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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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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 document 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 of 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 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*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 307, *Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This seventh edition cancels and replaces the sixth edition (ISO 3961:2018), of which it constitutes a minor revision.

The changes are as follows:

— entry errors have been corrected in Table 1.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Animal and vegetable fats and oils — Determination of iodine value

1 Scope

This document specifies a reference method for the determination of the iodine value (commonly known in the industry as IV) of animal and vegetable fats and oils, hereinafter referred to as fats.

Annex B describes a method for the calculation of the IV from fatty acid compositional data. This method is not applicable to fish oils. Furthermore, cold-pressed, crude and unrefined vegetable oils as well as (partially) hydrogenated oils can give different results by the two methods. The calculated IV is affected by impurities and thermal degradation products.

NOTE The method in Annex B is based upon the AOCS Official method Cd 1c-85[10].

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*

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 https://www.electropedia.org/

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

Note 1 to entry: The IV is expressed as a mass fraction in 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 B 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.

WARNING — Attention is drawn to the regulations which specify the handling of hazardous substances. Technical, organizational and personal safety measures shall be followed. Wijs reagent causes severe burns; vapours can cause lung and eye damage. A fume hood shall be used for the work.

5.1 Water, in accordance with ISO 3696[4], grade 3.

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

5.3 Starch solution. Mix 5 g of soluble starch in 30 ml of water (5.1) and add to 1 000 ml of boiling water. Boil for 3 min and allow to cool. Prepare fresh starch solution every day.

5.4 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.

5.5 Solvent, prepared by mixing one volume of cyclohexane (50 ml) and one volume of glacial acetic acid (50 ml), volume fractions φ = 50 ml/100 ml.

5.6 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. Wijs reagent is sensitive to temperature, moisture, and light. Store in the dark at < 30 °C.

Use commercially available Wijs reagent. Observe any shelf-life limitation of the reagent.

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, readability 0,000 1 g and weighing accuracy 0,001 g.

6.4 Volumetric flask, capacity 1 000 ml, ISO 1042[3], class A.

6.5 Pipette, capacity 25 ml, automatic, ISO 8655-2[7], or ISO 648[2], class A, fitted with an aspiration bulb.

6.6 Burette, capacity 25 ml and 50 ml, graduated in 0,1 ml divisions, ISO 385[1], class A, autotitrator, ISO 8655-3[8], as an alternative.

7 Sampling

Sampling is not part of the method specified in this document. A recommended sampling method is given in ISO 5555[5].

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

8 Preparation of the test sample and test portion

Prepare the sample in accordance with the method given in ISO 661.

According to the IV expected for the sample, weigh (6.3), to the nearest 0,001 g or 0,000 5 g, in a glass weighing scoop (6.1), the mass of test portion indicated in Table 1.

If the expected IV is not known, pre-test different test portions. The mass of the sample shall be such that there is an excess of Wijs reagent of between 50 % and 60 % of the amount added, i.e. 100 % to 150 % of the amount absorbed.

Table 1 — Initial (theoretical) test portion mass for the expected iodine value

Expected iodine value	Initial mass for 150 % excess g	Initial mass for 100 % excess g	Initial mass accuracy g	Solvent mixture ml
< 3	10	10	0,001	25
3	8,461	10,576	0,001	25
5	5,077	6,346	0,001	25
10	2,538	3,173	0,001	20
20	1,269	1,586	0,001	20
40	0,634	0,793	0,001	20
60	0,423	0,529	0,001	20
80	0,317	0,397	0,001	20
100	0,254	0,317	0,000 5	20
120	0,212	0,264	0,000 5	20
140	0,181	0,227	0,000 5	20
160	0,159	0,198	0,000 5	20
180	0,141	0,176	0,000 5	20
200	0,127	0,159	0,000 5	20

9 Procedure

9.1 Place the glass scoop containing the test portion in a 500 ml conical flask (6.2) and add the volume of solvent (5.5) indicated in Table 1. Add 25,00 ml of the Wijs reagent (5.6) by pipette (6.5). Insert the stopper, swirl the contents and place the flask in the dark.

