
**Animal and vegetable fats and oils —
Determination of peroxide value —
Iodometric (visual) endpoint
determination**

*Corps gras d'origines animale et végétale — Détermination de l'indice
de peroxyde — Détermination avec point d'arrêt iodométrique*

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Foreword

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The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

This fourth edition cancels and replaces the third edition (ISO 3960:2001), which has been technically revised.

Introduction

Over a period of many years, various methods have been developed for the determination of peroxides in fats and oils. The general principle of most of the methods is the liberation of iodine from potassium iodide in an acid medium. The method according to Wheeler was standardized more than 50 years ago by different standardization bodies, and it is widely used to control commodities by producers, receivers and official laboratories. In national and international food legislation (including the Codex Alimentarius), acceptable limits for the peroxide values are often specified. Due to anomalies in the reproducibility of the results, it was noticed that there are slight differences between the standardized methods. A very important point is the dependence of the result on the amount of sample used for the determination. As the determination of the peroxide value (PV) is a highly empirical procedure, ISO/TC 34/SC 11 has decided to fix the sample mass at 5 g for $PV > 1$, and at 10 g for $PV \leq 1$, and to limit the applicability of this method to animal and vegetable fats and oils with peroxide values from 0 milliequivalents to 30 milliequivalents of active oxygen per kilogram. The user of this International Standard should be aware that the results obtained can be slightly lower than with previous standards.

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Animal and vegetable fats and oils — Determination of peroxide value — Iodometric (visual) endpoint determination

1 Scope

This International Standard specifies a method for the iodometric determination of the peroxide value of animal and vegetable fats and oils with a visual endpoint detection. The peroxide value is a measure of the amount of oxygen chemically bound to an oil or fat as peroxides, particularly hydroperoxides.

The method is applicable to all animal and vegetable fats and oils, fatty acids and their mixtures with peroxide values from 0 meq to 30 meq (milliequivalents) of active oxygen per kilogram. It is also applicable to margarines and fat spreads with varying water content. The method is not suitable for milk fats and is not applicable to lecithins.

It is to be noted that the peroxide value is a dynamic parameter, whose value is dependent upon the history of the sample. Furthermore, the determination of the peroxide value is a highly empirical procedure and the value obtained depends on the sample mass. It is stressed that, due to the prescribed sample mass, the peroxide values obtained can be slightly lower than those obtained with a lower sample mass.

NOTE 1 A preferred method for the iodometric determination of the peroxide value for milk fats is specified in ISO 3976.

NOTE 2 A method for the potentiometric determination of the peroxide value is given in ISO 27107.

2 Normative references

The following referenced documents are indispensable for the application 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.

3.1

peroxide value

PV

quantity of those substances in the sample, expressed in terms of active oxygen, that oxidize potassium iodide under the conditions specified in this International Standard

NOTE The peroxide value is usually expressed in milliequivalents (meq) of active oxygen per kilogram of oil, but it may also be expressed (in SI units) as millimoles (mmol) of active oxygen per kilogram of oil. The value expressed in millimoles of active oxygen per kilogram is half that expressed in milliequivalents of active oxygen per kilogram. Multiplication of the peroxide value (meq of active oxygen per kg) by the equivalent mass of oxygen (equalling 8) gives the milligrams of active oxygen per kilogram of oil.

4 Principle

The test sample is dissolved in isooctane and glacial acetic acid, and potassium iodide is added. The iodine liberated by the peroxides is determined iodometrically (visually) with a starch indicator and a sodium thiosulfate standard solution. The endpoint of the titration is determined iodometrically (visually).

5 Reagents

WARNING — Attention is drawn to the national regulations that specify the handling of hazardous substances and users' obligations thereunder. Technical, organizational and personal safety measures shall be followed.

Use only reagents of recognized analytical grade, unless otherwise specified. All reagents shall be free of dissolved oxygen.

5.1 Water, demineralized, boiled and cooled down to 20 °C.

5.2 Glacial acetic acid, mass fraction of 100 %; degassed in an ultrasonic bath under vacuum or by purging with a current of pure and dry inert gas (carbon dioxide or nitrogen).

5.3 Isooctane, degassed in an ultrasonic bath under vacuum or by purging with a current of pure and dry inert gas (carbon dioxide or nitrogen).

5.4 Glacial acetic acid/isooctane solution, prepared by mixing 60 ml of glacial acetic acid and 40 ml of isooctane (volume fraction of glacial acetic acid: $\varphi = 60 \text{ ml}/100 \text{ ml}$, and volume fraction of isooctane: $\varphi = 40 \text{ ml}/100 \text{ ml}$).

