
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

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



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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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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.

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 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

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

This first edition cancels and replaces ISO/TS 16634-2:2009, which has been technically revised.

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*

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.

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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 CO₂ 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 O₂ volume fraction of 99,99 %.

5.3 Sulfur dioxide and halogen absorbent, to eliminate any sulfur from the sample [e.g. lead chromate (PbCrO₄) or steel wool].

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

Platinum catalyst [5 % of Pt on alumina (Al₂O₃)] 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 (P₂O₅) or granulated magnesium perchlorate [Mg(ClO₄)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 (C₄H₇NO₄) or ethylenediaminetetraacetic acid (C₁₀H₁₆N₂O₈) or glutamic acid (C₅H₉NO₄) or hippuric acid (C₉H₉NO₃) 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