Melt and dissolve fats and oils with a IV of 20 or less (hard or hardened fats) in warm solvent (60 °C). It is also recommended that all flasks and reagents be warmed before use. Closed vessels shall be used to avoid evaporation and change in concentration when warming the reagents.

NOTE The scoop remains in the flask.

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

9.2 Prepare a blank with solvent and reagent as in 9.1 but omitting the test portion.

9.3 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.

9.4 At the end of the reaction time (9.3), add 20 ml of potassium iodide (5.2) and 150 ml of water (5.1). Titrate against standard sodium thiosulfate solution (5.4) until the yellow colour due to iodine has almost

disappeared. Add a few drops of the starch solution (5.3) and continue the titration until the blue colour just disappears after vigorous shaking. Record the volume, V_2, of sodium thiosulfate solution required to reach the end point. Note that potentiometric determination of the end point is permissible.

9.5 Carry out the determination using the blank solution (9.2) concurrently. In the blank determination, in 9.4, record the volume of sodium thiosulfate solution required to reach the end point as V_1.

10 Calculation

Calculate the iodine value (commonly known in the industry as IV), in grams per 100 g of fat, using the Formula (1).

$$w_I = \frac{12{,}69 \times c(V_1 - V_2)}{m} \qquad (1)$$

where

c is the concentration, in moles per litre, of the sodium thiosulfate solution (5.4);

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.

The results are rounded as indicated in Table 2.

Table 2 — Rounding off of results

IV g/100 g	Round to
≤ 60	0,1
> 60	1

11 Precision

11.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method are summarized in Annex A. It is possible that the values derived from this interlaboratory test are not applicable to concentration ranges and matrices other than those given.

11.2 Repeatability limit, *r*

The repeatability limit, *r*, is the value less than or equal to which the absolute difference between two test results obtained under repeatability conditions may be expected to be with a probability of 95 %.

Repeatability conditions are conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time.

11.3 Reproducibility limit, *R*

The reproducibility limit, *R*, is the value less than or equal to which the absolute difference between two test results obtained under reproducibility conditions may be expected to be with a probability of 95 %.

Reproducibility conditions are conditions where independent test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment within short intervals of time.

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 International Standard used, i.e. ISO 3961:2024;

d) the method used (if the standard includes several);

e) all operating details not specified in this document, or regarded as optional, together with details of any incidents which can have influenced the test result(s);

f) the test result(s) obtained;

g) if the repeatability has been checked, the final quoted result obtained;

h) any unusual features observed;

i) the date of the test.

Annex A
(informative)

Interlaboratory test

The precision of the method has been established by an international interlaboratory test carried out in accordance with ISO 5725 (all parts)[6]. The test was organized by DIN in 2011.

The statistical results are given in Tables A.1 to A.3.

Table A.1 — Statistical results for the Wijs method

Parameter	Sample							
	A	B	C	D	E	F	G	H
	Hardened vegetable oil	Coconut oil	Butter fat	Palm fat	Olive oil	Rapeseed oil	Sun-flower seed oil	Fish oil
Number of participating laboratories	15	18	19	19	19	19	19	19
Number of laboratories retained after eliminating outliers	12	15	17	16	17	17	16	18
Number of individual test in all laboratories	24	30	34	32	34	34	32	36
Mean, $\bar{w}_I$, g/100 g	**0,78**	**8,33**	**32,99**	**51,18**	**81,5**	**113,1**	**124,9**	**199,1**
Repeatability standard deviation, s_r, g/100 g	0,07	0,07	0,17	0,21	0,6	0,8	0,6	1,1
Coefficient of variation of repeatability, %	9,1	0,9	0,5	0,4	0,7	0,7	0,5	0,6
Repeatability limit, r (2,8 s_r), g/100 g	**0,20**	**0,20**	**0,48**	**0,59**	**1,7**	**2,2**	**1,7**	**3,1**
Reproducibility standard deviation, s_R, g/100 g	0,11	0,13	0,55	0,50	1,2	1,4	1,4	5,5
Coefficient of variation of reproducibility, %	14,6	1,6	1,7	1,0	1,5	1,2	1,1	2,7
Reproducibility limit, R (2,8 s_R), g/100 g	**0,32**	**0,36**	**1,54**	**1,40**	**3,4**	**3,9**	**3,9**	**15,3**

