INTERNATIONAL STANDARD

ISO 8534

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Animal and vegetable fats and oils — Determination of water content — Karl Fischer method

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

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Reference number
ISO 8534:1996(E)

Foreword

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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.

International Standard ISO 8534 was prepared by Technical Committee ISO/TC 34, *Agricultural food products,* Subcommittee SC 11, *Animal and vegetable fats and oils.*

Annexes A to D of this International Standard are for information only.

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Animal and vegetable fats and oils — Determination of water content — Karl Fischer method

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WARNING — The chemicals pyridine, methanol, sulfur dioxide and chloroform are poisons. Therefore special care shall be taken during analysis and removal of the residues. Consult the restrictions for using these chemicals (poisons law, threshold limits for exposure of workers) in order to protect the environment and workers.

1 Scope

This International Standard specifies a method using Karl Fischer reagent for the determination of the water content of animal and vegetable fats and oils (hereinafter referred to as fats) that do not contain impurities such as soaps or other alkaline compounds which may react to produce high results.

It is only applicable to fats with low water content.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 661:1989, *Animal and vegetable fats and oils — Preparation of test sample.*

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

3 Definition

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

3.1 water content: Mass of water, as determined in accordance with the method specified in this International Standard, divided by the mass of the test portion.

It is expressed as a percentage by mass.

4 Principle

Addition of a test portion to a prepared mixture of solvent and reagent. Titration with Karl Fischer reagent and calculation of the water content.

5 Reagents

Use only reagents of recognized analytical grade, and water complying with grade 2 in accordance with ISO 3696.

5.1 Anhydrous solvent: 2-methoxyethanol, propan-1-ol or a specific solvent commercially prepared.

5.2 Karl Fischer reagent, preferably commercially prepared.

A non-pyridine reagent may be used if it has been shown to be suitable.[1]

Determine the water equivalent of the reagent on each day on which it is used. See 9.4.

NOTE 1 The preparation of Karl Fischer reagent containing pyridine is described in annex A.

5.3 Disodium tartrate dihydrate, ($Na_2C_4H_4O_6{\cdot}2H_2O$), crystalline.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Karl Fischer apparatus, fully automatic or semi-automatic, using an electrometric detection or visual determination of the endpoint. It is essential to use an airtight vessel which will prevent reaction with air humidity, and also to stir the contents of the reaction vessel efficiently.

7 Sampling

It is important the laboratory receive a sample which 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.

8 Preparation of test sample

Prepare the test sample in accordance with ISO 661.

9 Procedure

9.1 Preparation of the reaction vessel

Add solvent (5.1) to the reaction vessel of the Karl Fischer apparatus (6.1) until the electrode is immersed. Whilst stirring, add the Karl Fischer reagent (5.2) until the endpoint is reached, as indicated by the instrument reading or by a change in colour. Disregard the amount of reagent used.

9.2 Test portion

Weigh between 5 g and 20 g of the test sample to the nearest 0,01 g.

9.3 Determination

Add the test portion (9.2) to the reaction vessel and stir for 30 s to disperse it. Titrate with the Karl Fischer reagent to the same endpoint (9.1).

Record the amount of reagent used.

Carry out a duplicate determination by adding a second test portion and repeating the titration.

NOTE 2 If the test portion does not dissolve, another of the solvents (5.1) should be used, with the addition of chloroform if necessary.

9.4 Determination of the water equivalent of the reagent

The water equivalent of the reagent shall be determined or checked by following the procedure specified in 9.2 and 9.3 using disodium tartrate dihydrate (5.3) or a known mass of water.

10 Expression of results

The water content is given by the equation

$$w = \frac{V \times \rho}{m} \times 100\ \%$$

where

w is the water content, expressed as a percentage by mass;

V is the volume, in millilitres, of Karl Fischer reagent used;

ρ is the water equivalent of the Karl Fischer reagent, in grams per millilitre;

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

Take as the result the arithmetic mean of the two determinations provided that the requirements for repeatability (11.1) are met.

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[1] A suitable non-pyridine reagent is "Hydranal-composite 2", containing iodine, sulfur dioxide and an amine dissolved in the solvent. Hydranal-composite 2 is supplied by Riedel-de Haën AG, Seelze/Hannover, 3016 Germany. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

11 Precision

Details of an interlaboratory test on the precision of the method are summarized in annex C. The values derived from this interlaboratory test may not be applicable to concentration ranges and matrices other than those given.

For the values obtained for repeatability and reproducibility, a probability level of 95 % holds.

11.1 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, should not be greater than:

> 25 % of the mean of the two results for water contents not exceeding 0,05 % (m/m);
>
> 12 % of the mean of the two results for water contents exceeding 0,05 % (m/m).

