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Ģivalske in rastlinske maģļ obe in olja - Doloļ evanje ģtevila umiljenja

Animal and vegetable fats and oils -- Determination of saponification value

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

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

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 3657 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

This third edition cancels and replaces the second edition (ISO 3657:1988), which has been technically revised.

Annex A of this International Standard is for information only.

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. The saponification value is a measure of the free and esterified acids present in fats and fatty acids.

The method is applicable to refined and crude vegetable and animal fats.

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

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, this publication do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

3 Term and definition

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

3.1
saponification value
amount of potassium hydroxide in milligrams required to saponify 1 g of fat under the conditions specified in this International Standard

4 Principle

The test sample is saponified by boiling under reflux with an excess of ethanolic potassium hydroxide, followed by titration of the excess potassium hydroxide with standard volumetric hydrochloric acid solution.

5 Reagents

Use only reagents of recognized analytical grade, and distilled or demineralized water of equivalent purity.

5.1 Potassium hydroxide, $c(\mathrm{KOH}) = 0{,}5$ mol/l solution in 95 % ethanol (volume fraction).

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 (approx. 35 g) in the distillate. Allow to stand for several days, then decant the clear supernatant liquid from the precipitated potassium carbonate into a brown-glass stock bottle.

b) Add 4 g of aluminium tert-butylate 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 precipitated potassium carbonate into a brown-glass stock bottle.

5.2 Hydrochloric acid, standard volumetric solution, $c(\mathrm{HCl}) = 0{,}5$ mol/l.

5.3 Phenolphthalein solution, ($\rho = 0{,}1$ g/100 ml) in 95 % ethanol (volume fraction).

5.4 Alkali blue 6B solution, ($\rho = 2{,}5$ g/100 ml) in 95 % ethanol (volume fraction).

5.5 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, or an **automatic burette**.

6.5 Pipette, of 25 ml capacity, or an **automatic pipette**.

6.6 Analytical balance.

7 Sampling

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transportation or storage.

Sampling is not part of this method specified in this International Standard. A recommended sampling method is given in ISO 5555.

8 Preparation of test sample

Prepare the test sample in accordance with ISO 661.

The test samples shall be carefully mixed and filtered if visible impurities are present. If filtration is necessary, this shall be mentioned in the test report.

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).

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. Recommendations for the mass of the test portion are given in Table 1.

Table 1 — Mass of test portion

Expected saponification value	Mass of test portion
150 to 200	2,2 g to 1,8 g
200 to 250	1,7 g to 1,4 g
250 to 300	1,3 g to 1,2 g
$>$ 300	1,1 g to 1,0 g

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 (5.5). 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 hydrochloric acid (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.

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 hydrochloric acid (5.2) used for the blank test;

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