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Oilseeds — Determination of acidity of oils

Graines oléagineuses — Détermination de l'acidité de l'huile

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Reference number
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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.

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 729 was prepared by Technical Committee ISO/TC 34, *Agricultural food products.*

This second edition cancels and replaces the first edition (ISO 729 : 1985), of which it constitutes a minor revision.

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Oilseeds — Determination of acidity of oils

1 Scope

This International Standard specifies a method for the determination of the acidity of oils in oilseeds. The acidity is expressed, by preference, as an acid value or alternatively as conventionally calculated acidity.

NOTE 1 — This International Standard has been developed in alignment with ISO 660[1].

The acidity may be determined on the oil from the product as received (pure seeds and impurities), or, if required, on the pure seeds and possibly on the impurities.

The method is not applicable to cotton seeds with adherent cotton linters, or to oil palm or olive fruits.

NOTE 2 — Owing to the particularly poor results obtained during interlaboratory tests on seeds and fruits with high lauric acid content (copra and palm kernel), the application of this method to these oilseeds is at present problematic.

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 542 : 1980, *Oilseeds — Sampling.*

ISO 659 : 1988, *Oilseeds — Determination of hexane extract (or light petroleum extract), called "oil content".*

3 Definitions

For the purposes 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 oil.

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

According to the nature of the fat or oil, acidity may be expressed as shown in table 1.

Table 1

Nature of fat or oil	Expressed as	Molar mass (g/mol)
Copra oil, palm kernel oil and similar oils with high lauric acid content	Lauric acid	200
All other fats and oils	Oleic acid	282

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

4 Principle

Dissolution in a mixture of diethyl ether and ethanol of the oil extracted for the determination of the "oil content" of the seeds, and then titration of the free fatty acids present using an ethanolic potassium hydroxide solution.

5 Reagents

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

5.1 Diethyl ether/ethanol 95 % (V/V), 1 + 1 mixture by volume.

WARNING — Diethyl ether is highly flammable and can form explosive peroxides. Special precautions shall be taken when using it.

Neutralize the mixture exactly, immediately prior to use, using the ethanolic potassium hydroxide solution (5.2) in the presence of 0,3 ml of indicator (5.3) per 100 ml of this mixture.

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

1) ISO 660 : 1983, *Animal and vegetable fats and oils — Determination of acid value and of acidity.*

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5.2 Potassium hydroxide, standard volumetric solution in 95 % (V/V) ethanol, c(KOH) = 0,1 mol/l, or, if necessary, c(KOH) = 0,5 mol/l (see note 2 to 8.3).

The exact concentration shall be known and checked immediately prior to use. Use a solution prepared at least 5 days before and decant it into a glass bottle, tightly sealed with a rubber stopper. The solution shall be colourless or straw yellow.

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

The solution can also be prepared without distillation as follows. Add 4 ml of aluminium butylate to 1 000 ml of ethanol and leave 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.

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

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Apparatus required for the oil extraction method (ISO 659).

6.2 Burette, of 10 ml capacity, graduated in divisions of 0,05 ml.

6.3 Analytical balance.

7 Sampling

Sampling shall be carried out in accordance with ISO 542.

8 Procedure

8.1 Extraction

Carry out the extraction immediately after preparation of the sample, according to the method specified in ISO 659.

8.2 Test portion

Take as the test portion the whole of the extract obtained, weighed to the nearest milligram. Proceed with the determination (8.3) immediately after weighing.

8.3 Determination

Dissolve the test portion (8.2) in 50 ml to 150 ml of the previously neutralized diethyl ether/ethanol mixture (5.1).

Titrate, while stirring, with the 0,1 mol/l ethanolic potassium hydroxide solution (5.2) until the indicator changes colour (pink coloration of the phenolphthalein or red coloration of alkali blue 6B, lasting at least 10 s).

NOTES

1 The ethanolic potassium hydroxide standard volumetric solution (5.2) can be replaced by an aqueous potassium hydroxide or sodium hydroxide solution if the volume of water introduced does not cause separation of the phases.

