SVENSK STANDARD
SS-EN ISO 16634-1:2008

Fastställd/Approved: 2008-11-17
Publicerad/Published: 2008-12-18
Utgåva/Edition: 1
Språk/Language: engelska/English
ICS: 65.120; 67.050; 67.060; 67.200

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 (ISO 16634-1:2008)

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 16634-1

November 2008

ICS 65.120; 67.200; 67.050

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 1: Oilseeds and animal feeding stuffs (ISO 16634-1:2008)

Produits alimentaires - Détermination de la teneur en azote total par combustion selon le principe Dumas et calcul de la teneur en protéines brutes - Partie 1: Graines oléagineuses et aliments des animaux (ISO 16634-1:2008)

Lebensmittelerzeugnisse - Bestimmung des Gehaltes an Gesamtstickstoff mit dem Verbrennungsverfahren nach Dumas und Berechnung des Gehaltes an Rohprotein - Teil 1: Ölsaaten und Futtermittel (ISO 16634-1:2008)

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Ref. No. EN ISO 16634-1:2008: E

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Foreword

This document (EN ISO 16634-1:2008) 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of an identical text or by endorsement, at the latest by May 2009, and conflicting national standards shall be withdrawn at the latest by May 2009.

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Introduction

For a long time the Kjeldahl method has been the most frequently used method for the determination of protein content of food products. However, in recent years, the Kjeldahl method has increasingly been replaced by the Dumas method, which is faster and does not use dangerous chemicals.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 can be converted into protein content by using an appropriate factor. The value of this factor varies with the relative amounts of different proteins and their amino-acid composition in the 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 due to the fact that 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 calculated protein content of a product by both methods only approximates the true value, it is a matter of discretion which one is accepted. The most appropriate solution should be the use of 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 existing factors, which show the ratio of the protein 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 1: Oilseeds and animal feeding stuffs

1 Scope

This part of ISO 16634 specifies a method for the determination of the total nitrogen content and the calculation of crude protein content of oilseeds and animal feeding stuffs.

This method, like the Kjeldahl method, does not distinguish between protein nitrogen and non-protein nitrogen. For the calculation of protein content, various conversion factors are used (see Annex D).

This method is not applicable to milk and milk products, for which a method is specified in ISO 14891 | IDF 185[10].

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 referenced document (including any amendments) applies.

ISO 664, *Oilseeds — Reduction of laboratory sample to test sample*

ISO 665, *Oilseeds — Determination of moisture and volatile matter content*

ISO 771, *Oilseed residues — Determination of moisture and volatile matter content*

ISO 6496, *Animal feeding stuffs — Determination of moisture and other volatile matter content*

ISO 6498, *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 the total nitrogen determined by the procedure specified in this part of ISO 16634

NOTE The mass fraction is expressed as a percentage.

3.2
crude protein content
nitrogen content (3.1) multiplied by a factor, usually 6,25

NOTE 1 A listing of other factors for possible use with various commodities is given in Annex D.

NOTE 2 The factors for calculation of crude protein content from the total content of nitrogen are derived from the Kjeldahl method which is the reference method for the determination of total nitrogen content. As the method uses the same factors as the Kjeldahl method, the use of these factors has to be verified due to the slight difference in results between the Kjeldahl and Dumas methods.

4 Principle

Samples are converted to gases by heating in a combustion tube which gasifies samples. 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 one of 5.1.1 and 5.1.2.

5.1.1 Carbon dioxide, as pure as possible and of minimum volume fraction, $\varphi(CO_2) \geqslant 99{,}99$ %.

5.1.2 Helium, as pure as possible and of minimum volume fraction, $\varphi(He) \geqslant 99{,}99$ %.

5.2 Oxygen, as pure as possible and of minimum volume fraction, $\varphi(O_2) \geqslant 99{,}99$ %.

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 (filling material for the post-combustion tube).

Platinum catalyst [5 % of Pt on alumina (Al_2O_3)] is blended with CuO at a ratio of 1:7 parts or 1:8 parts according to 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. It is advisable 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 should be disaggregated before being inserted in 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 (wire, cuttings, turnings or **powder)**, or **tungsten** for the reduction tube.

The use of copper wires 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 support material, 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 known, constant, certified nitrogen content.

Minimum recovery should be 99 % mass fraction.

5.13 Light petroleum, with 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 size of openings 800 µm or 1 mm, made of non-ferrous material.

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

NOTE 1 Several commercial instruments are provided with an automatic sampler.

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

6.5 Dumas apparatus[1)], 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 Schemes of three available instruments are shown as examples in Figures B.1, B.2, and 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 silica and glassware carefully, and to remove fingerprints from the tubes using a suitable solvent (e.g. acetone) before inserting them in the furnace.

1) Elementar Analysensysteme, Sumika Chemical Analysis Service, and LECO Instruments produce suitable equipment available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this equipment. Equivalent products may be used if they can be shown to lead to the same results.