



SLOVENSKI STANDARD
SIST ISO 660:1995

01-december-1995

Rastlinske in živalske maščobe in olja - Določanje kislinskega števila in kislosti

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

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

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Ta slovenski standard je istoveten z: ISO 660:1983

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ICS:

67.200.10	Rastlinske in živalske maščobe in olja	Animal and vegetable fats and oils
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International Standard



660

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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

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

First edition – 1983-11-01

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UDC 664.3 : 543.852.1

Ref. No. ISO 660-1983 (E)

Descriptors : animal fats, vegetable fats, vegetable oils, tests, determination, acidity, acid number.

Price based on 4 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

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 660 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in September 1982.

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

Australia	Iran	Poland
Austria	Iraq	Portugal
Bulgaria	Israel	South Africa, Rep. of
Canada	Italy	Spain
Egypt, Arab Rep. of	Kenya	Tanzania
Ethiopia	Korea, Dem. P. Rep. of	Thailand
France	Mexico	United Kingdom
Germany, F. R.	Malaysia	USA
Hungary	New Zealand	USSR
India	Philippines	Yugoslavia

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

Netherlands

This International Standard cancels and replaces ISO Recommendation R 660-1968, of which it constitutes a technical revision.

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

1 Scope and field of application

This International Standard specifies two methods (titrimetric and potentiometric) for the determination of free fatty acids in animal and vegetable fats and oils. The acids are expressed preferably as the acid value or alternatively as acidity calculated conventionally.

The method is applicable to animal and vegetable fats and oils. It is not applicable to waxes.

2 References

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

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

3 Definitions

For the purpose of this International Standard, the following definitions apply.

3.1 acid value: Number of milligrams of potassium hydroxide required to neutralize the free fatty acids in 1 g of fat or oil.

3.2 acidity: Conventional expression of the percentage free fatty acids.

According to the nature of the fat or oil, it can be expressed as indicated in table 1.

Table 1

Nature of fat or oil	Expressed as	Molar mass g/mol
Coconut oil, palm kernel oil and similar oils	Lauric acid	200
Palm oil	Palmitic acid	256
Oils from certain cruciferae*	Erucic acid	338
All other fats and oils	Oleic acid	282

* In the case of rapeseed oil having low erucic acid contents, the acidity shall be expressed as oleic acid.

If the result is reported simply as "acidity", without further definition, this is, by convention, the acidity expressed as oleic acid.

If the sample contains mineral acids, these are, by convention, determined as fatty acids.

4 Titrimetric method

4.1 General

This method is most suitable for fats and oils which are not strongly coloured.

4.2 Principle

Dissolution of a test portion in a mixed solvent, followed by titration of the free fatty acids present with ethanolic potassium hydroxide solution.

4.3 Reagents

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

4.3.1 Diethyl ether/95 % (V/V) ethanol, 1 + 1 (V/V) mixture.

WARNING — Diethyl ether is very flammable and may form explosive peroxides. It shall be used with great caution.

Neutralize exactly, just before use, by adding the potassium hydroxide solution (4.3.2) in the presence of 0,3 ml of the phenolphthalein solution (4.3.3) per 100 ml of mixture.

NOTE — If it is not possible to use diethyl ether, a mixed solvent of ethanol and toluene can be used. If necessary, ethanol may be replaced by propan-2-ol.

4.3.2 Potassium hydroxide, ethanolic standard volumetric solution, $c(\text{KOH}) = 0,1 \text{ mol/l}$, or, if necessary, $c(\text{KOH}) = 0,5 \text{ mol/l}$.

The exact concentration of the ethanolic potassium hydroxide solution shall be known, and shall be checked immediately before use. Use a solution prepared at least 5 days before and

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decanted into a brown glass bottle, fitted with a rubber stopper. The solution shall be colourless or straw yellow.

NOTE -- A stable colourless solution of potassium hydroxide can be prepared in the following manner. Boil under reflux 1 000 ml of ethanol with 8 g of potassium hydroxide and 0,5 g of aluminium pellets for 1 h, then distil immediately. Dissolve the required amount of potassium hydroxide in the distillate. Allow the whole to stand for several days and decant the clear supernatant liquid from the deposited potassium carbonate.

