

SVENSK STANDARD

SS-EN ISO 5983-2:2009

Fastställt/Approved: 2009-06-15
Publicerad/Published: 2009-08-18
Utgåva/Edition: 2
Språk/Language: engelska/English
ICS: 65.120

Animal feeding stuffs – Determination of nitrogen content and calculation of crude protein content – Part 2: Block digestion and steam distillation method (ISO 5983-2:2009)

This preview is downloaded from www.sis.se. Buy the entire standard via <https://www.sis.se/std-70040>

Hitta rätt produkt och ett leveranssätt som passar dig

Standarder

Genom att följa gällande standard både effektiviserar och säkrar du ditt arbete. Många standarder ingår dessutom ofta i paket.

Tjänster

Abonnemang är tjänsten där vi uppdaterar dig med aktuella standarder när förändringar sker på dem du valt att abonnera på.

På så sätt är du säker på att du alltid arbetar efter rätt utgåva.

e-nav är vår online-tjänst som ger dig och dina kollegor tillgång till standarder ni valt att abonnera på dygnet runt. Med e-nav kan samma standard användas av flera personer samtidigt.

Leveranssätt

Du väljer hur du vill ha dina standarder levererade. Vi kan erbjuda dig dem på papper och som pdf.

Andra produkter

Vi har böcker som underlättar arbetet att följa en standard. Med våra böcker får du ökad förståelse för hur standarder ska följas och vilka fördelar den ger dig i ditt arbete. Vi tar fram många egna publikationer och fungerar även som återförsäljare. Det gör att du hos oss kan hitta över 500 unika titlar. Vi har även tekniska rapporter, specifikationer och "workshop agreement".

Matriser är en översikt på standarder och handböcker som bör läsas tillsammans. De finns på sis.se och ger dig en bra bild över hur olika produkter hör ihop.

Standardiseringsprojekt

Du kan påverka innehållet i framtida standarder genom att delta i någon av SIS ca 400 Tekniska Kommittéer.

Find the right product and the type of delivery that suits you

Standards

By complying with current standards, you can make your work more efficient and ensure reliability. Also, several of the standards are often supplied in packages.

Services

Subscription is the service that keeps you up to date with current standards when changes occur in the ones you have chosen to subscribe to. This ensures that you are always working with the right edition.

e-nav is our online service that gives you and your colleagues access to the standards you subscribe to 24 hours a day. With e-nav, the same standards can be used by several people at once.

Type of delivery

You choose how you want your standards delivered. We can supply them both on paper and as PDF files.

Other products

We have books that facilitate standards compliance. They make it easier to understand how compliance works and how this benefits you in your operation. We produce many publications of our own, and also act as retailers. This means that we have more than 500 unique titles for you to choose from. We also have technical reports, specifications and workshop agreements.

Matrices, listed at sis.se, provide an overview of which publications belong together.

Standardisation project

You can influence the content of future standards by taking part in one or other of SIS's 400 or so Technical Committees.

Europastandarden EN ISO 5983-2:2009 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av EN ISO 5983-2:2009.

Denna standard ersätter SS-EN ISO 5983-2:2006, utgåva 1.

The European Standard EN ISO 5983-2:2009 has the status of a Swedish Standard. This document contains the official English version of EN ISO 5983-2:2009.

This standard supersedes the Swedish Standard SS-EN ISO 5983-2:2006, edition 1.

© Copyright/Upphovsrätten till denna produkt tillhör SIS, Swedish Standards Institute, Stockholm, Sverige. Användningen av denna produkt regleras av slutanvändarlicensen som återfinns i denna produkt, se standardens sista sidor.

© Copyright SIS, Swedish Standards Institute, Stockholm, Sweden. All rights reserved. The use of this product is governed by the end-user licence for this product. You will find the licence in the end of this document.

Upplysningar om sakinnehållet i standarden lämnas av SIS, Swedish Standards Institute, telefon 08-555 520 00.

Standarder kan beställas hos SIS Förlag AB som även lämnar allmänna upplysningar om svensk och utländsk standard.

Information about the content of the standard is available from the Swedish Standards Institute (SIS), tel +46 8 555 520 00.

Standards may be ordered from SIS Förlag AB, who can also provide general information about Swedish and foreign standards.

SIS Förlag AB, SE 118 80 Stockholm, Sweden. Tel: +46 8 555 523 10. Fax: +46 8 555 523 11.

