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Animal and vegetable fats and oils — Determination of iodine value

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

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 3961 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 3961:1996), which has been technically revised.

Animal and vegetable fats and oils — Determination of iodine value

1 Scope

This International Standard specifies a reference method for the determination of the iodine value (IV) of animal and vegetable fats and oils, hereinafter referred to as fats.

Annex A describes a method for the calculation of the IV from fatty acid compositional data. This method is not applicable to fish oils.

NOTE The method in Annex A is based upon the AOCS Recommended Practice Cd 1c-85[4].

2 Normative references

The following referenced documents are indispensable for the application 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.

3.1
iodine value
IV
w_I
mass of halogen, expressed as iodine, absorbed by the test portion following the specified procedure, divided by the mass of the test portion

NOTE The IV is expressed as grams per 100 g of fat.

4 Principle

Dissolution of a test portion in solvent and addition of Wijs reagent. After a specified time, addition of potassium iodide and water, and titration of the liberated iodine with sodium thiosulfate solution.

NOTE Annex A describes a method for the calculation of the IV from fatty acid compositional data. However, this is not intended to be a rapid method. The method gives two results from one analytical procedure. The volumetric method is the reference method.

5 Reagents

Use only reagents of recognized analytical grade, and water in accordance with ISO 3696, grade 3.

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

5.1 Potassium iodide, solution, mass concentration, ρ(KI) = 100 g/l, not containing iodate or free iodine.

5.2 Starch solution: Mix 5 g of soluble starch in 30 ml of water and add to 1 000 ml of boiling water. Boil for 3 min and allow to cool. Prepare fresh starch solution every day.

5.3 Sodium thiosulfate, standard volumetric solution, amount of substance concentration $c(Na_2S_2O_3 \cdot 5H_2O)$ = 0,1 mol/l, standardized not more than 7 days before use.

5.4 Solvent, prepared by mixing one volume of cyclohexane and one volume of glacial acetic acid (50 ml + 50 ml).

5.5 Wijs reagent, containing iodine monochloride in acetic acid. The I/Cl ratio of the Wijs reagent shall be within the limits 1,10 ± 0,1.

Commercially available Wijs reagent shall be used. Any shelf-life limitation of the reagent shall be observed.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Glass weighing scoops, suitable for the test portion and for insertion into the flasks (6.2).

6.2 Conical flasks, capacity 500 ml, fitted with ground glass stoppers and showing no evidence of the presence of moisture.

6.3 Analytical balance, capable of weighing to an accuracy of ± 0,001 g.

6.4 Volumetric flask, capacity 1 000 ml, ISO 1042[2] class A.

6.5 Pipette, capacity 25 ml, automatic or ISO 648[1] class A, fitted with an aspiration bulb.

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 sample in accordance with the method given in ISO 661.

9 Procedure

9.1 Test portion and preparation of blank solution

9.1.1 According to the IV expected for the sample, weigh, to the nearest 0,001 g, in a glass weighing scoop (6.1), the mass of test portion indicated in Table 1.

Table 1 — Mass of test portion

Expected IV w_I g/100 g	Mass of test portion m g	Volume of solvent V ml
$w_I < 1,5$	15,00	25
$1,5 \leqslant w_I < 2,5$	10,00	25
$2,5 \leqslant w_I < 5$	3,00	20
$5 \leqslant w_I < 20$	1,00	20
$20 \leqslant w_I < 50$	0,40	20
$50 \leqslant w_I < 100$	0,20	20
$100 \leqslant w_I < 150$	0,13	20
$150 \leqslant w_I < 200$	0,10	20
NOTE The mass of the sample shall be such that there will be an excess of Wijs reagent of between 50 % and 60 % of the amount added, i.e. 100 % to 150 % of the amount absorbed.		

9.2 Determination

9.2.1 Place the glass scoop containing the test portion in a 500 ml conical flask (6.2) and add the volume of solvent (5.4) indicated in Table 1. Add 25,00 ml of the Wijs reagent (5.5) by pipette (6.5). Insert the stopper, swirl the contents and place the flask in the dark.

NOTE The scoop remains in the flask.

CAUTION — Do not use a mouth pipette for the Wijs reagent.

9.2.2 Prepare a blank with solvent and reagent as in 9.2.1 but omitting the test portion.

9.2.3 For samples having an IV below 150, leave the flasks in the dark for 1 h.

For samples with IVs above 150, and for polymerized products and oils containing conjugated fatty acids (such as tung oil, dehydrated castor oil) and any oils containing keto fatty acids (such as some grades of hydrogenated castor oil) and products oxidized to a considerable extent, leave the flasks in the dark for 2 h.

9.2.4 At the end of the reaction time (9.2.3), add 20 ml of potassium iodide (5.1) and 150 ml of water.

Titrate against standard sodium thiosulfate solution (5.3) until the yellow colour due to iodine has almost disappeared. Add a few drops of the starch solution (5.2) and continue the titration until the blue colour just disappears after very vigorous shaking. Record the volume, V_2, of sodium thiosulfate solution required to reach the endpoint. Note that potentiometric determination of the endpoint is permissible.

