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Rastlinske in živalske maščobe in olja - Določevanje kislinskega števila in kislosti (ISO/DIS 660:2019)

Animal and vegetable fats and oils - Determination of acid value and acidity (ISO/DIS 660:2019)

Tierische und pflanzliche Fette und Öle - Bestimmung der Säurezahl und der Azidität (ISO/DIS 660:2019)

Corps gras d'origines animale et végétale - Détermination de l'indice d'acide et de l'acidité (ISO/DIS 660:2019)

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Animal and vegetable fats and oils — Determination of acid value and acidity

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

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC *34*, Food Products, Subcommittee SC 11, Animal and vegetable fats and oils.

This fourth edition cancels and replaces the third edition (ISO 660:2009), which has been technically revised.

The main changes compared to the previous edition are as follows:

— Addition of a non-applicability statement for milk and milk products to the scope as ISO 1740 "Milkfat products and butter – Determination of fat acidity (Reference method)" applies,

— Additions of details on CMR classification on coloured indicators,

— Additions in Annex of data from the collaborative trial carried out to support this modification.

Animal and vegetable fats and oils — Determination of acid value and acidity

1 Scope

This International Standard specifies three methods (two titrimetric and one potentiometric) for the determination of the acidity in animal and vegetable fats and oils, hereinafter referred to as fats. The acidity is expressed preferably as acid value, or alternatively as acidity calculated conventionally.

This International Standard is applicable to refined and crude vegetable or animal fats and oils, soap stock fatty acids or technical fatty acids. The methods are not applicable to waxes.

Since the methods are completely non-specific, they cannot be used to differentiate between mineral acids, free fatty acids, and other organic acids. The acid value, therefore, also includes any mineral acids that may be present.

Milk and milk products (or fat coming from milk and milk products) are excluded from the scope of ISO 660.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply. ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
acid value
number of milligrams of potassium hydroxide required to neutralize the free fatty acids present in 1 g of fat, when determined in accordance with the procedure specified in this International Standard

Note 1 to entry: The acid value is expressed in milligrams per gram.

3.2
acidity
content of free fatty acids determined according to the procedure specified in this International Standard

Note 1 to entry: The acidity is expressed as a percentage by mass. If the result of the determination is reported as acidity without further explanation, this is, by convention, the acidity based on the oleic acid content.

4 Principle

The sample is dissolved in a suitable solvent mixture, and the acids present are titrated with an ethanolic or methanolic solution of potassium or sodium hydroxide.

The methods specified in 9.1 and 9.2 are reference methods.

5 Reagents

WARNING — Attention is drawn to the regulations which specify the handling of hazardous substances. Technical, organizational and personal safety measures shall be followed.

Use only reagents of recognized analytical grade, unless otherwise specified.

5.1 Solvent A for solvent mixture (5.3): ethanol, volume fraction, $\varphi \approx 96$ %.

As a replacement, propan-2-ol, volume fraction, $\varphi \approx 99$ %, can be used.

5.2 Solvent B for solvent mixture (5.3): diethyl ether, peroxide-free.

As a replacement, *tert*-butyl methyl ether, light petroleum (boiling range 40 °C to 60 °C) or toluene can be used.

WARNING — Diethyl ether is very flammable and may form explosive peroxides. Use with great caution.

5.3 Solvent mixture, mix equal volumes of solvent A and B, (e.g. φ_A = 50 ml/100 ml and φ_B = 50 ml/100 ml).

For hard or animal fats, a solvent mixture of one volume of solvent A (e.g. 25 ml) and three volumes of *tert*-butyl methyl ether or toluene (e.g. 75 ml) is recommended.

Neutralize, just before use, by adding potassium hydroxide solution in the presence of 0,3 ml of the coloured indicator solution per 100 ml of solvent mixture.

For the titration with aqueous KOH, the solvent propan-2-ol can be used.

5.4 Ethanol or methanol, of minimum volume fraction, φ = 95 %.

5.5 Sodium hydroxide or **potassium hydroxide**, ethanolic or methanolic standard volumetric solutions, amount of substance concentration c(NaOH) or c(KOH) = 0,1 mol/l and 0,5 mol/l. The concentration shall be checked with a standard volumetric HCl solution.

NOTE The ethanolic/methanolic sodium/potassium hydroxide solution can be replaced by an aqueous sodium/potassium hydroxide solution, but only if the volume of water introduced does not lead to phase separation.

