International Standard

ISO 17715

Flour from wheat (*Triticum aestivum* L.) — Amperometric method for starch damage measurement

*Farine de blé tendre (*Triticum aestivum *L.) — Méthode ampérométrique pour le mesurage de l'endommagement de l'amidon*

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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 document 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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This document was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 4, *Cereals and pulses*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 338, *Cereal and cereal products*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 17715:2013), which has been technically revised.

The main changes are as follows:

— possibility to use a ready-to-use solution of sodium thiosulfate has been added.

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

Damaged starch content is an important parameter in flour quality as it directly influences the flour water absorption capacity and therefore its use in the agri-food industry.

In the past, a number of methods based on various principles were developed to estimate such content, but comparing the results is difficult due to the different principles and units of measurement used.

A laboratory device is dedicated to the determination of damaged starch content using an amperometric method and which offers a choice of units of measurement according to individual references.

Flour from wheat (*Triticum aestivum* L.) — Amperometric method for starch damage measurement

1 Scope

This document specifies an amperometric method to determine the content of damaged starch in flour.

It is applicable to all flour samples from the industrial or laboratory milling of wheat (*Triticum aestivum* L.).

NOTE 1 Wheat can be milled in the laboratory in accordance with the methods described in ISO 27971[9] or in the BIPEA guidance document BY.102.D[10].

NOTE 2 In the absence of validity studies, the results on semi-wholemeal or wholemeal flour, although able to meet the conditions of repeatability given in Clause 9, require careful interpretation.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

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

3.1
damaged starch
starch granules present in wheat flour mechanically damaged during milling, leading to a greater capacity to absorb water and increasing susceptibility to amylolytic enzymes

Note 1 to entry: A damaged starch content that is too high has a negative effect on quality of flours.

4 Principle

Determination of damaged starch content of a flour sample by measurement of iodine absorption kinetics in an aqueous medium using an amperometric electrode.

The amperometric method is based on the existing proportionality between iodine absorption capacity and starch damage content.

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

5.1 **Water**, osmosed or demineralized or at least equivalent grade.

5.2 **Boric acid** or **citric acid**, powdered, for testing.

WARNING — The use of boric acid involves hazardous operations. This document does not purport to address all the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices.

5.3 Potassium iodide, powdered, for testing.

5.4 Sodium thiosulfate, a ready-to-use solution in water at 0,1 mol/l or prepared from a light bulb containing 0,1 mol sodium thiosulfate, to be diluted with water (5.1) in a 1 l flask. Powdered sodium thiosulfate can also be used where the concentration of the final solution is 0,1 mol/l. Protect the solution from light and use within three months.

6 Equipment

Usual laboratory apparatus and, in particular, the following shall be used.

6.1 CHOPIN SDmatic®,[1)] equipped with a reaction vessel and sample holder.

NOTE This document has been developed using the CHOPIN SDmatic®[1)]. It does not apply to the SD4 CHOPIN and Rapid FT devices which also measure damaged starch content, but using different technology.

6.2 Laboratory scales, with a display resolution of 10^{-2} g enabling weighing at 10^{-1} g accuracy.

6.3 Laboratory scales, with a display resolution of 10^{-4} g enabling weighing at 10^{-3} g accuracy.

6.4 Piston distributor, delivering 120 ml distilled water to the nearest 0,5 ml.

6.5 One-mark volumetric flask, capacity 1 000 ml, (see ISO 1042), class A.

7 Sampling

Sampling is not part of the method specified in this document. A recommended sampling method is given in ISO 24333[8].

It is important the laboratory receive a truly representative sample which has not been damaged or changed during transport or storage.

8 Procedure

8.1 Reagent weighing and dissolution

Weigh (6.2), to the nearest 0,5 g, 3,0 g boric acid (5.2) or 1,5 g citric acid (5.2) and 3,0 g potassium iodide (5.3) and add to a clean and dry reaction vessel (6.1). Add one drop (about 0,04 ml) of sodium thiosulfate solution (5.4) and dispense (6.4) 120 ml distilled water (5.1) into the vessel.

As the test begins with a heating and stirring phase, it is not necessary to obtain full dissolution of the reagents at this stage. In order to minimize losses during transfer, add powdered reagents directly to the reaction vessel.

1) CHOPIN SDmatic® is the trade name of a product supplied by CHOPIN Technologies. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

8.2 Sample weighing

Weigh (6.3), to the nearest 10^{-3} g, 1 000 g ± 0,100 g of the test sample of flour and place it in the pre-cleaned sample holder (6.1).

