INTERNATIONAL STANDARD 3657

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Animal and vegetable oils and fats — Determination of saponification value

Corps gras d'origines animale et végétale — Détermination de l'indice de saponification

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FOREWORD

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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 3657 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in January 1975.

It has been approved by the member bodies of the following countries :

Australia	Germany	New Zealand
Austria	Ghana	Poland
Belgium	Hungary	Romania
Brazil	India	South Africa, Rep. of
Bulgaria	Iran	Spain
Canada	Ireland	Thailand
Chile	Israel	Turkey
Ethiopia	Mexico	United Kingdom
France	Netherlands	Yugoslavia

The member body of the following country expressed disapproval of the document on technical grounds :

Portugal

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Printed in Switzerland

Animal and vegetable oils and fats — Determination of saponification value

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the saponification value of animal and vegetable oils and fats.

The method is not applicable to dark-coloured products or to products which are difficult to saponify or which contain mineral acids.

NOTE — Studies will be undertaken with a view to extending the applicability of the method to the above-mentioned products.

2 REFERENCE

ISO . . ., *Animal and vegetable oils and fats — Sampling.*[1)]

3 DEFINITION

saponification value : The number of milligrams of potassium hydroxide required to saponify 1 g of fat under the conditions specified.

4 PRINCIPLE

Boiling of a sample under reflux with ethanolic potassium hydroxide solution, followed by titration of the excess potassium hydroxide with standard volumetric hydrochloric acid solution.

5 REAGENTS

5.1 Potassium hydroxide, approximately 0,5 N solution in 94 to 97 % (*V*/*V*) ethanol.

This solution should be colourless or straw yellow. A stable, colourless solution can be prepared by either of the following procedures :

1) Reflux 1 litre of ethanol with 8 g of potassium hydroxide and 5 g of aluminium pellets for 1 h, then distil immediately. Dissolve the required amount of potassium hydroxide in the distillate. Allow to stand for several days, then decant the clear supernatant liquid from the potassium carbonate deposited.

2) Add 4 g of aluminium tertbutoxide to 1 litre of ethanol and allow the mixture to stand for several days. Decant the supernatant liquid and dissolve in it the required amount of potassium hydroxide. Allow to stand for several days, then decant the clear supernatant liquid from the potassium carbonate deposited.

Store this solution in a brown or yellow glass bottle fitted with a rubber stopper, and decant it for use.

5.2 Hydrochloric acid, 0,5 N standard volumetric solution, the normality being determined to three decimal places.

5.3 Phenolphthalein solution.

Dissolve 1 g of phenolphthalein in 100 ml of 90 % (*V*/*V*) ethanol.

5.4 Boiling aids.

6 APPARATUS

Usual laboratory equipment and in particular :

6.1 Conical flask, 250 ml capacity, made of alkali-resistant glass, with ground neck.

6.2 Reflux condenser, with ground glass joint fitting the conical flask (6.1).

6.3 Heating device (water bath, electric hot-plate or other suitable apparatus; a naked flame shall not be used).

6.4 Burette, 50 ml capacity, complying with class A of ISO/R 385.

6.5 Pipette, 25 ml capacity, complying with class A of ISO/R 648.

6.6 Analytical balance.

1) In preparation.

7 SAMPLING

See ISO . . .

8 PROCEDURE

8.1 Preparation of test sample

Melt the sample, if necessary, at about 10 °C above its melting point and filter at this temperature through a dry, fast filter paper to which a mixture of 4 g of anhydrous sodium sulphate and 1 g of filter aid has been added. Repeat as necessary until the filtrate is perfectly clear.

8.2 Test portion

Weigh, to the nearest 0,005 g, into the conical flask (6.1), a mass of the thoroughly mixed test sample (8.1) such that the volume of hydrochloric acid (5.2) required for the determination (8.3.2) will be approximately half that required for the blank test (8.4). This mass will usually be about 2 g.

8.3 Determination

8.3.1 Add to the test portion 25,0 ml of the ethanolic potassium hydroxide solution (5.1) and some boiling aids (5.4). Connect the reflux condenser (6.2) to the flask, place the flask on the heating device (6.3) and boil gently for at least 60 min, shaking from time to time.

8.3.2 Add to the hot solution 0,5 to 1 ml of phenolphthalein indicator solution (5.3) and titrate with the standard volumetric hydrochloric acid solution (5.2) until the pink colour of the indicator just disappears.

8.3.3 Carry out two determinations on the same test sample.

8.4 Blank test

Carry out a blank test following the procedure specified in 8.3, using again 25,0 ml of the ethanolic potassium hydroxide solution (5.1).

9 EXPRESSION OF RESULTS

9.1 Method of calculation and formula

The saponification value is equal to :

$$\frac{(V_0 - V_1) \times T \times 56,1}{m}$$

where

V_0 is the volume, in millilitres, of the standard volumetric hydrochloric acid solution (5.2) used for the blank test;

V_1 is the volume, in millilitres, of the standard volumetric hydrochloric acid solution (5.2) used for the determination;

T is the normality of the standard volumetric hydrochloric acid solution (5.2);

m is the mass, in grams, of the test portion.

Take as the result the mean of the two determinations, provided that the requirement of repeatability (see 9.2) is satisfied.

Express the result to one decimal place.

9.2 Repeatability

The difference between the results of two determinations on the same test sample carried out simultaneously or in rapid succession by the same analyst shall not exceed 0,5 % of the mean value.

10 TEST REPORT

The test report shall show the method used and the result obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the results.

The report shall include all details required for complete identification of the sample.