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Milled cereal products — Determination of fat acidity

Produits de mouture des céréales — Détermination de l'acidité grasse

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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 of the voluntary nature of standards, 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 4, *Cereals and pulses*.

This third edition cancels and replaces the second edition (ISO 7305:1998), which has been technically revised. The main changes compared with the previous edition are as follows:

— the indicator has been modified and the procedure relating to laboratory practices has been revised;

— the expression of results has been changed to milligrams of sodium hydroxide per 100 g of dry matter for more precision.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document describes a method of estimating the quantity of long-chain, non-esterified fatty acids which are liberated by the action of lipase during the storage of milled cereal products. It therefore provides a sensitive and significant test to characterize the state of conservation and the utilization values of these products.

The solvent used for the extraction, 95 % to 96 % ethanol, breaks all the low-energy links where fatty acids are involved, and solubilizes the latter rapidly and quantitatively, with the exclusion of the major part of amino acids and mineral salts.

Observation of the colour change at the end point of the titration is facilitated by the absence of turbidity in the solution.

Milled cereal products — Determination of fat acidity

1 Scope

This document specifies a method for the determination of the fat acidity of milled cereal products. It is applicable to flours and semolinas obtained from wheat and durum wheat, and to pasta.

NOTE This document appears to be applicable also to grains, flours and semolinas obtained from maize, and rye flour and oat flakes, but a further interlaboratory test is necessary before confirming this extension of the field of application.

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 712, *Cereals and cereal products — Determination of moisture content — Reference method*

ISO 12099, *Animal feeding stuffs, cereals and milled cereal products — Guidelines for the application of near infrared spectrometry*

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:

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

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

3.1
fat acidity
quantity of acids, essentially non-esterified fatty acids, extracted according to the procedure described in this document

Note 1 to entry: Fat acidity is expressed in milligrams of sodium hydroxide per 100 g of dry matter.

Note 2 to entry: A conversion of the result obtained by the calculation in Clause 11 can be done to express the result in grams of sulphuric acid.

4 Principle

Dissolution of the acids in ethanol at room temperature, centrifuging and titration of an aliquot portion of the supernatant liquid against sodium hydroxide.

5 Reagents

Use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity.

5.1 Ethanol, 95 % to 96 % (volume fraction).

5.2 Sodium hydroxide standard volumetric solution, c (NaOH) = 0,05 mol/l in ethanol (5.1) (volume fraction), free of carbonates.

The exact concentration shall be known and checked immediately prior to each series of determinations of fat acidity.

Use a solution prepared at least five days in advance and stored in a brown glass bottle, fitted with a rubber stopper. The solution shall be colourless or straw coloured.

5.3 Thymolphthalein, indicator solution, 1 g per 100 ml in ethanol (5.1) (volume fraction), changing from colourless to blue.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Sieves, of wire gauze, of nominal aperture size 160 µm and 500 µm.

6.2 Centrifuge tubes, of capacity 45 to 50 ml, hermetically stoppered.

6.3 Centrifuge, capable of a centrifugal acceleration of 2 000g.

6.4 Pipettes or automatic distributors, of capacities 20 ml and 30 ml.

6.5 Conical flask, of capacity 250 ml.

6.6 Microburette, graduated in 0,01 ml divisions.

6.7 Rotary stirrer, capable of 30 r/min to 60 r/min.

6.8 Analytical balance, capable of weighing to an accuracy of ±0,01 g.

6.9 Grinder, capable of grinding semolina and pasta without any appreciable heating.

7 Sampling

Sampling is not part of the method specified in this document.

8 Preparation of test sample

8.1 General

Acidity increases during storage, therefore the samples shall be stored in sealed bottles at about 4 °C. Allow the sample to return to laboratory temperature in the sealed bottle before taking test portions.

8.2 Products not requiring grinding

Products in which the particles completely pass a sieve of aperture size 500 µm (6.1) and for which at least 80 % passes a sieve of aperture size 160 µm (6.1) do not need to be ground before the determination. Mix the sample well before taking the test portion (10.1).

8.3 Products requiring grinding

Products not meeting the particle size characteristics mentioned above require grinding.

Grind about 50 g of sample using the grinder (6.9) until the particle size characteristics specified in 8.2 are achieved. Mix well before taking the test portion (10.1).

9 Determination of moisture content of the test sample

Determine the moisture content of the test sample in accordance with the method specified in ISO 712 or using an apparatus utilizing near infrared spectroscopy for which the performances have been demonstrated in accordance with ISO 12099.

10 Procedure

10.1 Number of determinations

Carry out two single determinations using the samples prepared in accordance with Clause 8.

If the absolute difference between two results is greater than that the repeatability limit defined in 12.2, repeat the determination until the result meets this requirement.

10.2 Test portion

Weigh, to the nearest 0,01 g, approximately 5 g of the test sample (see Clause 8) and place it in a centrifuge tube (6.2).

10.3 Determination

Using a pipette (6.4), add 30 ml of the ethanol (5.1) to the centrifuge tube (6.2). Seal the tube hermetically and place it on the rotary stirrer (6.7). Keep stirring for 1 h at room temperature. Then centrifuge (6.3) for 5 min.

By means of a pipette (6.4), transfer 20 ml of the supernatant liquid to an Erlenmeyer flask (6.5). Add five drops of Thymolphthalein (5.3).

Titrate the solution, using the microburette (6.6), with the sodium hydroxide solution (5.2) until a blue colour appears.

10.4 Blank test

Carry out a blank test in parallel with the determination, beginning at 10.3 and replacing the 20 ml of supernatant liquid by 20 ml of ethanol (5.1).

11 Expression of results

The fat acidity, A_{Na}, expressed in milligrams of sodium hydroxide per 100 g of dry matter, is obtained by Formula (1):

$$A_{\mathrm{Na}} = \frac{6\,000(V_1 - V_0)c}{m} \times \frac{100}{100 - w} \qquad (1)$$