8.3 Test

Place the reaction vessel in the well on the device.

Lower the head of the device, and insert the sample holder containing the flour (see 8.2) into the compartment.

Start the test. Indicate the exact mass of the test portion of flour weighed to the nearest 10^{-3} g. It is also possible to indicate the water and protein content of the sample if a result corrected on this basis is to be obtained, otherwise the default values should be left (mass fractions of 14 % and 12 %, respectively) for the two components. Confirm the start of the test.

The test lasts 6 min to 7 min. Ensure that all of the flour descends into the reaction vessel when the vibrator starts up. Use the tip of a brush or lightly blow to enable any remaining flour to fall.

Wait for the beep at the end of the test at which point the result is displayed.

8.4 Cleaning

Raise the head of the device and remove the reaction vessel. Rinse, then carefully and gently wipe the measuring electrode, the heating element and the stirrer.

Remove any residue from the vessel (do not dispose in the sink). Carefully wash and wipe dry the reaction vessel, which shall be free of all traces of reagent, sample or moisture, and which shall be left ready for use in a later test.

8.5 Number of determinations

Perform two determinations on the same test sample.

9 Expression of results

The result is expressed as $A_{I\,\%}$ (iodine absorption percentage) converted into UCD (Chopin–Dubois units). Equations provided by the manufacturer can be used to calculate the equivalence in another unit.

The arithmetic mean of the two determinations (see 8.5) shall be taken as the result if they meet the conditions of repeatability specified in 10.2 or in Table A.5. Otherwise, perform two new determinations.

Express the $A_{I\,\%}$ result, in percentage, to the nearest two decimal places.

Express the *UCD* result, in *UCD*, to the nearest one decimal place.

NOTE It can be useful to calculate starch damage on a constant water and protein content basis. In this case, flour moisture content and protein content can be determined in accordance with ISO 712-1[1] for moisture and ISO 20483[7] or ISO 16634-2[6] for protein.

10 Precision

10.1 Interlaboratory tests

Two interlaboratory tests established the repeatability and reproducibility limits of the method. The statistical results of the study are given in Annex A.

The values of each of the studies apply to the concentration ranges and flours from wheat (*Triticum aestivum* L.*).*

10.2 Repeatability limits, *r*

Repeatability limit is the value below which the absolute value, of the difference between two test results obtained in conditions of repeatability is located, with a probability of 95 %.

The repeatability limits, *r*, are obtained from Formulae (1) and (2). Some repeatability limit values are listed in Table A.5.

For $A_{\mathrm{I}\,\%}$:

$$r = \left(-0{,}007\,\mu_{A_{\mathrm{I}}\,\%} + 0{,}787\,1\right) \times 2{,}8 \tag{1}$$

where $\mu_{A_{\mathrm{I}}\,\%}$ is the mean iodine absorption capacity.

For UCD:

$$r = (-0{,}007\mu_{\mathrm{UCD}} + 0{,}473\,9) \times 2{,}8 \tag{2}$$

where μ_{UCD} is the mean Chopin–Dubois unit value.

10.3 Reproducibility limits, *R*

Reproducibility limit is the value below which the absolute value, of the difference between two test results obtained in conditions of reproducibility is located, with a probability of 95 %.

The reproducibility limits, *R*, are obtained from Formulae (3) and (4). Some reproducibility limit values are listed in Table A.6.

For $A_{\mathrm{I}\,\%}$:

$$R = \left(-0{,}03\,\mu_{A_{\mathrm{I}}\,\%} + 3{,}074\,5\right) \times 2{,}8 \tag{3}$$

For UCD:

$$R = (-0{,}041\mu_{\mathrm{UCD}} + 1{,}522\,2) \times 2{,}8 \tag{4}$$

10.4 Critical difference, d_{c}

10.4.1 General

The critical difference is the deviation between two average values obtained from two test results under repeatability conditions.

10.4.2 Comparison of two measurement groups in the same laboratory

The critical difference for comparing two average values obtained from two test results in the same laboratory under repeatability conditions, $d_{\mathrm{c},r}$, is given by Formula (5):

$$d_{\mathrm{c},r} = 2{,}8 s_r \sqrt{\frac{1}{2n_1} + \frac{1}{2n_2}} = 2{,}8 s_r \sqrt{\frac{1}{2}} = 1{,}98 s_r \tag{5}$$