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Animal and vegetable fats and oils — Determination of peroxide value — Potentiometric end-point determination

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

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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 27107 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

This corrected version of ISO 27107:2008 incorporates the following corrections:

— Introduction, lines 9 and 10, “greater than” and “less than or equal to” replace “>” and “⩽”, respectively;

— Introduction, line 11, “0 mmol to 15 mmol” has become “0 meq to 30 meq”;

— 5.6, final sentence, has been reedited to correct details of blue colour formation;

— 6.5 now contains a readability figure of 0,000 1 g, not 0,001 g;

— 9.2.2, line 1, now refers to 0,001 g instead of 0,001 mg;

— 9.2.2, paragraph 4, now contains a reedited calculation of the factor, using symbol F rather than f;

— the heading “10.1 Calculation” has been deleted;

— Clause 10, paragraph 1, has been revised to incorporate factor, F, from the revised 9.2.2;

— In Figure A.1, “PV =” has become “PV:” (five times).

Introduction

Over many years, various methods have been developed for the determination of peroxides in fats and oils. Their general principle is the liberation of iodine from potassium iodide in an acid medium. The method according to Wheeler (Reference [6]) was first adopted in standards more than 50 years ago by different bodies, and is widely used to control commodities by producers, receivers, and official laboratories. In national and international food legislation (including Codex Alimentarius), acceptable limits for 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 greater than 1, and at 10 g for PV less than or equal to 1, and to limit the applicability of this method to animal and vegetable fats and oils with peroxide values from 0 meq to 30 meq of active oxygen per kilogram. The users of this International Standard should be aware that the results obtained can be slightly lower than with previous standards.

Animal and vegetable fats and oils — Determination of peroxide value — Potentiometric end-point determination

1 Scope

This International Standard specifies a method for the potentiometric end-point determination of the peroxide value, in milliequivalents of active oxygen per kilogram, of animal and vegetable fats and oils.

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 of active oxygen per kilogram. It is also applicable to margarines and fat spreads with varying water content. The method is not applicable to milk fats or lecithins.

NOTE A method for the iodometric (visual) determination of the peroxide value is given in ISO 3960. For milk fats, a method is specified in ISO 3976.

2 Normative references

The following referenced documents are indispensable for the application of this document. 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 of active oxygen per kilogram of oil, but it may also be expressed (in SI units) as millimoles 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 (milliequivalents of active oxygen per kilogram) by the equivalent mass of oxygen (equalling 8) gives the active oxygen mass fraction in milligrams per kilogram of oil.

4 Principle

The sample is dissolved in isooctane and glacial acetic acid, and potassium iodide is added. The iodide liberated by the peroxides is determined volumetrically with a sodium thiosulfate standard solution. The end-point of the titration is determined electrochemically.

5 Reagents

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

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

5.1 Water, distilled, boiled, and cooled to 20 °C.

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

5.3 Isooctane (2,2,4-trimethylpentane), degassed in an ultrasonic bath under vacuum or by purging with a stream of pure and dry inert gas (carbon dioxide or nitrogen).

5.4 Glacial acetic acid/isooctane solution, prepared by mixing 60 ml glacial acetic acid (5.2) and 40 ml isooctane (5.3). Volume fraction of glacial acetic acid: φ = 60 ml/100 ml; volume fraction of isooctane: φ = 40 ml/100 ml.

The mixture is degassed in an ultrasonic bath under vacuum or by purging with a stream 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 ρ(KI) = 175 g/100 ml.

Dissolve approximately 14 g potassium iodide in approximately 8 g freshly boiled water (5.1) at room temperature. Make sure the solution remains saturated (i.e. some undissolved crystals remain in the container). 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 solution in 30 ml of the glacial acetic acid/isooctane solution (5.4). If a blue colour is formed and if more than one drop of sodium thiosulfate standard solution (5.7) is needed to remove it, discard the potassium iodide solution.

5.7 0,1 N sodium thiosulfate standard solution, amount of substance concentration $c(Na_2S_2O_3)$ = 0,1 mol/l.

Use only freshly boiled water (5.1) for the preparation of this solution, possibly purged with nitrogen. This solution can be used for 1 month and shall be stored in an amber-stained bottle.

5.8 0,01 N sodium thiosulfate standard solution, amount of substance concentration $c(Na_2S_2O_3)$ = 0,01 mol/l.

Pipette (6.3) 100 ml of the 0,1 N sodium thiosulfate standard solution (5.7) into a volumetric flask of capacity 1 000 ml (6.9). Make up to the mark with water (5.1). After homogenization, transfer the obtained 0,01 N sodium thiosulfate standard solution to an amber-stained bottle.

Prepare the 0,01 N sodium thiosulfate standard solution freshly from the 0,1 N sodium thiosulfate standard solution just before use or 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 (5.1) for the dilution, possibly purged with nitrogen.

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

5.10 Hydrochloric acid, amount of substance concentration c(HCl) = 4 mol/l.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 **Automatic titrator** with processor, dosing device, stirrer and electrodes.

If other apparatus is used, the procedure shall be optimized for the relevant apparatus. The apparatus shall be able to perform a dynamic titration (fast at the beginning, slow near the end-point). This is necessary to minimize the titration time whilst achieving a slow titration near the end-point.

6.2 **Combined platinum electrode**.