The mixture is degassed in an ultrasonic bath under vacuum or by purging with a current of pure and dry inert gas (carbon dioxide or nitrogen).

5.5 Potassium iodide, free from iodine and iodates.

5.6 Saturated potassium iodide solution, mass concentration $\rho = 175 \text{ g}/100 \text{ ml}$. Dissolve approximately 14 g of potassium iodide in approximately 8 g of freshly boiled water at room temperature.

Make sure the solution remains saturated (undissolved crystals). Store in the dark and prepare freshly every day. Test the solution as follows: add two drops of starch solution to 0,5 ml of the potassium iodide in 30 ml of the glacial acetic acid/isooctane solution. If more than one drop of the 0,1 mol/l sodium thiosulfate standard solution is needed to form a blue colour, discard the potassium iodide solution.

5.7 0,1 N sodium thiosulfate standard solution, $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,1 \text{ mol/l}$.

Use only freshly boiled water for the preparation of this solution, possibly purged with nitrogen. This solution can be used for one month and is stored in an amber-stained bottle.

5.8 0,01 N sodium thiosulfate standard solution, $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,01 \text{ mol/l}$ (see 9.2).

It is necessary to prepare this solution freshly from the 0,1 mol/l sodium thiosulfate standard solution before use or to determine the titre daily. As experience shows, the stability is limited and depends upon the pH value and the content of free carbon dioxide. Use only freshly boiled water for the dilution, possibly purged with nitrogen.

5.9 Starch solution, mass concentration $\rho = 1 \text{ g}/100 \text{ ml}$. Mix 0,5 g of starch and a small amount of cold water. Add this mixture, while stirring, to 50 ml of boiling water, boil it for a few seconds and cool immediately.

The solution shall be freshly prepared every day.

It is recommended to use potato starch for iodometry as this starch gives a darker blue colour. Equivalent reagents may also be used.

5.10 Potassium iodate (KIO₃) volumetric standard, secondary reference material, traceable to the National Institute of Standards and Technology (NIST), Gaithersburg, MD, USA.

5.11 Hydrochloric acid, $c(\text{HCl}) = 4 \text{ mol/l}$.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Erlenmeyer flask, of 250 ml capacity, with ground neck and ground glass stopper.

6.2 Burette, of 10 ml or 25 ml capacity, graduated in at least 0,05 ml, preferably with automatic zero adjustment (pellet titrators).

6.3 Manual or automatic dosing unit, of 20 ml capacity, with a resolution of at least 10 μl and an accuracy of $\pm 0,15 \%$ (e.g. a piston burette).

6.4 Pipettes, of 0,5 ml, 1 ml, 10 ml and 100 ml capacity (or automatic pipettes).

6.5 Measuring cylinders, of 50 ml and 100 ml capacity.

6.6 Analytical balance, with a readability of 0,001 g.

6.7 Magnetic stirrer, with magnetic stirring rod (of 2,5 cm) and heating plate.

6.8 Volumetric flask, of 1 000 ml capacity.

6.9 Volumetric flask, of 250 ml capacity.

6.10 Volumetric flask, of 500 ml capacity.

6.11 Microwave oven.

A microwave oven may be used to melt solid samples in an easy and quick manner. Careful and proper use of a microwave oven will not lead to any increase in the peroxide value. The suitable conditions shall be tested in advance.

7 Sampling

A representative sample should have been sent to the laboratory. It should not have 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 test sample in accordance with ISO 661.

The test sample for the determination of peroxide value shall be taken first and the peroxide value shall be determined immediately.

Homogenize the sample, preferably without heating and without aeration. Avoid direct solar radiation. Heat solid samples carefully to 10 °C above their melting point. Samples with visible impurities shall be filtered; the filtration shall be noted in the test report.

For some products, the amount of extracted fat/oil can be lower than 5 g, or the peroxide value of the fat/oil can be over 30 meq of active oxygen per kilogram. In these cases, the user should choose a smaller sample mass [see Clause 12 f)].

9 Procedure

9.1 General

Carry out all steps in diffuse daylight or in artificial light. Avoid direct exposure to sunlight. Observe that all vessels are free from oxidizing or reducing compounds.

Store the sodium thiosulfate standard solutions in amber-stained bottles.

9.2 Preparation and titre determination of the 0,01 N sodium thiosulfate standard solution

9.2.1 Preparation of the 0,01 N sodium thiosulfate standard solution

By means of a pipette (6.4), transfer 100 ml of the 0,1 N sodium thiosulfate standard solution (5.7) to a volumetric flask of 1 000 ml capacity (6.8). Dilute to the mark with freshly boiled water (5.1). After homogenization, transfer the obtained 0,01 N sodium thiosulfate standard solution to an amber-stained bottle.

