International Standard 3961

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Animal and vegetable oils and fats — 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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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.

International Standard ISO 3961 was developed by Technical Committee ISO/TC 34, *Agricultural food products,* and was circulated to the member bodies in October 1975.

It has been approved by the member bodies of the following countries :

Australia	Ghana	Poland
Austria	Hungary	Portugal
Belgium	India	Romania
Canada	Iran	South Africa, Rep. of
Chile	Israel	Thailand
Czechoslovakia	Korea, Rep. of	Turkey
France	Mexico	United Kingdom
Germany, F.R.	New Zealand	Yugoslavia

No member body expressed disapproval of the document.

NOTE — The method described in this International Standard is technically equivalent to IUPAC method II.D.7.3.

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Animal and vegetable oils and fats — Determination of iodine value

1 SCOPE

This International Standard specifies a method for the determination of the iodine value based on the Wijs procedure .

2 FIELD OF APPLICATION

The method is applicable to all animal and vegetable oils and fats intended for human and animal consumption.

3 REFERENCES

ISO/R 385, *Burettes.*

ISO 648, *Laboratory glassware — One-mark pipettes.*

4 DEFINITION

iodine value : The amount of iodine monochloride, expressed in grams of iodine, absorbed by 100 g of the product under the operating conditions described.

5 PRINCIPLE

Addition of a solution of iodine monochloride in a mixture of acetic acid and carbon tetrachloride to a test portion. Reduction of the excess of iodine monochloride after a specified reaction time by adding potassium iodide solution and water, and titration of the liberated iodine with standard volumetric sodium thiosulphate solution.

6 REAGENTS

All reagents shall be of analytical reagent quality. The water used shall be distilled water or water of at least equivalent purity.

6.1 Potassium iodide, 100 g/l solution containing no free iodine or iodate.

6.2 Starch solution.

Mix 5 g of soluble starch in 30 ml of water, add this mixture to 1 000 ml of boiling water and leave boiling for 3 min.

6.3 Sodium thiosulphate, 0,1 N standard volumetric solution.

6.4 Glacial acetic acid, free from ethanol and oxidizable matter.

6.5 Carbon tetrachloride, free from oxidizable matter.

NOTE — Check the absence of oxidizable matter in each of reagents 6.4 and 6.5 by shaking 10 ml of the reagent with 1 ml of saturated potassium dichromate solution and 2 ml of concentrated sulphuric acid, ρ_{20} 1,84 g/ml. No green coloration should appear.

6.6 Iodine monochloride, solution in acetic acid/carbon tetrachloride mixture (Wijs reagent).

This reagent is available commercially. It may also be prepared as follows :

Weigh 9 g of iodine trichloride (ICl_3) into a brown glass bottle of 1 500 ml capacity; dissolve in a mixture of 700 ml of glacial acetic acid (6.4) and 300 ml of carbon tetrachloride (6.5).

Take 5 ml of the solution and add 5 ml of the potassium iodide solution (6.1) and 30 ml of water. Titrate the liberated iodine with the 0,1 N sodium thiosulphate solution (6.3) in the presence of few drops of starch solution (6.2) as indicator.

Add 10 g of pure re-sublimed iodine to the bulk of the reagent and dissolve completely by shaking. Titrate the free iodine as above. The titre should now equal one and a half times that of the first determination. If this is not the case, add a small quantity of pure re-sublimed iodine until the titre slightly exceeds the limit of one and a half times. It is important that no trace whatever of iodine trichloride should remain, as it would cause secondary reactions.

Let the solution stand, then decant the clear liquid into a yellow or brown bottle. If stored in a well-stoppered bottle away from the light, the solution can be used for several months.

NOTE — If iodine trichloride is not available, the Wijs reagent may be prepared from iodine monochloride (ICl), as follows :

Dissolve 19 g of iodine monochloride in a mixture of 700 ml of glacial acetic acid (6.4) and 300 ml of carbon tetrachloride (6.5).

After addition of a few milligrams of pure re-sublimed iodine, titrate the free iodine as described above. Dilute, if necessary, with the specified mixture of solvents until 5 ml of the solution is equivalent to about 10 ml of the sodium thiosulphate solution (6.3).

The correct composition of the reagent should be verified according to the following procedure :

a) Into a wide-necked flask of about 250 ml capacity with ground glass stopper, put 50 ml of 50 % (*V/V*) hydrochloric acid solution and 50 ml of carbon tetrachloride. Add exactly 25 ml of Wijs reagent prepared with iodine monochloride and shake.

Titrate the free iodine present in the reddish-violet layer of carbon tetrachloride with 0,04 N potassium iodate solution, shaking vigorously until the layer becomes colourless.

