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Animal and vegetable fats and oils — Determination of saponification value

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

Reference number
ISO 3657 : 1988 (E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 3657 was prepared by Technical Committee ISO/TC 34, *Agricultural food products.*

This second edition cancels and replaces the first edition (ISO 3657 : 1977), of which it constitutes a technical revision.

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Animal and vegetable fats and oils — Determination of saponification value

1 Scope

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

If mineral acid is present, the results given by this method are not interpretable unless the mineral acids are determined separately.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 661 : 1980, *Animal and vegetable fats and oils — Preparation of test sample*.

ISO 5555 : 1983, *Animal and vegetable fats and oils — Sampling*.

3 Definition

For the purposes of this International Standard, the following definition applies.

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

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

All reagents shall be of recognized analytical grade and the water used shall be distilled water or water of equivalent purity.

5.1 Potassium hydroxide, c(KOH) $\approx$ 0,5 mol/l solution in 95 % (V/V) ethanol.

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

a) 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.

b) Add 4 g of aluminium tertbutylate 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, standard volumetric solution, c(HCl) = 0,5 mol/l.

5.3 Phenolphthalein, 10 g/l solution in 95 % (V/V) ethanol, or **alkali blue** 6B, 20 g/l solution in 95 % (V/V) ethanol.

5.4 Boiling aids.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

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

6.2 Reflux condenser, with a ground glass joint which fits the conical flask (6.1).

6.3 Heating device (e.g. a water-bath, electric hot-plate or other suitable apparatus). A naked flame is not suitable.

6.4 Burette, of 50 ml capacity, graduated in 0,1 ml divisions.

6.5 Pipette, of 25 ml capacity.

7 Sampling

Sampling shall be carried out in accordance with ISO 5555.

8 Preparation of the test sample

Prepare the test sample in accordance with ISO 661.

9 Procedure

9.1 Test portion

Weigh, to the nearest 5 mg, about 2 g of the test sample (clause 8) into a conical flask (6.1).

NOTE — The test portion of 2 g has been determined on the basis of saponification values of 170 to 200. For other saponification values, the mass should be altered accordingly so that about half the ethanolic potassium hydroxide solution is neutralized.

9.2 Determination

9.2.1 Using a pipette (6.5), 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, shaking from time to time, for 60 min or for 2 h in the case of oils and fats having a high melting point and which are difficult to saponify.

9.2.2 Add to the hot solution 0,5 ml to 1 ml of the phenolphthalein solution (5.3) and titrate with the standard volumetric hydrochloric acid solution (5.2) until the pink colour of the indicator just disappears. If the solution is strongly coloured, use 0,5 ml to 1 ml of alkali blue 6B solution (5.3).

9.3 Blank test

Carry out a blank test following the procedure specified in 9.2, using again 25,0 ml of the ethanolic potassium hydroxide solution (5.1) but omitting the test portion.

9.4 Number of determinations

Carry out two determinations on the same test sample.

10 Expression of results

The saponification value I_s is given by the formula

$$I_s = \frac{(V_0 - V_1) \times c \times 56{,}1}{m}$$

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;

c is the exact concentration, in moles per litre, of the standard volumetric hydrochloric acid solution (5.2);

m is the mass, in grams, of the test portion (9.1).

Take as the result the arithmetic mean of the two determinations, provided that the requirement for repeatability (clause 11) is satisfied.

Express the result to one decimal place.

11 Repeatability

The difference between the values of two determinations, carried out in rapid succession (or simultaneously) by the same analyst using the same apparatus on the same test sample, shall not exceed 0,5 % (relative) of the arithmetic mean value.

NOTE — This figure for repeatability has been accepted historically.

12 Test report

The test report shall specify the method used and the result obtained. It shall also mention all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the result.

The test report shall include all information necessary for the complete identification of the sample.

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Descriptors : animal fats, vegetable fats, animal oils, vegetable oils, chemical analysis, determination, saponification number.

Price based on 2 pages