INTERNATIONAL STANDARD

ISO 3961

Third edition
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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

Reference number
ISO 3961:1996(E)

Foreword

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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.

International Standard ISO 3961 was prepared by Technical Committee ISO/TC 36, *Agricultural food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

This third edition cancels and replaces the second edition (ISO 3961:1989), which has been technically revised.

Annexes A and B of this International Standard are for information only.

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Animal and vegetable fats and oils — Determination of iodine value

1 Scope

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

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 indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 661:1989, *Animal and vegetable fats and oils — Preparation of test sample.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

3 Definition

For the purposes of this International Standard, the following definition applies.

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

Iodine value 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.

5 Reagents

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

5.1 Potassium iodide (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.

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

5.4 Solvent, prepared by mixing equal volumes of cyclohexane and glacial acetic acid.

5.5 Wijs reagent, containing iodine monochloride in acetic acid.

The I/Cl ratio of the Wijs reagent shall be within the limits $1{,}10 \pm 0{,}1$.

NOTE 1 Commercially available Wijs reagent can be used.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

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

6.2 Conical flasks, of 500 ml capacity, fitted with ground glass stoppers and being completely dry.

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

7 Sampling

It is important that the laboratory receive a sample which is truly representative and has not 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.

8 Preparation of test sample

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

9 Procedure

NOTE 2 If it is required to check whether the repeatability requirement (see 11.1) is met, carry out two single determinations under repeatability conditions.

9.1 Test portion and preparation of blank solution

9.1.1 According to the iodine value 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 iodine value g/100 g	Mass of test portion g	Volume of solvent ml
Less than 1,5	15,00	25
1,5 to 2,5	10,00	25
2,5 to 5	3,00	20
5 to 20	1,00	20
20 to 50	0,40	20
50 to 100	0,20	20
100 to 150	0,13	20
150 to 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 flask (6.2) and add the volume of solvent (5.4) indicated in table 1. Add 25 ml of the Wijs reagent (5.5) by pipette. Insert the stopper, swirl the contents and place the flask in the dark.

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 iodine value below 150 leave the flasks in the dark for 1 h.

For samples with iodine values 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 with 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. Note that potentiometric determination of the endpoint is permissible.

9.2.5 Carry out the determination using the blank solution (9.2.2) concurrently.

10 Calculation

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

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

where

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

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

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

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

Round off the result as indicated in table 2.

Table 2 — Rounding off of results

Values in grams per 100 g

w_I	Round off to
Less than 20	0,1
20 to 60	0,5
60 and over	1

11 Precision

Details of an interlaboratory test on the precision of the method are summarized in annex A. The values derived from this interlaboratory test may not be applicable to concentration ranges and matrices other than those given.

11.1 Repeatability

The absolute difference between two independent single 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.

11.2 Reproducibility

The absolute difference between two single 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

w_I g/100 g	r	R
Less than 20	0,2	0,7
20 to 50	1,3	3,0
50 to 100	2,0	3,0
100 to 135	3,5	5,0

12 Test report

The test report shall specify

— the method in accordance with which sampling was carried out, if known;

— the method used;

— the test result(s) obtained; and

— if the repeatability has been checked, the final quoted result obtained.

It shall also mention all operating details not specified in this International Standard, or regarded as optional (e.g. reaction time, see 9.2.3), together with details of any incidents which may have influenced the test result(s).

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

Annex A
(informative)

Interlaboratory test

International collaborative studies carried out in 1988 to 1990 under the auspices of the IUPAC Commission for Oils, Fats and Waxes gave the statistical results shown in table A.1 when evaluated in accordance with ISO 5725.

Table A.2 displays the estimated mean determination for each laboratory, for each of two analytical methods (that given in the previous edition of this International Standard and the present method), and corrected for design imbalance in the samples analysed. These means were derived from the entire database, that is by analysing the average of the A and B determinations (see table A.3). Table A.2 also displays the derived mean difference between the results derived from the two methods together with the pooled overall difference and its standard error. The laboratory mean values were closely similar and the good agreement between laboratories for both methods is further demonstrated by the between-laboratory standard deviations given in table 3. These are only a little greater than the within-laboratory variation with a coefficient of variation of 1,2 % for the old method and 1,3 % for the new cyclohexane/acetic acid method. There is no consistent bias of one method over the other from one laboratory to another and the mean difference (table A.2) is not significantly different from zero.

The results of the fish oil collaborative trial confirm those of the collaborative study of the same two methods applied to a range of vegetable and animal oils, including one sample of fish oil with a low iodine value (table A.1) in showing that cyclohexane/acetic acid can be used in place of carbon tetrachloride without loss of precision.

Table A.1 — Results of interlaboratory tests

Sample	No. of laboratories	Mean iodine value g/100 g	r	R
Palm kernel oil	8	18,3	0,14	0,64
Beef fat	10	46,9	1,33	3,1
Crude palm oil	9	52,6	1,6	2,3
Refined palm oil	10	53,2	0,82	1,9
Hardened fish oil	17	72,8	1,6	2,3
Hardened soyabean oil	17	74,8	1,5	2,1
Hardened soyabean oil	17	102,3	2,2	5,1
Sunflower seed oil	10	132,9	3,6	4,8

Table A.2 — Mean determinations of iodine value

Values in grams per 100 g

Laboratory	Method given in		Mean difference
	ISO 3961:1989 using carbon tetrachloride	ISO 3961:1996 using cyclohexane/acetic acid (1/1)	
1	155,43	154,22	1,21
3	156,02	155,74	0,28
5	154,59	154,87	− 0,28
6	154,64	155,61	− 0,97
7	154,00	154,29	− 0,29
8	154,70	153,56	1,15
10	154,88	156,86	− 1,98
Overall:			−0,13 to +0,16