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ISO RECOMMENDATION

R 685

ANALYSIS OF SOAPS

DETERMINATION OF TOTAL ALKALI

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BRIEF HISTORY

The ISO Recommendation R 685, *Analysis of soaps – Determination of total 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. 935) 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 :

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

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.

ISO Recommendation R 685 March 1968

ANALYSIS OF SOAPS

DETERMINATION OF TOTAL ALKALI

1. SCOPE

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

1.1 Field of application

This method allows the simultaneous determination of the total alkali and the total crude fatty acids.** It is not applicable to coloured soaps, if the colour interferes with the methyl orange end point.***

2. DEFINITION

By *total alkali* is understood the sum of the alkali bases combined as soap with fatty and rosin acids, as well as those corresponding to the free caustic or carbonated alkali and to the silicates present (if any) which can be titrated under the test conditions.

The results are expressed as a percentage of sodium hydroxide (NaOH) or of potassium hydroxide (KOH), according to whether sodium or potassium soaps are concerned.

3. PRINCIPLE

Decomposition of the soap by a known amount of acid and titration of the excess of acid.

4. REAGENTS

4.1 *Sulphuric acid* or *hydrochloric acid*, standard volumetric solution, approximately N.

4.2 *Sodium hydroxide*, standard aqueous volumetric solution, approximately N, standardized with methyl orange.

4.3 *Beeswax*, *paraffin wax* or *stearic acid*, very light in colour (or a mixture of these) designated as "wax" in the text, free from impurities and previously dried by heating to about 120 °C.

4.4 *Methyl orange* solution, 0.2 per 100 ml in distilled water.

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* See also ISO Recommendation R 684, *Analysis of soaps – Determination of total free alkali.*

** The procedure specifies dissolving the soap and complete separation of fatty acids in accordance with the *Wax cake method* described in Annex A of ISO Recommendation R 455, *Analysis of soaps – Determination of total crude fatty acids.*

*** A potentiometric method applicable to coloured soaps will be developed later.

5. APPARATUS

Usual laboratory apparatus, in particular

5.1 *Porcelain or glass dish,* about 11 cm in diameter.

5.2 *Analytical balance.*

6. PROCEDURE

6.1 Test portion

Weigh, in the dish (5.1), to the nearest 0.001 g, 5 to 10 g of soap.

6.2 Determination

Dissolve the test portion in 100 ml of hot distilled water.

Add a few drops of methyl orange (4.4), then decompose the soap with an accurately measured, known volume of acid (4.1), sufficient to turn the indicator red. The fatty acids separate and come to the surface in a snow-like condition. Continue to heat on the water-bath, stirring from time to time, until the fatty acids have collected at the surface as a layer of clear liquid.

Add 20 g of wax (4.3), weighed to the nearest 0.01 g. When melting is complete, mix with the other fatty acids by means of a stirrer, so as to obtain a homogeneous layer. Take the dish (5.1) from the water-bath and allow to cool. The supernatant liquid solidifies as a cake.

When the cake is hard enough, pour off the underlying water as completely as possible. Collect this water in a conical flask. Pour into the dish (5.1) about 100 ml of boiling distilled water. The cake will melt. Wash it by stirring with the added water. Allow to cool again so that the cake reforms.*

Collect the washings quantitatively in the conical flask already containing the acid aqueous layer.

Titrate the mixed acid aqueous layer and washings with the sodium hydroxide solution (4.2).

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 alkali in the soap is equal to

$$(a)\ 0.040 \times (V_0 T_0 - V_1 T_1) \times \frac{100}{M}$$

expressed as sodium hydroxide (NaOH) for sodium soaps :

$$(b)\ 0.056 \times (V_0 T_0 - V_1 T_1) \times \frac{100}{M}$$

expressed as potassium hydroxide (KOH) for potassium soaps,

where

M is the mass, in grammes, of the test portion,
V_0 is the volume, in millilitres, of the acid solution (4.1) used,
T_0 is the exact normality of the acid solution (4.1),
V_1 is the volume, in millilitres, of the sodium hydroxide solution (4.2) used,
T_1 is the exact normality of the sodium hydroxide solution (4.2).

* The cake may be used for the determination of total crude fatty acids (see ISO Recommendation R 455).