If the carbon tetrachloride layer is already colourless, this indicates that the Wijs reagent does not contain free iodine and, consequently, the ratio of iodine to chlorine is less than 1; in this case it is necessary to add to 25 ml of the prepared Wijs reagent a quantity of pure re-sublined iodine so that the carbon tetrachloride layer develops a reddish-violet coloration. Next, calculate the quantity of iodine necessary for the total amount of Wijs reagent and dissolve this in the solution in question. Proceed once again as described above and titrate the free iodine in the reddish-violet layer of carbon tetrachloride with 0,04 N potassium iodate solution.

b) Into a second wide-necked flask of about 250 ml capacity with ground glass stopper, introduce exactly 25 ml of Wijs reagent containing a sufficient amount of free iodine. Add 15 ml of 150 g/l potassium iodide solution and about 150 ml of water.

Shake and titrate the liberated iodine with the sodium thiosulphate solution (6.3) in the presence of a few drops of starch solution (6.2) as indicator; at the end of the titration shake vigorously.

Calculation

$$\frac{\text{iodine}}{\text{chlorine}} = \frac{V_1T_1 + V_2T_2}{V_1T_1 - V_2T_2}$$

V_1 is the volume, in millilitres, of the sodium thiosulphate solution (6.3) used to determine the iodine from the iodine monochloride;

V_2 is the volume, in millilitres, of the potassium iodate solution used to determine free iodine;

T_1 is the exact normality of the sodium thiosulphate solution (6.3) used;

T_2 is the exact normality of the potassium iodate solution used.

It should be noted that, as the point at issue is to determine only whether the Wijs reagent really contains a slight excess of iodine, one can stop at the titration described under point a) of the procedure. At the moment that the carbon tetrachloride layer becomes coloured – either directly after adding 25 ml of the Wijs reagent to the mixture of hydrochloric acid solution and carbon tetrachloride, or after having dissolved a sufficient amount of pure re-sublimed iodine in the Wijs reagent – it is evident that the ratio to be determined is more than 1.

7 APPARATUS

Usual laboratory equipment and the following items :

7.1 Glass weighing scoops, of suitable capacity for the test portion.

7.2 Wide-necked glass bottles with ground glass stoppers (for example, iodine flasks), capacity about 250 ml.

7.3 Burettes, 50 ml, graduated in 0,1 ml, in accordance with ISO/R 385.

7.4 Pipettes, 20 and 25 ml, complying with ISO/R 648.

7.5 Analytical balance.

NOTE – The apparatus shall be scrupulously clean and perfectly dry.

8 SAMPLING

The sampling of animal and vegetable oils and fats will form the subject of a future International Standard.

9 PROCEDURE

9.1 Test portion

The mass of the test portion varies according to its expected iodine value in the following way :

Expected iodine value	Mass of test portion g
less than 5	3,00
5 to 20	1,00
21 to 50	0,40
51 to 100	0,20
101 to 150	0,13
151 to 200	0,10

Melt the sample, if necessary, at about 10 °C above its melting point and filter at this temperature through a dry, fast filter paper to which a mixture of 4 g of anhydrous sodium sulphate and 1 g of filter aid has been added. The filtrate must be perfectly clear.

9.2 Determination

The determination shall be carried out at room temperature.

Weigh the test portion to the nearest 0,000 1 g in a glass weighing scoop (7.1). Place it in a 250 ml bottle (7.2). Add 15 ml of the carbon tetrachloride (6.5) to dissolve the fat. Add exactly 25 ml of the Wijs reagent (6.6), insert the stopper, shake gently and place the bottle in the dark.

For products having an iodine value below 150, leave the bottle for 1 h; for those with an iodine value above 150 and for polymerized products or products oxidized to a considerable extent, leave it for 2 h.

At the end of this time, add 20 ml of the potassium iodide solution (6.1) and 150 ml of water.

Titrate with the sodium thiosulphate solution (6.3) until the yellow colour due to iodine has almost disappeared.

Add a few drops of the starch solution (6.2) and continue the titration until the blue colour just disappears after very vigorous shaking.

NOTE — Potentiometric determination of the end point is permissible.

Carry out two determinations on the same test sample.

9.3 Blank test

Carry out a blank test simultaneously under the same conditions.

10 EXPRESSION OF RESULTS

10.1 Method of calculation and formula

The iodine value is equal to

$$\frac{12{,}69\ T_1\ (V_3 - V_4)}{m}$$

where

T_1 is the exact normality of the sodium thiosulphate solution (6.3) used;

V_3 is the volume, in millilitres, of the sodium thiosulphate solution (6.3) used for the blank test;

V_4 is the volume, in millilitres, of the sodium thiosulphate solution (6.3) used for the determination;

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

10.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst shall not exceed 0,4 unit of iodine value.

11 TEST REPORT

The test report shall show the method used and the result obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional (for example reaction time — see 9.2), as well as any circumstances that may have influenced the result.

The report shall include all details required for complete identification of the sample.