INTERNATIONAL STANDARD

ISO 11214

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Modified starch — Determination of carboxyl group content of oxidized starch

Amidon modifié — Dosage des groupes carboxyles dans l'amidon oxydé

Reference number
ISO 11214:1996(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 11214 was prepared by Technical Committee ISO/TC 93, *Starch (including derivatives and by-products)*.

Annexes A and B of this International Standard are for information only.

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Modified starch — Determination of carboxyl group content of oxidized starch

1 Scope

This International Standard specifies a method for the determination of the carboxyl group content of oxidized starch.

The method is suitable for determining carboxyl group contents up to 1 % (*m*/*m*).

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 indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1666:—[1], *Starch — Determination of moisture content — Oven-drying method.*

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

ISO 3946:1982, *Starches and derived products — Determination of total phosphorus content — Spectrophotometric method.*

3 Principle

The carboxyl groups are converted into the acid form by adding a mineral acid to a homogenized test portion of the oxidized starch.

The cations and the excess acid are eliminated by washing with water.

The washed sample is gelatinized and titrated with sodium hydroxide standard solution.

For oxidized potato starch, the result is corrected for the phosphate group content.

4 Reagents and materials

Use only reagents of recognized analytical grade, unless otherwise specified, and water complying with grade 2 in accordance with ISO 3696. The water used shall be free of carbon dioxide.

4.1 Hydrochloric acid, 0,1 mol/l solution.

4.2 Sodium hydroxide, standard volumetric solution, c(NaOH) = 0,1 mol/l, free of carbon dioxide.

4.3 Phenolphthalein in ethanol, 1 g/l solution, 90 % (*V*/*V*).

4.4 Silver nitrate, 10 g/l solution.

5 Apparatus

Usual laboratory apparatus and in particular the following.

5.1 Beakers, of capacity 100 ml and 600 ml.

5.2 Magnetic stirrer.

1) To be published. (Revision of ISO 1666:1973)

5.3 Vacuum filter, equipped with a fritted glass crucible of medium porosity, or a Büchner funnel, of diameter 55 mm, equipped with a small, round filter paper (medium filtration rate).

5.4 Boiling water bath.

5.5 Mechanical stirrer.

5.6 Blade mill.

5.7 Test sieve, of aperture size 800 µm.

6 Preparation of the test sample

Pass the sample through the test sieve (5.7). If the sample does not pass through the sieve, grind it using the blade mill (5.6) so that it completely passes through the sieve. Homogenize the sample.

NOTE 1 In the case of oxidized maize or wheat starch, it may be desirable to defat the sample by Soxhlet extraction with a mixture of propanol and water [3 + 1 (*V*/*V*)] in order to correct for the contribution of lipids to the carboxyl group content.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,1 mg, 5 g of the test sample into a 100 ml beaker (5.1).

7.2 Conversion of the carboxyl salts

Add 25 ml of the hydrochloric acid solution (4.1) to the beaker and stir for 30 min on the magnetic stirrer (5.2).

7.3 Washing

Filter the suspension under vacuum through a fritted glass crucible or Büchner funnel (see 5.3). Wash the cake obtained with water until the filtrate contains no chloride ions. Test for the absence of chloride ions by adding 1 ml of the silver nitrate solution (4.4) to 5 ml of filtrate. Turbidity or precipitation occurs within 1 min if chloride is present. Washing the cake will take approximately 300 ml of water.

7.4 Gelatinization

Quantitatively transfer the cake obtained into a 600 ml beaker (5.1) with 100 ml of water. Add 200 ml of water, place the beaker in the boiling water bath (5.4), stir continuously with the mechanical stirrer (5.5) until the starch gelatinizes and continue stirring for another 15 min.

NOTES

2 Direct heating with a hotplate or Bunsen burner may overheat or scorch the material which will lead to higher results.

3 A complete gelatinization makes rapid titration easier and increases the sensitivity of the detection of the end-point.

7.5 Titration

Remove the beaker from the boiling bath and titrate while still hot with the sodium hydroxide solution (4.2), using the phenolphthalein solution (4.3) as indicator until a persistent pink colour is obtained.

