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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 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 (see 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.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso. org/iso/foreword. html.

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

This fifth edition cancels and replaces the fourth edition (ISO 3960:2007), of which it constitutes a minor revision to exclude fat coming from milk and milk products.

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 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 user of this document 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 — Iodometric (visual) endpoint determination

1 Scope

This document 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.

Milk and milk products (or fat coming from milk and milk products) are excluded from the scope of this document.

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 documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp/

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 document

Note 1 to entry: 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 of that expressed in milliequivalents of active oxygen per kilogram. Multiplication of the peroxide value (meq of active oxygen per kilogram) 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: φ = 60 ml/100 ml, and volume fraction of isooctane: φ = 40 ml/100 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 ρ(KI) = 175 g/100 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 (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, $c(Na_2S_2O_3)$ = 0,1 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, $c(Na_2S_2O_3)$ = 0,01 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 ρ = 1 g/100 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 (KIO_3) volumetric standard, secondary reference material, traceable to the National Institute of Standards and Technology (NIST), Gaithersburg, MD, USA.

5.11 Hydrochloric acid, $c(HCl)$ = 4 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 ±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, readable to 0,000 1 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.