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SWEDISH STANDARDS INSTITUTE

Food products – Determination of the total nitrogen content by combustion according to the Dumas principle and calculation of the crude protein content –
Part 2: Cereals, pulses and milled cereal products (ISO 16634-2:2016)

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Europastandarden EN ISO 16634-2:2016 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av EN ISO 16634-2:2016.

Denna standard ersätter ISO/TS 16634-2:2009, utgåva 1.

The European Standard EN ISO 16634-2:2016 has the status of a Swedish Standard. This document contains the official English version of EN ISO 16634-2:2016.

This standard supersedes the Swedish Standard ISO/TS 16634-2:2009, edition 1.

Förhållandet till övriga delar under samma huvudtitel - Utdrag ur Förord i ISO 16634-2:2016
Relations to other parts under the same general title - Extract from the Foreword of ISO 16634-2:2016

ISO 16634 consists of the following parts, under the general title, Food products — Determination of the total nitrogen content by combustion according to the Dumas principle and calculation of the crude protein content:

- Part 1: Oilseeds and animal feeding stuffs
- Part 2: Cereals, pulses and milled cereal products

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), telephone +46 8 555 520 00. Standards may be ordered from SIS Förlag AB, who can also provide general information about Swedish and foreign standards.

Denna standard är framtagen av kommittén för Livsmedel och foder, SIS/TK 435.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 16634-2

June 2016

ICS 67.060; 67.050

Supersedes CEN ISO/TS 16634-2:2009

English Version

Food products - Determination of the total nitrogen content by combustion according to the Dumas principle and calculation of the crude protein content - Part 2: Cereals, pulses and milled cereal products (ISO 16634-2:2016)

Produits alimentaires - Détermination de la teneur en azote total par combustion selon le principe Dumas et calcul de la teneur en protéines brutes - Partie 2: Céréales, légumineuses et produits céréaliers de mouture (ISO 16634-2:2016)

Lebensmittelerzeugnisse - Bestimmung des Gehaltes an Gesamtstickstoff mit dem Verbrennungsverfahren nach Dumas und Berechnung des Gehaltes an Rohprotein - Teil 2: Getreide, Hülsenfrüchte und gemahlene Getreideerzeugnisse (ISO 16634-2:2016)

This European Standard was approved by CEN on 12 May 2016.

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-CENELEC 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-CENELEC Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

Contents

Page

European Foreword

This document (EN ISO 16634-2:2016) has been prepared by Technical Committee ISO/TC 34 "Food products" in collaboration with Technical Committee CEN/TC 338 “Cereal and cereal products” the secretariat of which is held by AFNOR.

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 2016, and conflicting national standards shall be withdrawn at the latest by December 2016.

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 CEN ISO/TS 16634-2:2009.

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, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Endorsement notice

The text of ISO 16634-2:2016 has been approved by CEN as EN ISO 16634-2:2016 without any modification.

Introduction

For a long time, the Kjeldahl method has been the most frequently used method for the determination of the protein content of food products. In recent years, the Dumas method has gained importance compared to the Kjeldahl method because it is faster and does not use dangerous chemicals. Although the principles of the two methods are different, both measure the nitrogen content of the product. Nitrogen content can be converted into protein content by using an appropriate factor. The value of this factor varies depending on the relative amounts of different proteins and their amino-acid composition in a given product.

Neither the Dumas nor the Kjeldahl method distinguishes between protein and non-protein nitrogen. In most cases, results obtained by the Dumas method are slightly higher than those of the Kjeldahl method. This is because the Dumas method measures almost all of the non-protein nitrogen, whereas the Kjeldahl method measures only a part of it.

Taking into consideration that the protein content of a product calculated by both methods only approximates to the true value, it is a matter of discretion which one is accepted. The best solution is to use a second factor for the elimination of the systematic error caused by the non-protein nitrogen content of the different products.

However, this second factor has to be determined for each product like the existing factors which indicate the ratio of the protein content to the nitrogen content.

Food products — Determination of the total nitrogen content by combustion according to the Dumas principle and calculation of the crude protein content —

Part 2:
Cereals, pulses and milled cereal products

1 Scope

This part of ISO 16634 specifies a method for the determination of the total nitrogen content and the calculation of the crude protein content of cereals, pulses and milled cereal products.

This method, like the Kjeldahl method (see References [1] and [6]), does not distinguish between protein nitrogen and non-protein nitrogen. For the calculation of the protein content, various conversion factors are used (see 3.2).

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for 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
mass fraction of the total nitrogen

Note 1 to entry: Determined by the procedure specified in this part of ISO 16634.

Note 2 to entry: The mass fraction is expressed as a percentage.

3.2
crude protein content
nitrogen content (3.1) multiplied by a factor

Note 1 to entry: A 5,7 factor is generally used for cereals for human food (such as wheat, rye and their milled products) and 6,25 for malting barley and cereals for feed and other products falling within the scope of this part of ISO 16634.