NOTE 4 The end-point (at pH = 8,3) may be determined electrometrically.

7.6 Determination of moisture content

Determine the moisture content of the test sample in accordance with ISO 1666.

7.7 Determination of phosphorus content

For oxidized potato starch, determine the phosphorus content, w_P, in percentage by mass, of the test sample in accordance with ISO 3946.

8 Expression of results

8.1 Calculation

Calculate the carboxyl group content, based on the dry substance, using the equation

$$w_C = \frac{cVM_C \times 100}{m} \times \frac{100}{100 - w_m}$$

where

w_C is the total carboxyl group content, in percentage by mass, based on the dry substance;

c is the concentration, in moles per litre, of sodium hydroxide solution used for the titration;

V is the volume, in millilitres, of sodium hydroxide solution used for the titration;

M_C is the relative molecular mass of the carboxyl function ($M_C = 0{,}045$);

m is the mass, in grams, of the test portion (7.1);

w_m is the moisture content, in percentage by mass, of the test sample.

NOTE 5 The lipid content of maize and wheat starch contributes to the value obtained for the carboxyl group content by approximately 0,1 % (m/m). In the case of oxidized maize or wheat starch, the total carboxyl group content may be corrected for, if desired, either by deducting 0,1 % (m/m) from w_C or by determining w_C of a test sample which has been defatted by Soxhlet extraction with a mixture of propanol and water [3 + 1 (V/V)] (see also note 1).

8.2 Correction for phosphate groups

The phosphate groups attached to oxidized potato starch contribute to the value obtained for the carboxyl group content. For oxidized potato starch, correct the total carboxyl group content resulting from 8.1 by deducting $3w_P$ from w_C.

NOTE 6 The correction quantity, $3w_P$, is derived from the following calculation:

$$3w_P \approx n \times \frac{M_C}{M_P} \times w_P$$

where

w_P is the phosphorus content, in percentage by mass, of the test sample, obtained in 7.7;

n is the number of free acid groups left on phosphorus ($n = 2$);

M_C is the relative molecular mass of the carboxyl function ($M_C = 0{,}045$);

M_P is the relative molecular mass of phosphorus ($M_P = 0{,}031$).

9 Precision

The precision of the method was established by an interlaboratory test carried out in accordance with ISO 5725-2 (reference [2] in annex B). See annex A for a summary of the statistical results of this test.

The probability level is 95 % when the repeatability and reproducibility limits are obtained.

9.1 Repeatability

The absolute difference between two independent 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 6 % of the higher of the two results.

9.2 Reproducibility

The absolute difference between two independent 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 16 % of the higher of the two results.

10 Test report

The test report shall specify

a) a reference to this International Standard;

b) the method used;

c) the result(s) obtained;

d) 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.

Annex A
(informative)

Table A.1 — Statistical results of the interlaboratory test

Parameter	Sample[1)]					
	M1	M2	M3	P1	P2	P3
Number of laboratories retained after eliminating outliers	6	6	6	6	6	6
Number of outliers (laboratories)	-	-	-	-	-	-
Number of accepted results	6	6	6	6	6	6
Mean carboxyl group content [% (*m/m*)]	0,32	0,66	0,66	0,29	0,68	0,68
Repeatability standard deviation, s_r [% (*m/m*)]	0,008 6	0,007 5	0,12	0,007	0,013	0,019
Repeatability relative standard deviation, %	2,67	1,14	1,89	2,64	1,97	2,87
Repeatability limit, $r = 2{,}8 \times s_r$ [% (*m/m*)]	0,024	0,021	0,035	0,020	0,038	0,054
Reproducibility standard deviation, s_R [% (*m/m*)]	0,023	0,033	0,031	0,022	0,028	0,034
Reproducibility relative standard deviation, %	7,04	4,95	4,75	7,58	4,07	5,05
Reproducibility limit, $R = 2{,}8 \times s_R$ [% (*m/m*)]	0,064	0,093	0,089	0,061	0,078	0,097

1) M: modified maize starch.

P: modified potato starch.

Annex B
(informative)

Bibliography

[1] ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

[2] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

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Descriptors: carbohydrates, starches, chemical analysis, determination of content, volumetric analysis.

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