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Animal and vegetable fats and oils — Determination of peroxide value

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

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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 3960 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

This third edition cancels and replaces the second edition (ISO 3960:1998), which has been technically revised.

Annex A of this International Standard is for information only.

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Animal and vegetable fats and oils — Determination of peroxide value

1 Scope

This International Standard specifies a method for the determination of the peroxide value of animal and vegetable fats and oils.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

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

3 Term and definition

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

3.1
peroxide value
quantity of those substances in the sample, expressed in terms of active oxygen, which oxidize potassium iodide under the conditions specified in this International Standard, divided by the mass of the test portion

NOTE 1 Peroxide value is usually expressed in industry as milliequivalents per kilogram.

NOTE 2 Peroxide value may also be expressed (in SI units) as millimoles per kilogram. The value expressed in millimoles per kilogram is half that expressed in milliequivalents per kilogram (see clause 10).

4 Principle

A test portion, in solution in acetic acid and isooctane, is treated with a solution of potassium iodide. The liberated iodine is titrated with a standard volumetric sodium thiosulfate solution.

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise stated. All reagents and the water shall be free of dissolved oxygen.

5.1 Water, complying with grade 3 of ISO 3696.

5.2 Glacial acetic acid, freed from oxygen by purging with a current of pure and dry inert gas (carbon dioxide or nitrogen).

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WARNING — Glacial acetic acid is moderately toxic by ingestion and inhalation. It is a strong irritant to skin and tissue.

5.3 Isooctane, freed from oxygen by purging with a current of pure and dry inert gas (carbon dioxide or nitrogen).

WARNING — Isooctane is flammable and a fire risk. Explosive limits in air are 1,1 % **(by volume) to** 6,0 % **(by volume). It is toxic by ingestion and inhalation. A properly operating fume hood should be used when working with this solvent.**

5.4 Acetic acid/isooctane solution [60:40 (by volume)], prepared by mixing 3 volumes of glacial acetic acid (5.2) with 2 volumes of isooctane (5.3).

5.5 Potassium iodide solution, saturated, recently prepared and free from free iodine and iodates.

Make sure the solution remains saturated as indicated by the presence of undissolved crystals. Store in the dark. Test daily by adding 2 drops of starch solution (5.8) to 0,5 ml of the potassium iodide solution in 30 ml of the acetic acid/isooctane solution (5.4). If a blue colour is formed which requires more than 1 drop of sodium thiosulfate solution (5.7) to discharge the colour, discard the potassium iodide solution and prepare a fresh solution.

5.6 Sodium thiosulfate solution, $c(Na_2S_2O_3) = 0{,}1$ mol/l, standardized just before use.

Dissolve 24,9 g of sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$) in distilled water and dilute to 1 litre.

5.7 Sodium thiosulfate solution, $c(Na_2S_2O_3) = 0{,}01$ mol/l, standardized just before use.

Prepare by diluting previous solution (5.6).

5.8 Starch solution, 5 g/l.

Mix 1 g of starch and a small amount of cold distilled water. Add this mixture, while stirring, to 200 ml of boiling water. Add 250 mg of salicylic acid as preservative and boil for 3 min. Immediately remove from the heat and cool.

If long storage is required, keep the solution in a refrigerator at between 4 °C and 10 °C. A fresh starch solution shall be prepared when the endpoint of the titration from blue to colourless fails to be sharp. If stored under refrigeration, the starch solution should be stable for about 2 to 3 weeks.

The sensitivity of the starch solution may be tested as follows. To 5 ml of the starch solution in 100 ml of water, add 0,05 % of potassium iodide solution (5.5) and 1 drop of 0,05 % sodium hypochlorite solution. It is essential that the deep blue colour produced be discharged by 0,05 ml of sodium thiosulfate solution (5.6).

6 Apparatus

All equipment used shall be free from reducing or oxidizing substances. Do not grease ground glass surfaces.

Usual laboratory apparatus and, in particular, the following.

6.1 Conical flasks, of 250 ml capacity, with ground glass stoppers.

7 Sampling

It is important that the laboratory receive a sample that 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 [1].