11.2 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, should not be greater than:

> 30 % of the mean of the two results for water contents not exceeding 0,2 % (m/m);
>
> 20 % of the mean of the two results for water contents exceeding 0,2 % (m/m).

12 Test report

The test report shall specify:

— the method in accordance with which sampling was carried out, if known;

— the method used;

— the test result(s) obtained; and

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

It shall also mention all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s).

The test report shall include all information necessary for the complete identification of the sample.

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Annex A
(informative)

Preparation of Karl Fischer reagent containing pyridine

Place 670 ml of methanol, containing not more than 500 mg/kg of water, or 2-methoxyethanol, containing not more than 500 mg/kg of water, in a dry 1 litre brown-glass flask, fitted with a ground glass stopper. As the reaction is exothermic, maintain the flask at about 0 °C, for example by immersing it in an ice bath or in crushed solid carbon dioxide.

Add about 85 g of iodine. Stopper the flask and shake it occasionally until the iodine is completely dissolved. Then add approximately 270 ml of pyridine containing not more than 500 mg/kg of water. Stopper the flask again and mix thoroughly. Using the method described below, dissolve 65 g of sulfur dioxide in this solution, ensuring that the temperature of the liquid does not exceed 20 °C.

Replace the ground glass stopper by an attachment for introducing sulfur dioxide (consisting of a cork bearing a thermometer and glass inlet tube 6 mm × 8 mm, reaching to within 10 mm of the bottom of the flask) and a small capillary tube for connecting to the atmosphere.

Place the whole assembly with the cooling bath on a balance and weigh to the nearest 1 g. Connect the inlet attachment to a cylinder of sulfur dioxide by means of a flexible connection and a drying tube filled with desiccant and gently open the tap on the cylinder.

Adjust the rate of flow of sulfur dioxide so that all the gas is absorbed without the liquid showing any sign of rising up the inlet tube. Ensure that the temperature of the liquid does not rise above 20 °C. Close the tap on the cylinder as soon as the increase in mass reaches 65 g.

Immediately remove the flexible connection and reweigh the flask and its inlet attachment. The mass of dissolved sulfur dioxide should be between 60 g and 70 g. A slight excess is not harmful.

Stopper the flask, mix the solution and leave for at least 24 h before using it because the water equivalent of the reagent decreases rapidly to begin with and then much more slowly.

The water equivalent of the reagent thus prepared should be between 3,5 mg/ml and 4,5 mg/ml. It should be determined daily if methanol has been used in its preparation, but may be determined less frequently if 2-methoxyethanol has been used.

It is possible to prepare Karl Fischer reagent having a lower water content by diluting the solution prepared as described above with the solvent (5.1).

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Annex B
(informative)

Information on the use of the coulometric method

In the coulometric variation of the Karl Fischer determination of water, iodine necessary for the reaction with water is produced by the anodic oxidation of iodine. The iodine produced is proportional to the quantity of electricity consumed, the unit of which is the coulomb.

The commercial instruments which have been developed to make use of this principle are sophisticated, usually being fully automated and computerized. Such an instrument contains two cells, anodic and cathodic, separated by a membrane and containing electrolytes into which platinum electrodes dip. The reaction takes place in the anodic cell. The rest of the instrument facilitates the reaction, the measurement of the number of coulombs of electricity consumed and its conversion into the water content.

The coulometric method is more sensitive than the volumetric method and permits the determination of lower water contents. It is also sensitive to atmospheric moisture and chemical side reactions. It cannot be relied on for arbitration or referee purposes. Clause 11 is not applicable to results obtained using the coulometric method.

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Annex C
(informative)

Results of interlaboratory test

The precision of the method has been established by an international interlaboratory test carried out in 1983 by AOAC, in accordance with ISO 5725. In this test 15 laboratories participated and samples with different water contents were investigated.

The results are shown in table C.1.

NOTE 3 These results were obtained using a reagent containing pyridine. No results for interlaboratory tests using non-pyridine reagents are yet available.

Table C.1

Sample	**I**	**II**	**III**	**IV**
Number of laboratories retained after eliminating outliers	12	14	13	15
Mean water content, % (m/m)	0,029	0,114	0,220	3,065
Coefficient of variation of repeatability, %	24,1	14,0	12,7	10,4
Repeatability, 2,83 s_r	0,007	0,016	0,028	0,319
Coefficient of variation of reproducibility, %	31,0	28,9	18,1	20,3
Reproducibility, 2,83 s_R	0,009	0,033	0,040	0,623

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Annex D
(informative)

Bibliography

[1] ISO 5555:1991, *Animal and vegetable fats and oils — Sampling.*

[2] ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests* (now withdrawn), was used to obtain the precision data.

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