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R 455

ANALYSIS OF SOAP

DETERMINATION OF TOTAL CRUDE FATTY ACIDS

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BRIEF HISTORY

The ISO Recommendation R 455, *Analysis of Soap. Determination of Total Crude Fatty Acids*, 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 1961 and led, in 1962, to the adoption of a Draft ISO Recommendation.

In June 1963, this Draft ISO Recommendation (No. 583) 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	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	

One Member Body opposed the approval of the Draft: India.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in November 1965, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 455 November 1965

ANALYSIS OF SOAP

DETERMINATION OF TOTAL CRUDE FATTY ACIDS

1. SCOPE

The purpose of this ISO Recommendation is to specify the method of determining total crude fatty acids in commercial soaps, excluding compounded products.

In the Annex, there is also described a method which should be used only for routine controls and for soaps in which the fatty acids are not contaminated with substances insoluble in water, derived from additives (silicates etc.).

2. TERMINOLOGY

Total crude fatty acids means the water-insoluble fatty material which is collected by decomposing the soap with a strong mineral acid under the operating conditions described. This term includes unsaponifiable matter, glycerides and any resinic acids contained in the soap, in addition to the so-called fatty acids.

3. PRINCIPLE

The fatty acids are extracted with diethyl ether and titrated with a solution of sodium hydroxide in ethanol.

4. REAGENTS

4.1 Diethyl ether, pure.

4.2 Ethanol solution, 95 % by volume.

4.3 Acid solution:

either sulphuric acid, $d = 1.83$, diluted 1/5 ; *
or hydrochloric acid, $d = 1.19$, diluted 1/3. *

4.4 Sodium chloride solution, 10 g of sodium chloride dissolved in 100 ml of distilled water.

4.5 Sodium hydroxide ethanolic solution, analytical grade, recently standardized to approximately 0.5 N.

4.6 Methyl-orange solution, 0.2 g in 100 ml of distilled water.

4.7 Phenolphthalein solution, 1 g in 100 ml of the ethanol solution (4.2).

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* Diluted p_1/p_2 means that p_1 parts by volume of the specified solution are diluted to give p_2 parts by volume of the final mixture.

5. APPARATUS

Ordinary laboratory equipment and in particular,

(*a*) Porcelain or glass basins of about 250 ml.

(*b*) Separating funnels, of about 500 ml.

(*c*) Water-bath.

(*d*) Oven regulated at 120°C.

(*e*) Desiccator.

(*f*) Analytical balance.

6. PROCEDURE

6.1 Test portion

Weigh 5 to 10 g of soap into the basin to an accuracy of 0.001 g.

6.2 Determination

Make a hot solution in 100 ml of distilled water. Pour this aqueous solution into a separating funnel, rinsing the basin with small quantities of distilled water.

Add a few drops of the methyl-orange (4.6) and then about 10 ml of the acid solution (4.3). The indicator should turn red. If it does not, add 2 ml more of the acid solution (4.3). Leave to cool to room temperature.

Add 100 ml of the diethyl ether (4.1). Shake the mixture vigorously for 1 min and allow to stand until the two phases are completely separated.

Draw off the acidified water into a second separating funnel. Make a second extract of this acid solution, mixing in the same way, with 50 ml of the diethyl ether (4.1).

Draw off the acidified water. Combine the ethereal solutions in the same separating funnel. Wash twice in succession, using 50 ml of the sodium chloride solution (4.4) each time (mixing for 1 min each time). Verify that the last wash is neutral to methyl-orange (4.6). If it is not, continue washing in the same way until the washing solution is neutral to methyl-orange (4.6).

After drawing off the last washing solution, filter the ethereal solution, if necessary, through a filter paper. Collect in a tared flask. Wash the filter with small portions of the diethyl ether (4.1). Distil off nearly all the diethyl ether by boiling gently.

Dissolve the residue in 20 ml of the ethanol solution (4.2). Neutralize the ethanolic solution of fatty acids with the ethanolic sodium hydroxide solution (4.5),* using 2 to 3 drops of phenolphthalein (4.7) as indicator. Note the volume used.**

Remove the ethanol by placing on a boiling water-bath. Heat the flask in the oven at 120°C until constant mass is reached, i.e., until the difference in weight, after drying in the oven for an additional 15 min, does not exceed 5 mg.*** Weigh the dry soap.

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* Do not use ethanolic potassium hydroxide solution.

** If the fatty acid colour masks the end-point, this may be determined potentiometrically; this method will be standardized later.

*** Before weighing, leave the flask in the desiccator just long enough for it to reach ambient temperature.

7. EXPRESSION OF RESULTS

7.1 Method of calculation and formula

If E is the mass, in grammes, of the test portion,

m is the mass, in grammes, of dry soap,

n is the number of millilitres of ethanolic sodium hydroxide solution (4.5) used,

T is the exact normality of the ethanolic sodium hydroxide solution (4.5),

the percentage of total crude fatty acids in the soap is

$$\left[m-(n\times T\times 0.022)\right]\times\frac{100}{E}$$

Round the result to the nearest 0.1 per cent.

If M_0 is the nominal mass, in grammes, of the piece of soap,

M_1 is the mean mass, in grammes, of the piece of soap at the time of analysis,

the percentage of total crude fatty acids in the initial soap is

$$\left[m-(n\times T\times 0.022)\right]\times\frac{100}{E}\times\frac{M_1}{M_0}$$

7.2 Reproducibility

±0.2 absolute.

7.3 Notes

1. When analysis certificates refer only to " fatty acids ", this always means " total crude fatty acids ".
2. If resin is present, and if a test has been made for it, the result should preferably be expressed as " fatty and resin acids ", but the term " fatty acids " by itself does not imply that the soap is free from resin.
3. If the determination of " pure fatty acids " is required, deduct from the percentage of " total crude fatty acids " the sum of the percentages of unsaponifiable matter, unsaponified matter and any resinic acids.

8. TEST REPORT

In addition to the results, the test report should state all the test conditions and details of procedure not mentioned in this ISO Recommendation, as well as any other factors which may have affected the results.

ANNEX A

WAX CAKE METHOD

A.1 PRINCIPLE

The soap is dissolved and the fatty acids are completely separated by acidification. They are then solidified with beeswax in the form of a cake, which is washed with boiling water, dried and weighed.

A.2 REAGENTS

A.2.1 Beeswax, paraffin wax or stearic acid (or mixture of these) described as "wax" in the text, free from impurities and previously dried by heating at approximately 120°C.

A.2.2 *Acid solution :*

either sulphuric acid, $d = 1.83$, diluted 1/5 ;
or hydrochloric acid, $d = 1.19$, diluted 1/3.

A.2.3 Methyl orange solution: 0.2 g in 100 ml of distilled water.

A.3 APPARATUS

Ordinary laboratory equipment, and in particular,

(*a*) Porcelain or glass basin (C1), approximately 11 cm diameter.

(*b*) Basin (C2), 8 to 9 cm diameter and 4 to 5 cm deep, preferably flat-bottomed and containing a small stirrer thermometer.

(*c*) Water-bath.

(*d*) Sand-bath.

(*e*) Desiccator.

(*f*) Analytical balance.

A.4 PROCEDURE

A.4.1 Test portion

Weigh approximately 10 g of soap with an accuracy of 0.01 g.