9.2.5 Carry out the determination using the blank solution (9.2.2) concurrently. In the blank determination, in 9.2.4, record the volume of sodium thiosulfate solution required to reach the endpoint as V_1.

10 Calculation

The IV, w_I, expressed in grams per 100 g of fat, is given by the equation:

$$w_I = \frac{12{,}69 \times c\,(V_1 - V_2)}{m}$$

where

c is the concentration, in moles per litre, of the sodium thiosulfate solution (5.3);

V_1 is the volume, in millilitres, of sodium thiosulfate solution used for the blank test;

V_2 is the volume, in millilitres, of sodium thiosulfate solution used for the determination;

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

Round off the result as indicated in Table 2.

Table 2 — Rounding off of results

IV w_I g/100 g	Round off to
$w_I < 20$	0,1
$20 \leqslant w_I < 60$	0,5
$w_I \geqslant 60$	1

11 Precision

11.1 General

The values given may not be applicable to concentration ranges and matrices other than those given.

11.2 Repeatability

The absolute difference between two independent test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, should not be greater than the value of r indicated in Table 3.

11.3 Reproducibility

The absolute difference between two independent test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, should not be greater than the value of R indicated in Table 3.

Table 3 — Repeatability and reproducibility limits

IV w_I g/100 g	Repeatability limit r	Reproducibility limit R
$w_I < 20$	0,2	0,7
$20 \leqslant w_I < 50$	1,3	3,0
$50 \leqslant w_I < 100$	2,0	3,0
$100 \leqslant w_I < 135$	3,5	5,0

12 Test report

The test report shall contain at least the following information:

a) all information necessary for the complete identification of the sample;

b) the sampling method used, if known;

c) the test method used, with reference to this International Standard;

d) all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

e) the test result(s) obtained, or, if the repeatability has been checked, the final quoted result obtained.

Annex A
(informative)

Calculated iodine value

A.1 General

This annex describes a method for calculating the IV of edible oils directly from fatty acid compositions determined by gas chromatography of methyl esters of fatty acids. It is applicable to triglycerides and free fatty acids and their hydrogenated products. For oils with an unsaponifiable content greater than 0,5 % (e.g. fish oils), the calculation tends to produce underestimates, and hence is not applicable.

IMPORTANT — While this procedure provides an IV, it is not intended to be a rapid method. The method gives two results from one analysis.

A.2 Procedure

A.2.1 Determine the fatty acid composition of the oil or fatty acid mixture.

A.2.2 Calculate the IVs for groups of components as described in A.2.2.1 and A.2.2.2.

NOTE The calculation tends to produce underestimates for materials with low IVs.

A.2.2.1 Triglycerides

The IV for triglycerides, $w_{I,1}$, is given by

$$w_{I,1} = (w_{16:1} \times 0{,}950) + (w_{18:1} \times 0{,}860) + (w_{18:2} \times 1{,}732) + (w_{18:3} \times 2{,}616) + (w_{20:1} \times 0{,}785) + (w_{22:1} \times 0{,}723)$$

A.2.2.2 Free fatty acids

The IV for free fatty acids, $w_{I,2}$, is given by

$$w_{I,2} = (w_{16:1} \times 0{,}997\ 6) + (w_{18:1} \times 0{,}898\ 6) + (w_{18:2} \times 1{,}810) + (w_{18:3} \times 2{,}735) + (w_{20:1} \times 0{,}817\ 5) + (w_{22:1} \times 0{,}749\ 7)$$

where

$w_{16:1}$ is the percentage mass fraction of hexadecenoic acid;

$w_{18:1}$ is the percentage mass fraction of octadecenoic acid;

$w_{18:2}$ is the percentage mass fraction of octadecadienoic acid;

$w_{18:3}$ is the percentage mass fraction of octadecatrienoic acid;

$w_{20:1}$ is the percentage mass fraction of eicosenoic acid;

$w_{22:1}$ is the percentage mass fraction of docosenoic acid.

The subscripts, in the format n_C:n_{ene} denote the number of carbon atoms in the molecule, n_C, followed by the number of double bonds, n_{ene}.

Calculated iodine values based on gas chromatographic (GC) fatty acid determination of non-triglyceride lipid materials, such as partial esters of glycerol, partial esters of sorbitol/sorbitan/isosorbide, partial esters of polyoxyethylene sorbitol/sorbitan/isosorbide or glycerol, provide the calculated iodine value of only the fatty acids used to prepare the partial esters. To obtain the actual iodine value of partial esters with non-fatty acid polyol diluents, the chlorinated Wijs reagent IV method should be used. IV values of partial esters via the Wijs method are lower than those obtained by GC because of the dilution effect of the polyol.