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

3 If the solution becomes turbid during titration, add a sufficient volume of the diethyl ether/ethanol solution (5.1) to produce a clear solution.

4 For strongly coloured oils, it may be preferable to use the potentiometric method specified in ISO 660.

8.4 Number of determinations

Carry out two determinations on the same test sample.

9 Expression of results

9.1 Method of calculation

9.1.1 Expression as acid value

It is recommended that the result of the analysis be expressed as acid value (see 3.1).

The acid value is equal to

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

where

V is the volume, in millilitres, of the potassium hydroxide standard volumetric solution used (8.3);

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

m is the mass, in grams, of the test portion (8.2);

56,1 in the molar mass, expressed in grams per mole, of potassium hydroxide.

Take as the result the arithmetic mean of the two determinations (see 8.4).

Express the results to the nearest 0,01 unit.

9.1.2 Expression as conventional acidity

The acidity can be calculated from the results obtained for the determination of acid value.

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

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

V, c and m have the same meanings as in 9.1.1.

Take as the result the arithmetic mean of the two determinations (see 8.4).

Express the results to the nearest 0,01 unit.

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9.1.3 In the case of separate analyses of oil from pure seed or from impurities

When the acidity of oil from impurities is determined, the oil shall, if possible, be extracted from 10 g of impurities.

The acid value and the acidity of the total oil of the seed as received can be calculated from the following formulae:

a) by the acid value

$$\frac{a_1 w_{x1} w_{h1} + a_2 w_{x2} w_{h2}}{w_{x1} w_{h1} + w_{x2} w_{h2}}$$

b) for acidity, as a percentage

$$\frac{w_{p1} w_{x1} w_{h1} + w_{p2} w_{x2} w_{h2}}{w_{x1} w_{h1} + w_{x2} w_{h2}}$$

where

a_1 is the acid value of the oil from the pure seed;

a_2 is the acid value of the oil from the impurities;

w_{h1} is the percentage, by mass, of oil from the pure seed;

w_{h2} is the percentage, by mass, of oil from the impurities;

w_{p1} is the acidity, as a percentage by mass, of the oil from the pure seeds;

w_{p2} is the acidity, as a percentage by mass, of the oil from the impurities;

w_{x1} is the percentage, by mass, of pure seed in the material as received;

w_{x2} is the percentage, by mass, of fines and oleaginous impurities in the material as received.

In the case of groundnuts,

w_{h2} is the percentage by mass of oil in the total fines (fines from seed and from foreign material) and other impurities;

w_{x1} is the percentage by mass of pure seed (not including meal);

w_{x2} is the percentage by mass of total fines and other impurities.

9.2 Precision

Two international interlaboratory tests involving, respectively, 14 laboratories, each carrying out two determinations (No. 1), and 18 laboratories, each carrying out three determinations (No. 2), gave the statistical results (determined in accordance with ISO 5725[1]) shown in table 2.

10 Test report

The test report shall specify the method used and the results obtained, indicating clearly the method of expression used and recording whether the result corresponds to the oil from the seed as received or the oil from the pure seed. 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 results.

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

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Table 2

Results as acid values

Sample	Colza		Sunflower		Soya	
	Interlaboratory test		Interlaboratory test		Interlaboratory test	
	No. 1	No. 2	No. 2	No. 1	No. 1	No. 2
Number of laboratories retained after elimination of outliers	13	18	18	12	13	16
Mean	0,98	1,55	1,44	2,26	1,82	1,96
Standard deviation of repeatability, s_r	0,06	0,09	0,09	0,12	0,07	0,20
Coefficient of variation of repeatability	6,0 %	5,8 %	6,4 %	5,5 %	3,7 %	10,3 %
Repeatability, 2,83 s_r	0,17	0,25	0,26	0,35	0,19	0,57
Standard deviation of reproducibility, s_R	0,17	0,40	0,43	0,70	0,43	0,43
Coefficient of variation of reproducibility	18 %	26 %	30 %	31 %	24 %	22 %
Reproducibility, 2,83 s_R	0,48	1,14	1,23	1,98	1,22	1,21

1) ISO 5725 : 1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

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