International Standard 7305

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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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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Milled cereal products — Determination of fat acidity

1 Scope and field of application

This International Standard specifies a method for the determination of "fat" acidity of the following milled cereal products: flours and semolinas obtained from wheat and durum wheat. It is also applicable to pasta.

NOTE — This method appears to be applicable also to flours and semolinas obtained from maize, but a further interlaboratory test would be necessary to confirm this extension of the field of application.

2 References

ISO 712, *Cereals and cereal products — Determination of moisture content (Routine reference method).*

ISO 2170, *Cereals and pulses — Sampling of milled products.*

3 Definition

fat acidity: A conventional term for acids, essentially free fatty acids, extracted under the conditions described in this International Standard.

It is expressed in grams of sulfuric acid per 100 g of dry matter.

4 Principle

Dissolution of the acids in 95 % (V/V) ethanol at room temperature, followed by centrifuging and titration of an aliquot portion of the supernatant liquid against sodium hydroxide.

5 Reagents

All the reagents used shall be of recognized analytical grade and the water used shall be distilled water or water of equivalent purity.

5.1 Ethanol, 95 % (V/V).

5.2 Sodium hydroxide, standard volumetric solution, $c(NaOH) = 0{,}05$ mol/l, in 95 % (V/V) ethanol free from 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 5 days in advance and stored in a brown glass bottle, fitted with a rubber stopper. The solution shall be colourless or straw coloured.

NOTE — It is recommended that the ethanol be purified as follows. Dissolve 5 to 10 g of sodium hydroxide in 1 litre of ethanol and add 0,5 g of aluminium turnings, boil the mixture under reflux for 1 h, then distil the ethanol. Dissolve the required quantity of sodium hydroxide (i.e. to give a concentration of 2 g/l) in the distillate, leave to stand for 5 days and use the supernatant solution.

5.3 Phenolphthalein, indicator solution, 1 g per 100 ml 95 % (V/V) ethanol.

6 Apparatus

6.1 Sieves, of wire gauze, of nominal aperture sizes 1 mm (for flour, if necessary), and 160 and 500 µm (for semolina and pasta).

6.2 Centrifuge tubes, of borosilicate or neutral glass, of capacity 45 ml, hermetically stoppered.

6.3 Centrifuge, capable of a centrifugal acceleration of 2 000g (generally 5 000 to 6 000 r/min).

6.4 Pipettes, of capacities 10 and 20 ml, class A.

6.5 Conical flask, of capacity 250 ml.

6.6 Micro-burette, graduated in divisions of 0,01 ml.

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

6.8 Balance, accurate to 0,01 g.

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

6.10 Orange filter, photographic-type cellulose acetate filter, blue absorbing.

7 Sampling and storage conditions

Proceed in accordance with ISO 2170.

Acidity gradually increases at normal temperatures; therefore the samples shall not be kept at laboratory temperature for more than 1 day. If necessary, store them in sealed bottles at about 4 °C.

If the sample has been stored at 4 °C, allow it to return to laboratory temperature in the sealed bottle before taking test portions.

8 Procedure

8.1 Preparation of the test sample

8.1.1 Flour

In the case of flour having particle size characteristics satisfying the requirements of 8.1.2, take about 50 g of the flour and sift it, if necessary, using a sieve of aperture size 1 mm (6.1) so as to break up any lumps present. Mix well before taking the test portion.

For other flours, proceed as in 8.1.2.

8.1.2 Semolina and pasta

Grind about 50 g of the semolina or pasta in the grinder (6.9) so that all of it passes a sieve of aperture size 500 µm (6.1) and at least 80 % (m/m) passes a sieve of aperture size 160 µm (6.1). Mix well before taking the test portion.

8.2 Moisture content of the test sample

Determine the water content of the test sample in accordance with ISO 712.

NOTE — This operation is not necessary if the result is to be expressed in relation to the product as received.

8.3 Test portion

Weigh, to the nearest 0,01 g, approximately 5 g of the test sample (8.1) and place it in a centrifuge tube (6.2).

8.4 Determination

8.4.1 Pipette 30 ml of 95 % (V/V) ethanol (5.1) into the centrifuge tube. Seal the tube hermetically and agitate for 1 h using the rotary stirrer (6.7) working at a temperature of 20 ± 5 °C. Then remove the stopper and centrifuge for 5 min with an acceleration of 2 000g.

8.4.2 Transfer, by means of a pipette (6.4), 20 ml of the supernatant liquid to a conical flask (6.5). Add 5 drops of phenolphthalein (5.3).

Titrate, using a micro-burette (6.6), against the sodium hydroxide solution (5.2) until a pale pink colour lasting several seconds appears, using an orange filter (6.10), **placed to the operator's eye,** to enhance the colour change of the indicator.

8.5 Blank test

Carry out a blank test in parallel with the determination, beginning at 8.4.2 and replacing the 20 ml of supernatant liquid by 20 ml of ethanol (5.1).

8.6 Number of determinations

Carry out two determinations on test portions taken from the same test sample.

9 Expression of results

The fat acidity, expressed in grams of sulfuric acid per 100 g of dry matter, is equal to

$$\frac{7{,}35(V_1 - V_0)c}{m} \times \frac{100}{100 - w}$$

where

c is the exact concentration, expressed in moles per litre, of the standard volumetric sodium hydroxide solution used;

m is the mass, in grams, of the test portion (8.3);

V_1 is the volume, in millilitres, of the sodium hydroxide solution used in the determination (8.4);

V_0 is the volume, in millilitres, of the sodium hydroxide solution used in the blank test (8.5);

w is the moisture content, expressed as a percentage by mass, of the test sample (8.2).

Express the result to three decimal places.

NOTES

1 If it is desired to express the acidity as milligrams of potassium hydroxide per 100 g of dry matter, use the following formula:

$$\frac{8416(V_1 - V_0)c}{m} \times \frac{100}{100 - w}$$

2 If it is desired to express the acidity in relation to the product as received, omit the factor $100/(100 - w)$ in the formula.

10 Precision

Two interlaboratory tests organized at the international level with, respectively, 24 laboratories (test No. 1) and 21 laboratories (test No. 2) participating, each laboratory performing two determinations, gave the statistical results [assessed in accordance with ISO 5725[1)]] set out in the table.

11 Test report

The test report shall show the method used, the date of the test and the results obtained. It shall also mention any operating details not specified in this International Standard, or regarded as optional, together with details of any incidents likely to have influenced the results.

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

Table

Values expressed in grams of sulfuric acid per 100 g of dry matter

Sample	Wheat semolina Test No. 1	Wheat flour A Test No. 1	Wheat flour Test No. 2	Wheat flour B Test No. 1	Durum wheat semolina Test No. 2
Number of laboratories retained after eliminating outliers	19	20	21	20	21
Mean	0,015	0,026	0,039	0,064	0,040
Repeatability standard deviation, s_r Coefficient of variation of repeatability Repeatability, 2,83 s_r	0,000 7 4,7 % 0,002	0,001 3 5,0 % 0,003	0,001 0 2,6 % 0,003	0,001 3 2,0 % 0,003	0,001 5 3,7 % 0,004
Reproducibility standard deviation, s_R Coefficient of variation of reproducibility Reproducibility, 2,83 s_R	0,004 2 28 % 0,012	0,003 7 14 % 0,010	0,005 9 15 % 0,017	0,006 4 10 % 0,018	0,005 8 14 % 0,016

1) ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests.*