SLOVENSKI STANDARD
SIST EN ISO 5983-2:2005

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Krma - Določanje dušika in izračun vsebnosti surovih beljakovin - 2. del: Metoda razklopa v bloku in destilacije z vodno paro (ISO 5983-2:2005)

Animal feeding stuffs - Determination of nitrogen content and calculation of crude protein content - Part 2: Block digestion/steam distillation method (ISO 5983-2:2005)

Futtermittel - Bestimmung des Stickstoffgehaltes und Berechnung des Rohproteingehaltes - Teil 2: Blockaufschluss/Dampfdestillationsverfahren (ISO 5983-2:2005)

Aliments des animaux - Détermination de la teneur en azote et calcul de la teneur en protéines brutes - Partie 2: Méthode de digestion en bloc et distillation a la vapeur (ISO 5983-2:2005)

Ta slovenski standard je istoveten z: **EN ISO 5983-2:2005**

ICS:

65.120	Krmila	Animal feeding stuffs

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EN ISO 5983-2

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English Version

Animal feeding stuffs - Determination of nitrogen content and calculation of crude protein content - Part 2: Block digestion/steam distillation method (ISO 5983-2:2005)

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This European Standard was approved by CEN on 1 June 2005.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (EN ISO 5983-2:2005) has been prepared by Technical Committee ISO/TC 34 "Agricultural food products" in collaboration with Technical Committee CEN/TC 327 "Animal feeding stuffs - Methods of sampling and analysis", the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by January 2006, and conflicting national standards shall be withdrawn at the latest by January 2006.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Endorsement notice

The text of ISO 5983-2:2005 has been approved by CEN as EN ISO 5983-2:2005 without any modifications.

INTERNATIONAL STANDARD

ISO 5983-2

First edition
2005-07-01

Animal feeding stuffs — Determination of nitrogen content and calculation of crude protein content —

Part 2: Block digestion/steam distillation method

Aliments des animaux — Détermination de la teneur en azote et calcul de la teneur en protéines brutes —
Partie 1: Méthode de digestion en bloc et distillation à la vapeur

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Contents

Page

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 5983-2 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 10, *Animal feeding stuffs*.

This first edition of ISO 5983-2, together with ISO 5983-1:2005, cancels and replaces ISO 5983:1997, which has been technically revised.

ISO 5983 consists of the following parts, under the general title *Animal feeding stuffs — Determination of nitrogen content and calculation of crude protein content*:

— *Part 1: Kjeldahl method*

— *Part 2: Block digestion/steam distillation*

Animal feeding stuffs — Determination of nitrogen content and calculation of crude protein content —

Part 2: Block digestion/steam distillation method

WARNING — The use of this method may involve the use of hazardous materials, operations and equipment. This standard does not purport to address all the safety risks associated with its use. It is the responsibility of the user of this method to establish appropriate health and safety practices and determine the applicability of local regulatory limitations prior to use.

1 Scope

This part of ISO 5983 specifies a method for the determination of nitrogen content of animal feeding stuffs according to the Kjeldahl method, and a method for the calculation of the crude protein content.

It concerns a semi-micro rapid routine method using block-digestion, copper catalyst and steam distillation into boric acid.

The method is applicable to the determination of greater than 0,5 % Kjeldahl nitrogen in animal feeding stuffs, pet foods and their raw materials.

The method does not measure oxidized forms of nitrogen nor heterocyclic nitrogen compounds.

The method does not distinguish between protein nitrogen and non-protein nitrogen.

NOTE If it is of importance to determine the content of non-protein-nitrogen, an appropriate method can be used.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referred document (including any amendments) applies.

ISO 385:2005, *Laboratory glassware — Burettes*

ISO 1871, *Agricultural food products — General directions for the determination of nitrogen by the Kjeldahl method*

ISO 6498:1998, *Animal feeding stuffs — Preparation of test samples*

3 Terms and definitions

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

3.1
nitrogen content
mass fraction of nitrogen determined by the procedure specified in this document

NOTE The nitrogen content is expressed as a percentage by mass or in grams per kilogram.

3.2
crude protein content
amount of nitrogen content (3.1) multiplied by the factor 6,25

NOTE The crude protein content is expressed as a percentage by mass or in grams per kilogram.

4 Principle

The test portion is digested using a block-digestion or equivalent apparatus. Concentrated sulfuric acid is used to convert protein nitrogen to ammonium sulfate at a boiling point elevated by the addition of potassium sulfate. A copper catalyst is used to enhance the reaction rate. An excess of sodium hydroxide is added to the cooled digest to liberate ammonia.

The liberated ammonia is distilled, using a manual, semi-automatic or fully automatic steam distillation unit. In the case of manual or semi-automatic steam distillation, distillation of the ammonia into an excess of boric acid solution is followed by titration with hydrochloric acid solution to a colorimetric endpoint. Where a fully automatic system is employed, automatic titration of the ammonia is carried out simultaneously with the distillation.

