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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
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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.

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 documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2, www.iso.org/directives.

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

The committee responsible for this document is ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

This fifth edition cancels and replaces the fourth edition (ISO 3961:2009), 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 (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.[9]

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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.

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 solution 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 International Standard. 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	Initial mass for 100 % excess	Initial mass accuracy	Solvent mixture
	g	g	g	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	0,846	1,586	0,001	20
40	0,634	0,793	0,001	20
60	0,432	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 endpoint. Note that potentiometric determination of the endpoint 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 endpoint 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 following formula.

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

where

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

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 test method used, with reference to this International Standard (ISO 3961:2013);

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;

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