The solution can also be prepared without distillation in the following manner. Add 4 ml of aluminium butylate to 1 000 ml 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. This solution is ready for use.

4.3.3 Phenolphthalein, 10 g/l solution in 95 to 96 % (V/V) ethanol, or **alkali blue** (in the case of strongly coloured samples), 20 g/l solution in 95 to 96 % (V/V) ethanol.

4.4 Apparatus

Usual laboratory equipment, and in particular

4.4.1 Analytical balance.

4.4.2 Conical flask, of capacity 250 ml.

4.4.3 Burette, of capacity 10 ml, graduated in 0,1 ml divisions.

4.5 Sampling

Take the laboratory sample in accordance with ISO 5555.

4.6 Procedure

4.6.1 Preparation of the test sample

Prepare the test sample in accordance with ISO 661.

4.6.2 Test portion

Take a test portion, according to the expected acid value, in accordance with table 2.

Table 2

Expected acid value	Mass of test portion	Accuracy of weighing of the test portion
	g	g
< 1	20	0,05
1 to 4	10	0,02
4 to 15	2,5	0,01
15 to 75	0,5	0,001
> 75	0,1	0,000 2

Weigh the test portion into the 250 ml conical flask (4.4.2).

4.6.3 Determination

Dissolve the test portion (4.6.2) in 50 to 150 ml of the previously neutralized diethyl ether/ethanol mixture (4.3.1).

Titrate, whilst swirling the solution, with the 0,1 mol/l potassium hydroxide solution (4.3.2) (see note 3) to the end point as indicated by the indicator (pink colour of phenolphthalein persisting for at least 10 s).

NOTES

1 In the case of very low acid values (< 1), it is preferable to pass a gentle flow of nitrogen through the test solution.

2 The ethanolic standard volumetric potassium hydroxide solution (4.3.2) can be replaced by an aqueous potassium or sodium hydroxide solution when the volume of water introduced does not lead to phase separation.

3 If the quantity of 0,1 mol/l potassium hydroxide solution required exceeds 10 ml, use the 0,5 mol/l solution.

4 If the solution becomes turbid during titration, add a sufficient quantity of the mixed solvent (4.3.1) to give a clear solution.

4.6.4 Number of determinations

Carry out two determinations on the same test sample.

5 Potentiometric method

5.1 Principle

Potentiometric titration of the free fatty acids in a test portion with an isopropanolic potassium hydroxide solution in a non-aqueous medium.

5.2 Reagents

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

5.2.1 Methyl isobutyl ketone, neutralized just before use by adding the isopropanolic potassium hydroxide solution (5.2.2) in the presence of phenolphthalein until the indicator turns pink.

5.2.2 Potassium hydroxide, standard volumetric solution, $c(\text{KOH}) = 0,1 \text{ mol/l}$ or $0,5 \text{ mol/l}$.

5.2.2.1 Potassium hydroxide, standard volumetric solution, $c(\text{KOH}) = 0,1 \text{ mol/l}$, in propan-2-ol.

Dissolve 7 g of potassium hydroxide pellets in propan-2-ol and dilute to 1 000 ml with propan-2-ol.

5.2.2.2 Potassium hydroxide, standard volumetric solution, $c(\text{KOH}) = 0,5 \text{ mol/l}$, in propan-2-ol.

Dissolve 35 g of potassium hydroxide pellets in propan-2-ol and dilute to 1 000 ml with propan-2-ol.

5.2.2.3 Standardization

The exact concentration of the solution shall be determined immediately before use.

Weigh, to the nearest 0,000 2 g, 0,15 g (for solution 5.2.2.1) or 0,75 g (for solution 5.2.2.2) of benzoic acid of purity 99,9 %, transfer to a beaker (5.3.2) and dissolve in 50 ml of the methyl isobutyl ketone (5.2.1).

Introduce the electrodes of the pH meter (5.3.4), start the stirrer (5.3.5), and titrate with the potassium hydroxide solution (5.2.2.1 or 5.2.2.2) to the equivalence point (see note 1 to 5.5.3).

The concentration of the potassium hydroxide solution (5.2.2.1 or 5.2.2.2), expressed in moles per litre, is given by the formula

$$\frac{1\,000 \times m_0}{122,1 \times V_0}$$

where

m_0 is the mass, in grams, of benzoic acid used;

V_0 is the volume, in millilitres, of potassium hydroxide solution (5.2.2.1 or 5.2.2.2) used.