Prepare daily the 0,01 N sodium thiosulfate standard solution freshly from the 0,1 N sodium thiosulfate standard solution before use, or determine the titre. As experience shows, the stability is limited and depends upon the pH value and the content of free carbon dioxide. Use only freshly boiled water for the dilution, possibly purged with nitrogen.

9.2.2 Determination of the titre of the 0,01 N sodium thiosulfate standard solution (factor determination)

Weigh, to the nearest 0,001 mg, 0,27 g to 0,33 g of potassium iodate (KIO₃) into a volumetric flask (250 ml or 500 ml) (6.9 or 6.10) and dilute to the mark with freshly boiled water (5.1), cooled down to room temperature.

By means of a pipette (6.4), transfer 5 ml or 10 ml of this potassium iodate solution into a 250 ml Erlenmeyer flask (6.1). Add 60 ml of freshly boiled water, 5 ml of 4 mol/l hydrochloric acid, and 25 mg to 50 mg of potassium iodide (5.5) or 0,5 ml of the saturated potassium iodide solution (5.6).

Titrate this solution using the iodometric (visual) method to determine the factor of the 0,01 N sodium thiosulfate standard solution (see 9.2.1).

Calculate the exact concentration, c_{stand} , of the 0,01 N sodium thiosulfate standard solution using the following formula:

$$c_{\text{stand}} = \frac{m_{\text{KIO}_3} \times V_1 \times 6 \times 1\,000 \times \rho_{\text{KIO}_3}}{M_{\text{KIO}_3} \times V_2 \times V_3 \times c_{\text{thio}} \times 100}$$

where

- m_{KIO_3} is the mass of potassium iodate, in grams;
- 6 is the equivalent mass for the titre ($1 \text{ mol KIO}_3 = 3 \text{ mol I}_2$);
- V_1 is the volume of the potassium iodate solution used for the titre determination (5 ml or 10 ml);
- V_2 is the total volume of the potassium iodate solution, in millilitres (250 ml or 500 ml);
- V_3 is the volume of the 0,01 N sodium thiosulfate standard solution used for the determination, in millilitres;
- ρ_{KIO_3} is the mass concentration of the potassium iodate, in grams per 100 g;
- M_{KIO_3} is the molecular mass of the potassium iodate (214 g/mol);
- c_{thio} is the approximate concentration of the 0,01 N sodium thiosulfate standard solution, in moles per litre (= 0,01).

9.3 Determination of peroxide value

9.3.1 Rinse the carefully cleaned Erlenmeyer flask (6.1) with nitrogen or carbon dioxide. Weigh the following into the flask, to the nearest 0,1 mg:

- 5,0 g \pm 0,1 g of test sample for expected peroxide values from > 1 to 30; or
- 10,0 g \pm 0,1 g of test sample for expected peroxide values from 0 to 1.

Rinse the Erlenmeyer flask with the glacial acetic acid/isooctane solution (5.4) prior to use to ensure that the flask does not contain any oxidizing or reducing substances.

9.3.2 Dissolve the test sample in 50 ml of the glacial acetic acid/isooctane solution by gentle swirling.

In the case of fats with high melting points (hard fats and animal fats), carefully add to the melted fat 20 ml of isooctane (5.3) by gentle swirling, and then immediately add 30 ml of glacial acetic acid (5.2). Also warm the test sample gently, where necessary.

9.3.3 Add 0,5 ml of the saturated potassium iodide solution (5.6). Close the Erlenmeyer flask and mix with a magnetic stirrer (6.7) without creating a large vortex, or manually without aeration for exactly 60 s (use a timer accurate to ± 1 s).

9.3.4 Open the Erlenmeyer flask, immediately add 100 ml of demineralized water, rinse the ground glass stopper and swirl.

9.3.5 Immediately titrate the liberated iodine with the 0,01 N sodium thiosulfate standard solution (5.8) from yellow orange to pale yellow and, after addition of 0,5 ml of starch solution (5.9), from violet to colourless. Stop the titration as soon as the solution is colourless for 30 s.

NOTE 1 The phase being titrated is the lower one. There is a delay of 15 s to 30 s in the change of colour with the 0,01 N sodium thiosulfate standard solution (5.8).

NOTE 2 In the case of peroxide values below 1, the starch solution can be added at the beginning of the titration.

9.3.6 In a parallel blank test, not more than 0,1 ml of the 0,01 N thiosulfate solution shall be used. If the blank test is higher, then replace the saturated potassium iodide solution as it could be unsuitable.