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Animal and vegetable fats and oils — Determination of water content — Karl Fischer method (pyridine free)

Corps gras d'origines animale et végétale — Détermination de la teneur en eau — Méthode de Karl Fischer (sans pyridine)

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Contents

Page

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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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 third edition cancels and replaces the second edition (ISO 8534:2008), of which it constitutes a minor revision to exclude the applicability for fat coming from milk and milk products.

Introduction

The determination of the water content of fats and oils according to Karl Fischer is carried out by two different procedures. This document specifies the volumetric Karl Fischer method for the determination of higher milligram levels of water (high level moisture). It is used for samples having between 1 mg and 100 mg of water in the sample.

Annex B specifies a coulometric titration, which requires between 10 µg and 10 mg water in the sample. The coulometric method is more sensitive than the volumetric method and permits the determination of lower water contents.

Animal and vegetable fats and oils — Determination of water content — Karl Fischer method (pyridine free)

1 Scope

This document specifies a method for the determination of the water content of animal and vegetable fats and oils (hereinafter referred to as fats) using Karl Fischer apparatus and a reagent which is free of pyridine.

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

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 http://www.iso.org/obp

3.1
water content
mass, in grams per 100 g of sample, of water as determined in accordance with the method specified in this document

Note 1 to entry: The water content is expressed as a percentage mass fraction.

4 Principle

Dissolved fat is titrated against an iodine solution and sulfur dioxide (SO_2) is oxidized by iodine in the presence of water. In principle, the chemical reaction in Formula (1) takes place:

$$H_2O + I_2 + SO_2 + CH_3OH + 3RN \rightarrow [RNH]SO_4CH_3 + 2[RNH]I \qquad (1)$$

The alcohol reacts with SO_2 and a nitrogenous base (RN) to form an intermediate alkylsulfite salt, which is then oxidized by iodine to an alkylsulfate salt. This oxidation reaction consumes water contained in the sample. The end point is monitored potentiometrically.

5 Reagents

WARNING — Comply with any local regulations which specify the handling of hazardous substances. Technical, organizational and personal safety measures shall be followed.

It is recommended that "ready for use" working solvents be used, either one-component reagents (5.1.1) or two-component reagents (5.1.2). Reagents with a titre of 1 mg and 2 mg water per millilitre are required for acceptable performance.

5.1 Karl Fischer reagents, consist of one-component reagents or two-component reagents for volumetric determination.

5.1.1 One-component reagents, contain all the reactant in the titrant solution: iodine, sulfur dioxide, and imidazole, dissolved in a suitable alcohol. Methanol is typically used as the working medium in the titration cell.

Absolute methanol is the solvent of choice. But for fats and oils, use a mixture of absolute methanol and absolute chloroform (the methanol content should be at least 25 % volume fraction, or optimally 50 % volume fraction).

5.1.2 Two-component reagents, consist of all necessary reactants for the titration, but in two different solutions. The titrating agent (usually known as the titrant) contains only iodine and methanol, while the solvent containing the other Karl Fischer reaction components is used as the working medium in the titration cell.

5.2 Water standard, commercially prepared standard with a certified concentration of 10 mg/g (1,0 % mass fraction).

6 Apparatus

Usual laboratory apparatus and, in particular the following.

6.1 Karl Fischer apparatus, set up according to the manufacturer's recommendations for the determination of water in fats and oils. Set up and conduct protocols for routine maintenance as recommended by the manufacturer. Use an airtight vessel and do not place the instrument in high humidity areas. Do not place instruments or handle samples near water sources, such as taps, sinks, and dishwashers in the laboratory.

6.2 Analytical balance, readable to the nearest 0,1 mg.

6.3 Syringes, of capacity 1 ml, 2 ml, 5 ml, 10 ml, and 20 ml.

To ensure accurate and reproducible results from the water standard, use a glass gastight syringe. For water standard 10,0, use a 10 ml syringe and for either the water standard 1,00 or 0,10, use a 5 ml syringe. In addition to the appropriate size syringe, use a needle that is long enough to allow for a subsurface injection when injecting through the instrument's septum.

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 document. A recommended sampling method is given in ISO 5555.

8 Preparation of the test sample

Prepare the test sample in accordance with ISO 661.

The determination of water is conducted by adjusting the sample size to have between 1 mg and 100 mg water for the volumetric titration (the main body of this document) and between 10 µg and 10 mg for the coulometric titration (Annex B) using Karl Fischer instruments and reagents which have been validated with standard water solutions over the necessary range. For the volumetric determination, a minimum amount of 0,5 ml Karl Fischer reagent shall be used for the titration.

9 Procedure

9.1 Titre

9.1.1 The titre shall be determined daily for each bottle of titrant.

9.1.2 Prepare the instrument according to the manufacturer's recommendations for calibration.

9.1.3 Add 20 ml to 40 ml of working solvent (5.1) to the titration vessel. The solvent should cover the platinum electrodes.

9.1.4 Titrate the vessel to a stable dry end point.

CAUTION — Take care not to overtitrate.

9.1.5 Determine the titre of the titrant using the water standard (5.2) and a syringe (6.3). Sample mass is determined by difference.

9.1.5.1 Weigh, to the nearest 0,1 mg, approximately 1 g of the water standard into a syringe, placed on the analytical balance (6.2).

Upon opening the ampoule, withdraw a small portion of the standard to rinse the syringe; 1 ml to 2 ml is sufficient. Rinse the entire interior of the syringe and discard the rinsings. Then, immediately transfer the remaining standard to the syringe and expel any air bubbles. Using mass by difference make at least three injections from the syringe.

9.1.5.2 When the mass displayed is stable, tare the balance.

9.1.5.3 Inject the water sample into the titration vessel and close the vessel.

9.1.5.4 Place the syringe back on the balance. Record the mass of the water injected to the nearest 0,1 mg. The mass will be displayed as a negative value.

9.1.5.5 Enter the sample mass in the instrument.

9.1.6 Start the titration and record the titre when a stable end point is reached. Some instruments may require calculation of titre from the displayed percentage of water.

9.1.7 Average a minimum of three titre determinations. Record the arithmetic average.

9.1.8 Update the instrument titre value with the new setting.