Note 2 to entry: The factors for calculation of the crude protein content from the total nitrogen content are derived from the Kjeldahl method, which is the reference method for the determination of total nitrogen content.

4 Principle

Samples are converted into gases by heating in a combustion tube. Interfering components are removed from the resulting gas mixture. The nitrogen compounds in the gas mixture, or a representative part of them, are converted to molecular nitrogen which is quantitatively determined by a thermal-conductivity detector. The nitrogen content is calculated by a microprocessor.

5 Reagents

Use only reagents of recognized analytical grade or reagents of equivalent purity as specified by instrument manufacturers. Except for the reference materials (5.12), all reagents shall be free from nitrogen.

5.1 Carrier gas(es), use either 5.1.1 or 5.1.2.

5.1.1 Carbon dioxide, as pure as possible, but with a minimum CO_2 volume fraction of 99,99 %.

5.1.2 Helium, as pure as possible, but with a minimum He volume fraction of 99,99 %.

5.2 Oxygen, as pure as possible, but with a minimum O_2 volume fraction of 99,99 %.

5.3 Sulfur dioxide and halogen absorbent, to eliminate any sulfur from the sample [e.g. lead chromate ($PbCrO_4$) or steel wool].

5.4 Copper oxide/platinum catalyst, for the post-combustion tube.

Platinum catalyst [5 % of Pt on alumina (Al_2O_3)] is blended with CuO in the ratio 1 part:7 parts or 1 part:8 parts in accordance with the manufacturer's recommendations.

To prevent separation as a result of the different bulk densities of the two materials, it is recommended not to prepare the mixture before filling the tube, but to pour the platinum catalyst and copper oxide simultaneously into the post-combustion tube using a suitable funnel.

5.5 Silver or copper wool.

This shall be disaggregated before being inserted into the post-combustion or reduction tube.

5.6 Silica (quartz) or glass wool or cotton wool, as recommended by the instrument manufacturer.

5.7 Copper or tungsten (wire, cuttings, turnings or powder), for the reduction tube.

The use of copper or tungsten in one of these forms can improve the precision of analytical results for samples with low nitrogen contents (about 1 % mass fraction).

5.8 Diphosphorus pentoxide (P_2O_5) or granulated magnesium perchlorate [$Mg(ClO_4)_2$], or another suitable drying agent, to fill the drying tubes.

5.9 Hollow corundum spheres or aluminium oxide pellets, for the combustion tube.

5.10 Copper oxide (CuO), as filling material for the combustion tube.

5.11 Sodium hydroxide (NaOH), on a support material.

5.12 Aspartic acid ($C_4H_7NO_4$) or ethylenediaminetetraacetic acid ($C_{10}H_{16}N_2O_8$) or glutamic acid ($C_5H_9NO_4$) or hippuric acid ($C_9H_9NO_3$) standard, or other suitable reference materials with a known, constant, certified nitrogen content.

The minimum recovery should preferably be 99 % mass fraction.

5.13 Light petroleum, with a boiling range between 30 °C and 60 °C, or acetone or ethanol.

6 Apparatus

Usual laboratory equipment and, in particular, the following.

6.1 Analytical balance, capable of weighing to the nearest 0,000 1 g.

6.2 Grinding device, appropriate to the nature of the sample.

6.3 Sieve, of nominal opening size 800 µm or 1 mm, made of non-ferrous material.

6.4 Crucibles (e.g. made of stainless steel, quartz, ceramic material or platinum) or tin capsules or tin foils or nitrogen-free filter paper, suitable for the Dumas apparatus used.

NOTE 1 Several instruments provided with an automatic sampler are commercially available.

NOTE 2 Some solid samples (e.g. powders) can be pressed to form pellets.

6.5 Dumas apparatus, fitted with a furnace able to maintain a given temperature greater than or equal to 850 °C, with a thermal-conductivity detector and suitable device for signal integration.

Suitable Dumas apparatus operates according to the general flowchart given in Annex A, although different arrangements and components may be used.

NOTE Schematic diagrams of three commercially available instruments are shown as examples in Figures B.1 to B.3.

To avoid leaks, the sealing O-rings shall be slightly lubricated with high-vacuum grease prior to installation.

Experience has shown that it is important to clean all pieces of silicaware and glassware carefully and to remove fingerprints from tubes, using a suitable solvent (5.13), before inserting them into the furnace.

7 Sampling

A representative sample should have been sent to the laboratory. This sample should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this part of ISO 16634. Recommended sampling methods are given in ISO 24333 for cereals and cereal products.

8 Preparation of the test sample

The test sample shall be prepared from the laboratory sample in such a way that a homogeneous test sample is obtained.

Using a suitable grinding device (6.2), grind the laboratory sample. Generally, pass the ground material through a sieve (6.3) of nominal opening size 800 µm for small sample sizes (under 300 mg) or a sieve of