E-mail: sis.sales@sis.se Internet: www.sis.se

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 5983-2

June 2009

ICS 65.120

Supersedes EN ISO 5983-2:2005

English Version

Animal feeding stuffs - Determination of nitrogen content and calculation of crude protein content - Part 2: Block digestion and steam distillation method (ISO 5983-2:2009)

Aliments des animaux - Dosage de l'azote et calcul de la teneur en protéines brutes - Partie 2: Méthode de digestion en bloc et distillation à la vapeur (ISO 5983-2:2009)

Futtermittel - Bestimmung des Stickstoffgehaltes und Berechnung des Rohproteingehaltes - Teil 2: Blockaufschluss- und Dampfdestillationsverfahren (ISO 5983-2:2009)

This European Standard was approved by CEN on 30 May 2009.

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 CEN Management Centre 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 CEN Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: Avenue Marnix 17, B-1000 Brussels

Contents	Page
Foreword.....	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions.....	2
4 Principle	2
5 Reagents	2
6 Apparatus	4
7 Sampling	4
8 Preparation of test sample.....	5
9 Procedure	5
9.1 General.....	5
9.2 Test portion	5
9.3 Determination.....	5
9.4 Blank test.....	6
9.5 Recovery tests	7
10 Calculation and expression of results.....	7
10.1 Calculation.....	7
10.2 Calculation of crude protein content.....	8
10.3 Expression of crude protein content results	8
11 Precision	8
11.1 Interlaboratory tests	8
11.2 Repeatability.....	9
11.3 Reproducibility.....	9
12 Test report	9
Annex A (informative) Results of interlaboratory tests.....	10
Annex B (informative) Results of a proficiency test; comparison of the colorimetric and potentiometric endpoint determination of the titration	14
Bibliography	15

Foreword

This document (EN ISO 5983-2:2009) has been prepared by Technical Committee ISO/TC 34 "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 December 2009, and conflicting national standards shall be withdrawn at the latest by December 2009.

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

This document supersedes EN ISO 5983-2:2005.

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

Endorsement notice

The text of ISO 5983-2:2009 has been approved by CEN as a EN ISO 5983-2:2009 without any modification.

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

Part 2: Block digestion and steam distillation method

WARNING — The use of this method may involve the use of hazardous materials, operations and equipment. This part of ISO 5983 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 is suitable for use as 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 % mass fraction 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 1871, *Food and feed products — General guidelines for the determination of nitrogen by the Kjeldahl method*

ISO 6498, *Animal feeding stuffs — Guidelines for sample preparation*¹⁾

1) To be published. (Revision of ISO 6498:1998)

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 part of ISO 5983

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

3.2

crude protein content

nitrogen content (3.1) as a mass fraction multiplied by the factor 6,25

NOTE The crude protein content is expressed as a percentage mass fraction 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 and the endpoint of the titration can also be detected by means of a potentiometric pH system.

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 In principle, sulfuric acid can also be used for the titration.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity.

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

These tablets are commercially available.

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 integral 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), at least 98 % mass fraction, nitrogen-free ($\rho_{20} \approx 1,84$ 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, e.g. with a mass fraction of 30 % aqueous emulsion.

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

5.6 Indicator solutions.

5.6.1 Methyl red solution. Dissolve 100 mg of methyl red (C₁₅H₁₅N₃O₂) in 100 ml of ethanol or methanol.

5.6.2 Bromocresol green solution. Dissolve 100 mg of bromocresol green (C₂₁H₁₄Br₄O₅S) in 100 ml of ethanol or methanol.

5.7 Concentrated boric acid solution, $c(\text{H}_3\text{BO}_3) = 40,0 \text{ g/l}$.

Dissolve 400 g of boric acid in about 5 l to 6 l of hot water. Mix and add more hot 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 should turn green when 100 ml of 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 is 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, $c(\text{H}_3\text{BO}_3) = 10,0 \text{ g/l}$ (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 water, mix and add more hot 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 should turn green when 100 ml of 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 is 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 mol/l NaOH into 1 l of 1 % mass fraction boric acid usually gives good adjustments.

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

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 [(NH₄)₂SO₄], min. 99,5 % mass fraction, with certified purity. Dry ammonium sulfate at 102 °C ± 2 °C for 4 h and store in a desiccator.

The percentage mass fraction of nitrogen in ammonium sulfate (at 99,5 % mass fraction purity) is 21,09.

5.11 Ammonium iron(II) sulfate [(NH₄)₂Fe(SO₄)₂·6H₂O], with certified purity.

The percentage mass fraction of nitrogen in ammonium iron(II) sulfate (at 100 % mass fraction purity) is 7,145.