The nitrogen content is calculated from the amount of ammonia produced. The crude protein content is obtained by multiplying the result by the conventional conversion factor of 6,25.

NOTE 1 As in ISO 5983-1, the automatic titration of the ammonia can also be carried out with endpoint detection using a potentiometric pH system (see Annex B).

NOTE 2 In principle, sulfuric acid could also be used for the titration.

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified, and distilled or demineralized water or water of equivalent purity.

5.1 Kjeldahl catalyst tablets, comprising of 3,5 g of potassium sulfate and 0,4 g of copper(II) sulfate pentahydrate per tablet.

These tablets may be purchased ready prepared.

Other types of tablet may be used provided that

a) they contain a quantity of potassium sulfate such that 7 g of potassium sulfate and 0,8 g of copper(II) sulfate pentahydrate can be dispensed using an integer number of whole tablets, and

b) they do not contain salts of toxic metals such as selenium or mercury.

5.2 Sulfuric acid (H_2SO_4), with a mass fraction of at least 98 %, nitrogen-free (approximately ρ_{20} = 1,84 g/ml).

5.3 Hydrogen peroxide solution, containing approximately 30 g of H_2O_2 per 100 ml.

5.4 Antifoaming agent.

A silicone preparation is recommended, for example with a mass fraction of 30 % aqueous emulsion.

5.5 Sodium hydroxide (NaOH) solution, approximately 40 % (mass fraction), nitrogen-free (< 5 µg of N per gram).

5.6 Indicator solutions.

5.6.1 Methyl red solution.

Dissolve 100 mg of methyl red ($C_{15}H_{15}N_3O_2$) in 100 ml of ethanol or methanol.

5.6.2 Bromocresol green solution.

Dissolve 100 mg of bromocresol green ($C_{21}H_{14}Br_4O_5S$) in 100 ml of ethanol or methanol.

5.7 Concentrated boric acid solution, $c(H_3BO_3) = 40{,}0$ g/l.

Dissolve 400 g of boric acid in about 5 l to 6 l of hot deionized water. Mix and add more hot deionized water to a volume of about 9 l. Allow to cool to room temperature. Add 70 ml of the methyl red solution (5.6.1) and 100 ml of the bromocresol green solution (5.6.2) and mix. Dilute to a final volume of 10 l with water and mix well. Depending on the water used, the pH of the boric acid solution can differ from batch to batch. Often an adjustment with a small volume of alkali is necessary to obtain a positive blank (0,05 ml to 0,15 ml of titrant). The colour shall turn green when 100 ml of distilled water are added to 25 ml of the boric acid solution. If still red, titrate with 0,1 mol/l NaOH until "neutral grey" and calculate the amount of alkali needed for the 10 l batch.

Store the solution, which will be red in colour, at room temperature and protect the solution from light and sources of ammonia fumes during storage.

5.8 Dilute boric acid solution, $c(H_3BO_3) = 10{,}0$ g/l (optional trapping solution for titrators that automatically begin titration when distillation begins).

Dissolve 100 g of boric acid in about 5 l to 6 l of hot deionized water, mix and add more hot deionized water to a volume of about 9 l. Allow to cool to room temperature. Add 70 ml of the methyl red solution (5.6.1) and 100 ml of the bromocresol green solution (5.6.2) and mix. Dilute to a final volume of 10 l. Depending on the water used, the pH of the boric acid solution can differ from batch to batch. Often an adjustment with a small volume of alkali is necessary to obtain a positive blank (0,05 ml to 0,15 ml of titrant). The colour shall turn green when 100 ml of distilled water are added to 25 ml of the boric acid solution. If still red, titrate with 0,1 mol/l NaOH until "neutral grey" and calculate the amount of alkali needed for the 10 l batch.

Store the solution, which will be light green in colour, at room temperature and protect the solution from light and sources of ammonia fumes during storage.

NOTE The addition of about 3 ml to 4 ml of 0,1 M NaOH into 1 l of 1 % boric acid usually gives good adjustments.

5.9 Hydrochloric acid standard volumetric solution, $c(HCl) = 0{,}100\ 0$ mol/l.

Other concentrations of HCl or sulfuric acid may be used if this is corrected for in the calculations. The concentrations should always be expressed to four decimal places.

5.10 Ammonium sulfate [$(NH_4)_2SO_4$], min. 99,5 % (mass fraction), with certified purity. Dry ammonium sulfate at 102 °C ± 2 °C for 4 h and store in a desiccator.

Percent nitrogen in ammonium sulfate (at 99,5 % purity) is 21,09.

5.11 Ammonium iron(II) sulfate [$(NH_4)_2{\cdot}Fe(SO_4)_2{\cdot}6H_2O$], with certified purity.