Ensure that the sample is taken and stored away from strong light, kept cold, and contained in completely filled dark-coloured glass containers, hermetically sealed with ground glass stoppers.

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8 Preparation of test sample

Ensure that the packaging of the fat or oil is not damaged, and is well closed. When parameters other than the peroxide value have to be investigated, the test portion for the peroxide value shall be taken first from the laboratory sample.

Prepare the test sample in accordance with ISO 661.

9 Procedure

9.1 General

Carry out the test in artificial light or diffuse daylight.

NOTE If it is required to check that the repeatability requirement (see 11.2) is met, carry out two single determinations in accordance with 9.2 and 9.3.

9.2 Test portion

Rinse the conical flask (6.1) with a current of pure dry inert gas (carbon dioxide or nitrogen). Into the flask, weigh a mass of the sample to the accuracy given in Table 1 and in accordance with the expected peroxide value.

Table 1 — Mass of test portion and accuracy of weighing

Expected peroxide value meq/kg	Mass of test portion g	Weighing accuracy g
0 to 12	5,0 to 2,0	± 0,01
12 to 20	2,0 to 1,2	± 0,01
20 to 30	1,2 to 0,8	± 0,01
30 to 50	0,8 to 0,5	± 0,001
50 to 90	0,5 to 0,3	± 0,001

9.3 Determination

Add 50 ml of acetic acid/isooctane solution (5.4) to the conical flask and replace the stopper. Swirl the flask until the sample has dissolved. Using a suitable volumetric pipette, add 0,5 ml of saturated potassium iodide solution (5.5) and replace the stopper. Allow the solution to react for 1 min ± 1 s, thoroughly shaking it at least three times during this period, then immediately add 30 ml of distilled water.

Titrate the solution with sodium thiosulfate solution (5.7), adding it gradually and with constant, vigorous agitation, until the yellow iodine colour has almost disappeared. Add about 0,5 ml of starch solution (5.8) and continue the titration with constant agitation, especially near the endpoint, to liberate all of the iodine from the solvent layer, adding the sodium thiosulfate solution (5.7) drop by drop until the blue colour just disappears.

There is a 15 s to 30 s delay in neutralizing the starch indicator for peroxide values of 70 meq/kg and greater, due to the tendency of isooctane to float on the surface of the aqueous medium and the time necessary to mix adequately the solvent and the aqueous titrant, thus liberating the last traces of iodine. A small amount [0,5 % to 1,0 % (m/m)] of a high HLB emulsifier (e.g. Tween 60)[1)] may be added to the reaction mixture to retard the phase separation and decrease the time lag in the liberation of iodine.

1) Tween 60 is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

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In the case of fats of low solubility, such as hard fats and animal fats, the following procedure shall be used.

Add 20 ml isooctane to the flask, replace the stopper, and dissolve the sample by swirling. Immediately add 30 ml of acetic acid.

9.4 Blank determination

Carry out a blank determination in parallel with the sample determination. If the result of the blank determination exceeds 0,1 ml of 0,01 mol/l sodium thiosulfate solution (5.7), replace the impure reagents and repeat the sample determination.

10 Expression of results

10.1 Peroxide value expressed in milliequivalents of active oxygen per kilogram

Calculate the peroxide value, P, by the following equation:

$$P = \frac{1\,000\,(V - V_0)\,c}{m}$$

where

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

V_0 is the volume of sodium thiosulfate solution used for the blank determination, in millilitres;

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

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

10.2 Peroxide value expressed in millimoles per kilogram

The peroxide value, P', may be calculated, if required, from the following equation:

$$P' = \frac{1\,000\,(V - V_0)\,c}{2m}$$

11 Precision

11.1 Interlaboratory tests

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

11.2 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, will in not more than 5 % of cases be greater than 10 % of the mean of the two results, for peroxide values less than or equal to 10 meq/kg.

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11.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, will in not more than 5 % of cases be greater than 75 % of the mean of the two results, for peroxide values less than or equal to 10 meq/kg.

12 Test report

The test report shall specify:

— all information necessary for the complete identification of the sample;

— the sampling method used, if known;

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

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

— the test result(s) obtained, and the units in which the result is expressed;

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

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