exactly the same quantity of fatty acids (4.3) as in the test proper, weighed to the same quantity of fatty acids (4.3) as in the test proper, weighed to the same precision of 0.001 g.

6.3 Determination

Weigh in the dish (5.2), to the nearest 0.001 g, about 0.5 g of fatty acids (4.3).^{*} Transfer them together with the test sample, quantitatively into the flask (5.1) using 100 ml of the ethanol solution (4.1). Fit the flask (5.1) to a reflux condenser. Gently heat until the soap is completely dissolved, then keep boiling gently for 5 minutes. Allow to cool to the ambient temperature.

Titrate with the ethanolic solution of potassium hydroxide (4.2) in the presence of the indicator (4.4).

Carry out two determinations on the same sample.

7. EXPRESSION OF RESULTS

7.1 Method of calculation and formulae

The percentage by mass of total free alkali in the soap is equal to

$$(a) 0.040 \times T \times (V_0 - V_1) \times \frac{100}{M}$$

expressed as sodium hydroxide (NaOH) for sodium soaps;

$$(b) 0.056 \times T \times (V_0 - V_1) \times \frac{100}{M}$$

expressed as potassium hydroxide (KOH) for potassium soaps;

where

M is the weight, in grammes, of the test portion,

V_0 is the volume in millilitres, of potassium hydroxide (4.2) used in the blank test,

V_1 is the volume, in millilitres, of potassium hydroxide (4.2) used in the determination,

T is the exact normality of the ethanolic potassium hydroxide solution (4.2).

^{*} This mass may be increased for certain soaps with a high total free alkali content.