6.3 **Pipettes**, of capacities 0,5 ml, 1 ml, 10 ml and 100 ml. Suitable automatic pipettes may also be used.

6.4 **Measuring cylinders**, of capacities 50 ml and 100 ml.

6.5 **Analytical balance**, readable to 0,000 1 g.

6.6 **Magnetic stirrer**, with magnetic stirring rod of length 25 mm, and heating plate.

6.7 **Erlenmeyer flask**, of capacity 250 ml.

6.8 **Beaker**, of capacity 250 ml, and of tall form.

6.9 **Volumetric flask**, of capacity 1 000 ml.

6.10 **Volumetric flask**, of capacity 250 ml.

6.11 **Volumetric flask**, of capacity 500 ml.

6.12 **Microwave oven**.

6.13 **Amber-stained bottles,** of capacity 1 000 ml.

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 the test sample

Prepare the test sample in accordance with ISO 661.

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

Take the test portion for the determination of peroxide value first, before taking test portions for any other test, and determine the peroxide value immediately.

9 Procedure

9.1 General

Carry out all steps in diffuse daylight or in artificial light. Avoid direct exposure to sunlight. Ensure 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 0,01 N sodium thiosulfate standard solution

See 5.8.

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

Weigh, to the nearest 0,001 g, 0,27 g to 0,33 g potassium iodate(V) into a volumetric flask [250 ml (6.10) or 500 ml (6.11)] and make up to the mark with water (5.1).

Pipette (6.3) 5 ml or 10 ml of this potassium iodate(V) solution into a 250 ml beaker (6.8). Add 60 ml freshly boiled water (5.1), 5 ml of HCl (5.10) and 0,5 ml of the saturated potassium iodide solution (5.6).

Titrate this solution with the 0,01 N sodium thiosulfate standard solution to determine the factor of the 0,01 N sodium thiosulfate standard solution.

Calculate the factor, F, of the 0,01 N sodium thiosulfate solution using the following formula:

$$F = \frac{m_{KIO_3} \cdot V_1 \cdot 6 \cdot 1\,000 \cdot w_{KIO_3}}{M_{KIO_3} \cdot V_2 \cdot V_3 \cdot c_{thio} \cdot 100}$$

where

m_{KIO_3} is the mass of potassium iodate, in grams;

6 is the equivalent mass for the titer (1 mol KIO_3 ⇔ 3 mol I_2);

V_1 is the volume of the potassium iodate solution, used for the titer determination (5 ml or 10 ml);

V_2 is the total volume of potassium iodate solution, in millilitres (250 ml or 500 ml);

V_3 is the volume of 0,01 N thiosulfate solution, used for the determination, in millilitres;

w_{KIO_3} is the purity of potassium iodate in g/100 g;

M_{KIO_3} is the molecular mass of potassium iodate (214 g/mol);

c_{thio} is the concentration of the sodium thiosulfate standard solution in moles per litre (0,01 mol/l).

9.3 Determination of peroxide value

9.3.1 Purge the carefully cleaned Erlenmeyer flask (6.7) with nitrogen or carbon dioxide. Weigh into the flask, to the nearest 0,1 mg:

a) either a 5,0 g ± 0,1 g test portion for expected peroxide values from > 1 to 30;

b) or a 10,0 g ± 0,1 g test portion for expected peroxide values from 0 to 1.

The peroxide value is a dynamic value, dependent upon the history of the test sample. Furthermore, the determination of the peroxide value is a highly empirical procedure and the value obtained depends on the mass of the test portion. The user of this International Standard should be aware that due to the prescribed test portion mass, the peroxide values obtained can be slightly lower than those obtained with test portions of lower mass. 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 active oxygen per kilogram. In these cases, the user should choose a smaller test portion mass. As the test portion mass influences the result, report it together with the result.

9.3.2 Dissolve the test portion in 50 ml of the glacial acetic acid/isooctane solution (5.4) 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 portion gently, if necessary.

9.3.3 Add the magnetic stirring rod (6.6) and 0,5 ml of the saturated potassium iodide solution (5.6), stir the test portion on the stirrer of the automatic titrator (6.1) for exactly 60 s (use a timer accurate to ±1 s) at a medium speed to avoid spraying.

9.3.4 Immediately add 30 ml to 100 ml of water (5.1). The amount depends on the apparatus used.

NOTE The greater amount of water is necessary due to phase inversion and depends upon the apparatus used. The phase being titrated is the lower one. With higher amounts of water, the potentiometric difference between the starting and end-point of the titration is bigger (~100 mV). This results in a titration curve with a sharp turning point.

9.3.5 Immerse the combined platinum electrode (6.2) into the test sample and start the titration with the 0,01 N sodium thiosulfate standard solution (5.8) while stirring at high speed.

9.3.6 In a parallel blank test, not more than 0,1 ml of the 0,01 N thiosulfate solution shall be used.

9.3.7 Most titration equipment evaluates the equivalent point automatically; otherwise determine the end-point graphically using the point of inflection method.

NOTE Typical end-point titration curves are shown in Figure A.1.

10 Calculation and expression of results

Calculate the peroxide value (commonly known in the industry as “PV”), in milliequivalents of active oxygen per kilogram, using the following formula:

$$\frac{(V - V_0)\cdot c_{\text{thio}}\cdot F\cdot 1000}{m}$$

where

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

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

F is the factor of the 0,01 N sodium thiosulfate solution, determined according to 9.2;