5.3 Apparatus

Usual laboratory equipment, and in particular

5.3.1 Analytical balance.

5.3.2 Beaker, of capacity 150 ml, tall-form.

5.3.3 Burette, of capacity 10 ml, graduated in 0,1 ml divisions.

5.3.4 pH-meter, equipped with glass and calomel electrodes.

Contact between the saturated potassium chloride solution and the test solution shall be made across a sintered glass or porcelain plate at least 3 mm thick.

NOTE — It is advisable to store the glass electrode for 12 h before titration in distilled water or, preferably, in methyl isobutyl ketone. Dry it very gently with a filter paper before making the measurement. Rinse it, immediately after the determination, with methyl isobutyl ketone, then with propan-2-ol, and finally with distilled water.

If the electrode does not function satisfactorily, try to regenerate it by keeping it for 24 h in 1 mol/l isopropanolic hydrochloric acid solution. After this treatment, wash the electrode with distilled water, then with propan-2-ol and methyl isobutyl ketone.

The use of porcelain or thick sintered glass plates to ensure contact between the saturated potassium chloride solution and the test solution prevents diffusion currents and adventitious potentials.

5.3.5 Stirrer, preferably a magnetic stirrer.

5.4 Sampling

Take the laboratory sample in accordance with ISO 5555.

5.5 Procedure

5.5.1 Preparation of the test sample

Prepare the test sample in accordance with ISO 661.

5.5.2 Test portion

Weigh, to the nearest 0,01 g, 5 to 10 g of the test sample into the beaker (5.3.2).

5.5.3 Determination

Dissolve the test portion (5.5.2) in 50 ml of the methyl isobutyl ketone (5.2.1).

Introduce the electrodes of the pH-meter (5.3.4), start the stirrer (5.3.5), and titrate with the potassium hydroxide solution (5.2.2.1 or 5.2.2.2) (according to the expected acidity of the sample) to the equivalence point.

NOTES

1 The equivalence point is generally near to the value 10 on the pH scale and can be determined graphically by observing the inflection point on the neutralization curve.

It can also be calculated by taking the value for which the first differential of the variation of pH as a function of the volume of potassium hydroxide solution added reaches a maximum, or the value for which the second differential becomes zero.

2 It is not possible to determine the inflection point in the case of crude cottonseed oils rich in gossypol. In this case, use a conventional determination of the inflection point, fixed arbitrarily at the pH of the equivalence point of the neutralization of oleic acid by potassium hydroxide in the solvent used for the titration, as follows.

Dissolve approximately 0,282 g of oleic acid in 50 ml of the methyl isobutyl ketone (5.2.1). Plot the neutralization curve of the oleic acid by the potassium hydroxide solution (5.2.2) to be used. Read from the curve the pH of the inflection point (corresponding, in principle, to the addition of 10 ml of 0,1 mol/l potassium hydroxide solution). Using this value, read from the neutralization curve of the cottonseed oil the amount of potassium hydroxide solution used to "neutralize" the cottonseed oil.

5.5.4 Number of determinations

Carry out two determinations on the same test sample.

6 Expression of results

6.1 Acid value

The acid value is equal to

$$\frac{56,1 \times V \times c}{m}$$

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where

V is the volume, in millilitres, of standard volumetric potassium hydroxide solution used;

c is the exact concentration, in moles per litre, of the standard volumetric potassium hydroxide solution used;

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

Take as the result the arithmetic mean of the two determinations.

6.2 Acidity

The acidity can be calculated from the results obtained for the determination of the acid value, either by the titrimetric method (clause 4) or by the potentiometric method (see clause 5).

The acidity, expressed as a percentage by mass, is equal to

$$V \times c \times \frac{M}{1\,000} \times \frac{100}{m}$$

$$= \frac{V \times c \times M}{10 \times m}$$

where

V is the volume, in millilitres, of the standard volumetric potassium hydroxide solution used;

c is the exact concentration, in moles per litre, of the standard volumetric potassium hydroxide solution used;

M is the molar mass, in grams per mole, of the acid chosen for expression of the result (see table 1);

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

Take as the result the arithmetic mean of the two determinations.

7 Test report

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

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

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