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Cereals and pulses — Determination of the nitrogen content and calculation of the crude protein content — Kjeldahl method

Céréales et légumineuses — Détermination de la teneur en azote et calcul de la teneur en protéines brutes — Méthode de Kjeldahl

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Contents

Page

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. 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.

The committee responsible for this document is ISO/TC 34, *food and food products*, Subcommittee SC 4, *cereals and pulses*.

This second edition cancels and replaces the first edition (ISO 20483:2006), which has been technically revised.

Cereals and pulses — Determination of the nitrogen content and calculation of the crude protein content — Kjeldahl method

WARNING — The use of this International Standard can involve hazardous materials, operations and equipment. This International Standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for the determination of the nitrogen content of cereals, pulses and derived products, according to the Kjeldahl method, and a method for calculating the crude protein content.

The method does not distinguish between protein nitrogen and non-protein nitrogen. If it is important to determine the non-protein nitrogen content, an appropriate method would be applied.

NOTE In certain cases, full recovery of the nitrogen in nitrates and nitrites is not possible by this method.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable to its application. 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 6540, *Maize — Determination of moisture content (on milled grains and on whole grains)*

ISO 24557, *Pulses — Determination of moisture content — Air-oven method*

3 Terms and definitions

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

3.1
nitrogen content
quantity of nitrogen determined after application of the procedure described

Note 1 to entry: It is expressed as a mass fraction of dry product, as a percentage.

3.2
crude protein content
quantity of crude protein obtained from the nitrogen content as determined by applying the specified method, calculated by multiplying this content by an appropriate factor depending on the type of cereal or pulse

Note 1 to entry: It is expressed as a mass fraction of dry product, as a percentage.

4 Principle

A test portion is digested by sulfuric acid in the presence of a catalyst. The reaction products are made alkaline, then distilled. The liberated ammonia is collected in a boric acid solution, which is titrated with a sulfuric acid solution, in order to determine the nitrogen content and calculate the crude protein content.

5 Reagents

WARNING — The reagents described in 5.3, 5.8, 5.9 and 5.13 shall be handled with caution.

5.1 Use only nitrogen-free reagents of recognized analytical grade, except for the reference materials, and distilled or demineralized water or water of equivalent purity

5.2 Kjeldahl tablets, corresponding to the following composition: copper (II) sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) = 2,8 %, titanium oxide (TiO_2) = 2,8 % and potassium sulfate (K_2SO_4) = 94,3 %.

Alternatively, copper(II) sulfate pentahydrate, titanium oxide and potassium sulfate may also be mixed in the corresponding ratio.

5.3 Sulfuric acid, $c(H_2SO_4)$ = 18 mol/l, $\rho_{20}(H_2SO_4)$ = 1,84 g/ml.

5.4 Antifoaming agent: Paraffin oil, silicone or even antifoam tablets may be used to prevent foaming.

5.5 Acetanilide (C_8H_9NO) or tryptophan ($C_{11}H_{12}N_2O_2$), of minimum assay 99 % (mass fraction).

5.6 Boric acid, aqueous solution, $\rho_{20}(H_3BO_3)$ = 40 g/l, or any other concentration recommended for the apparatus being used.

5.7 Coloured indicator

Add volumes of Solution A (5.7.1) and Solution B (5.7.2) as recommended for the apparatus being used (for example: 5 volumes of Solution A and 1 volume of Solution B) or any other coloured indicator recommended for the apparatus.

NOTE 1 It is possible to use a ready-to-use solution of boric acid containing the coloured indicator (5.7.1 and 5.7.2).

NOTE 2 The ratio of Solutions A and B can be adjusted depending on the apparatus.

The titration may also be carried out potentiometrically by the use of a pH electrode, which shall be checked every day.

5.7.1 Solution A

Bromocresol green ($C_{21}H_{14}Br_4O_5S$): 200 mg.

Ethanol (C_2H_5OH), with a volume fraction of 95 %: quantity sufficient for 100 ml of solution.

5.7.2 Solution B

Methyl red ($C_{15}H_{15}N_3O_2$): 200 mg.

Ethanol (C_2H_5OH), with a volume fraction of 95 %: quantity sufficient for 100 ml of solution.