Table A.2 — Statistical results for the calculation from fatty acid composition (see Annex B)

Parameter	Sample						
	A	B	C	D	E	F	G
	Hardened vegetable oil	Coconut oil	Butter fat	Palm fat	Olive oil	Rapeseed oil	Sun-flower seed oil
Number of participating laboratories	18	18	18	18	18	18	18
Number of laboratories retained after eliminating outliers	17	15	16	16	18	14	15
Number of individual test in all laboratories	34	30	32	32	36	28	30
Mean, $\bar{w}_I$, g/100 g	**0,22**	**8,61**	**30,16**	**51,49**	**80,3**	**111,3**	**124,5**
Repeatability standard deviation, s_r, g/100 g	0,04	0,09	0,17	0,32	0,3	0,2	0,3
Coefficient of variation of repeatability, %	16,6	1,0	0,6	0,6	0,4	0,2	0,2
Repeatability limit, r (2,8 s_r), g/100 g	**0,10**	**0,25**	**0,48**	**0,91**	**0,8**	**0,5**	**0,8**
Reproducibility standard deviation, s_R, g/100 g	0,23	0,87	1,85	1,00	1,6	0,6	0,7
Coefficient of variation of reproducibility, %	104,5	10,1	6,1	1,9	2,0	0,5	0,6
Reproducibility limit, R (2,8 s_R), g/100 g	**0,64**	**2,44**	**5,18**	**2,80**	**4,5**	**1,6**	**2,0**

Table A.3 — Comparison of $\bar{w}_I$, r, R for both determination methods

Parameter		Sample						
		A	B	C	D	E	F	G
		Hardened vegetable oil	Coconut oil	Butter fat	Palm fat	Olive oil	Rapeseed oil	Sun-flower seed oil
Mean, $\bar{w}_I$, g/100 g	Wijs titration	**0,78**	**8,33**	**32,99**	**51,18**	**81,5**	**113,1**	**124,9**
	Calculation	**0,22**	**8,61**	**30,16**	**51,49**	**80,3**	**111,3**	**124,5**
	Difference	**0,56**	**0,28**	**2,83**	**0,31**	**1,20**	**1,78**	**0,40**
Repeatability limit, r, g/100 g	Wijs titration	**0,20**	**0,20**	**0,48**	**0,59**	**1,7**	**2,2**	**1,7**
	Calculation	**0,10**	**0,25**	**0,48**	**0,91**	**0,8**	**0,5**	**0,8**
Reproducibility limit, R, g/100 g	Wijs titration	**0,32**	**0,36**	**1,54**	**1,40**	**3,4**	**3,9**	**3,9**
	Calculation	**0,64**	**2,44**	**5,18**	**2,80**	**4,5**	**1,6**	**2,0**

Annex B
(informative)

Calculated iodine value for non-fish oils

B.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, free fatty acids, fatty acid methyl esters and their hydrogenated products. For oils with an unsaponifiable content greater than 0,5 % mass fraction (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.

B.2 Procedure

B.2.1 Determine the fatty acid composition of the oil or fatty acid mixture (ISO 12966-4)[9].

All positional isomers as well as all *cis*-/*trans*-isomers shall be included in the calculation.

B.2.2 Calculate the IVs for groups of components as described in Clause B.3.

B.3 Calculation

The iodine value for triglycerides (TAG) is calculated using Formula (B.1):

$$\mathrm{IV} = \sum \omega_i \times C_i \qquad \text{(B.1)}$$

where

IV is the calculated iodine value of the sample, given in g iodine per 100 g sample;

ω_i is the percentage of the fatty acid component *i*;

C_i the contribution factor for the triglyceride component *i*, listed in Table B.1;

The contribution factors C_i listed in Table B.1 for the individual triglyceride components *i* are calculated using Formula (B.2):

$$C_i = \frac{253{,}81 \times n_i}{M_i} \qquad \text{(B.2)}$$

where

n_i is the number of olefinic double bonds in the triglyceride component *i*;

M_i is the molar mass of the triglyceride component *i*;

253,81 is the molar mass of the iodine molecule (I_2).