5.6 Thymolphthalein or Alkali blue 6B, solution in ethanol, mass concentration, ρ = 2 g/100 ml or, failing that, **Phenolphthalein** solution in ethanol, mass concentration, ρ = 1 g/100 ml (CAS number: 1324-80-7).

NOTE 1 Phenolphthalein is classified as CMR substance whereas thymolphthalein and alkali blue are not.

NOTE 2 A laboratory test has been done in order to compare the 3 colour indicators (see Annex B)

For dark-coloured fats, **alkali blue** or **thymolphthalein** shall be used.

Phenolphthalein is a known carcinogenic compound and should only be used when no alternative is available.

5.7 **Water** in accordance with ISO 3696, grade 3.

6 Apparatus

Usual laboratory equipment and, in particular, the following.

6.1 **Burette**, capacity 10 ml, graduated in 0,02 ml, ISO 385[1] class A.

6.2 **Burette**, capacity 25 ml, graduated in 0,05 ml, ISO 385[1] class A.

6.3 **Analytical balance**, capable of being read to the nearest 0,001 g.

6.4 **Automatic titration apparatus** (based on potentiometric electrode) or potentiometer.

6.5 **Combined pH electrode** for non-aqueous acid/base titrations.

6.6 **Graduated volumetric flasks**, volume 1 000 ml, ISO 1042[2] class A.

7 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 5555 [3].

8 Preparation of test sample

Prepare the test sample in accordance with ISO 661, except that if the sample contains volatile fatty acids, the test sample shall not be heated and filtered.

9 Procedure

9.1 Cold solvent method using indicator (Reference method)

9.1.1 Depending on the expected magnitude of the acid value, select the test portion mass and alkali concentration from Table 1.

9.1.2 According to Table 1 weigh the test portion into a 250 ml conical flask.

9.1.3 Add 50 ml to 100 ml of the neutralized solvent mixture (5.3) and dissolve the test portion if necessary with gentle warming.

For high melting point samples, use an ethanol-toluene mixture.

9.1.4 After the addition of an indicator (5.6), titrate with constant swirling using standard potassium hydroxide solution (5.5). The endpoint of the titration is reached when the addition of a single drop of alkali produces a slight but definite colour change persisting for at least 15 s.

Table 1 — Test portion masses and alkali concentrations

Product group (examples)	Acid value approx.	Mass of test portion g	Concentration of KOH mol/l	Accuracy of weighing of the test portion g
Refined vegetable oils Animal fats	0 to 1	20	0,1	0,05
Crude vegetable oils	1 to 4	10	0,1	0,02
Technical grade animal fats	4 to 15	2,5	0,1	0,01
Soap stock fatty acids	15 to 75	0,5 3,0	0,1 0,5	0,001
Technical fatty acids	> 75	0,2 1,0	0,1 0,5	0,001

9.2 Cold solvent method using potentiometric titration (Reference method)

9.2.1 According to Table 1, weigh the test portion into a 150 ml beaker.

9.2.2 Add 50 ml to 100 ml of the neutralized solvent mixture (5.3) and dissolve the sample, if necessary with gentle warming.

For high melting point samples, use an ethanol-toluene mixture.

9.2.3 Introduce the combined electrode in the solvent mixture and connect it with the automatic titration apparatus.

9.2.4 Start the stirrer for at least 30 s and then titrate with constant swirling using standard potassium hydroxide solution (5.5).

9.2.5 As soon as the equivalence point is reached, record the amount of standard solution used.

9.3 Hot ethanol method using indicator

9.3.1 Under the conditions specified in this method, short-chain fatty acids, if present, are volatile.

9.3.2 Weigh into a flask a sufficient mass of the test sample as shown in Table 1, according to the colour and expected acid value.

9.3.3 Heat to boiling 50 ml of the ethanol containing 0,5 ml of the phenolphthalein indicator in a second flask. While the temperature of the ethanol is still above 70 °C, neutralize it carefully with a solution of 0,1 mol/l sodium or potassium hydroxide.

The endpoint of the titration is reached when the addition of a single drop of alkali produces a slight but definite colour change persisting for at least 15 s.

Larger volumes of ethanol and indicator may be necessary for dark-coloured fats. Moreover, for dark-coloured fats, alkali blue or thymolphthalein shall be used.