

Animal feeding stuffs — Determination of calcium, sodium, phosphorus, magnesium, potassium, sulphur, iron, zinc, copper, manganese, cobalt and molybdenum after pressure digestion by ICP-AES

ICS 65.120

National foreword

This Draft for Development is the UK implementation of CEN/TS 15621:2007.

This publication is not to be regarded as a British Standard.

It is being issued in the Draft for Development series of publications and is of a provisional nature. It should be applied on this provisional basis, so that information and experience of its practical application can be obtained.

Comments arising from the use of this Draft for Development are requested so that UK experience can be reported to the European organization responsible for its conversion to a European standard. A review of this publication will be initiated not later than 3 years after its publication by the European organization so that a decision can be taken on its status. Notification of the start of the review period will be made in an announcement in the appropriate issue of *Update Standards*.

According to the replies received by the end of the review period, the responsible BSI Committee will decide whether to support the conversion into a European Standard, to extend the life of the Technical Specification or to withdraw it. Comments should be sent to the Secretary of the responsible BSI Technical Committee at British Standards House, 389 Chiswick High Road, London W4 4AL.

The UK participation in its preparation was entrusted to Technical Committee AW/10, Animal feeding stuffs.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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English Version

**Animal feeding stuffs - Determination of calcium, sodium,
phosphorus, magnesium, potassium, sulphur, iron, zinc, copper,
manganese, cobalt and molybdenum after pressure digestion by
ICP-AES**

Aliments pour animaux - Détermination du calcium, du sodium, du phosphore, du magnésium, du potassium, du soufre, du fer, du zinc, du cuivre, du manganèse, du cobalt et du molybdène après digestion sous pression par spectrométrie d'émission atomique à plasma couplage inductif (spectrométrie ICP-AES)

Futtermittel - Bestimmung von Calcium, Natrium, Phosphor, Magnesium, Kalium, Schwefel, Eisen, Zink, Kupfer, Mangan, Kobalt und Molybdän nach Druckaufschluss mittels ICP-AES

This Technical Specification (CEN/TS) was approved by CEN on 9 June 2007 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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Foreword

This document (CEN/TS 15621:2007) has been prepared by Technical Committee CEN/TC 327 “Animal feeding stuffs - Methods of sampling and analysis”, the secretariat of which is held by NEN.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: 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.

1 Scope

This Technical Specification specifies a method for the determination of the minerals calcium, sodium, phosphorus, magnesium, potassium and sulphur and the elements iron, zinc, copper, manganese, cobalt, molybdenum in animal feeding stuffs by inductively coupled plasma atomic emission spectrometry (ICP-AES) after pressure digestion.

The method limit of quantification for each element is dependent on the sample matrix as well as of the instrument. The method is not applicable for determination of low concentrations of elements. A limit of quantification of 1 mg/kg should normally be obtained.

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.

EN 13805: 2002, *Foodstuffs – Determination of trace elements – Pressure digestion*.

EN ISO 3696:1995, *Water for analytical laboratory use – Specification and test methods (ISO 3696:1987)*.

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

limit of detection (LOD)

smallest measured content, from which it is possible to deduce the presence of the analyte with reasonable statistical certainty

NOTE The limit of detection is numerically equal to three times the standard deviation of the mean of blank determinations ($n \geq 10$, where n = number of measures) performed under reproducibility conditions.

3.2

limit of quantification (LOQ)

lowest content of the analyte which can be measured with reasonable statistical certainty

NOTE If both trueness and precision are constant over a concentration range around the limit of detection, then the limit of quantification is numerically equal to ten times the standard deviation of the mean of blank determinations ($n \geq 10$, where n = number of measures) performed under reproducibility conditions.

3.3

feed additives

substances are feed additives when they comply with the definition of feed additives given in regulation EU 1831/2003

3.4

animal feeding stuffs

substances are animal feeding stuffs when they comply with the definition of animal feeding stuffs given in regulation EU 178/2002

4 Principle

For the determination of calcium, sodium, phosphorus, magnesium, potassium, sulphur, iron, zinc, copper, manganese, cobalt and molybdenum, a test portion of the sample is digested under pressure.

The concentration of the elements is determined by inductively coupled plasma atomic emission spectrometry (ICP-AES) using external calibration or standard addition technique.

WARNING – The use of this Technical Specification can involve hazardous materials, operations and equipment. This Specification does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this Technical Specification to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

5.1 Water, complying with grade 2 as defined in EN ISO 3696.

5.2 Nitric acid, concentrated, not less than 65 % (mass fraction), $c(\text{HNO}_3) = 14,4 \text{ mol/l}$, having a density of approximately $\rho(\text{HNO}_3) 1,42 \text{ g/ml}$.

5.3 Nitric acid solution of 2 % (v/v), to be prepared: pipette 20 ml nitric acid (5.2) in a 1000 ml volumetric flask (6.4) and fill to the mark with water.

5.4 Hydrogen peroxide, not less than 30 % (mass fraction).

5.5 Element stock solutions

Ca, Na, P, Mg, K, S, Fe, Zn, Cu, Mn, Co, Mo

$c = 1000 \text{ mg/l}$.

The user shall choose a suitable stock solution. Both single-element stock solutions and multi-element stock solutions with adequate specification stating the acid used and the preparation technique are commercially available. It is advisable to use certified stock solutions.

NOTE Element stock solutions with concentrations different from 1000 mg/l may be used as well.

5.6 Standard solutions

Depending on the scope, different multi-element standard solutions may be necessary. In general, when combining multi-element standard solutions, their chemical compatibility and the possible hydrolysis of the components shall be regarded. Spectral interferences from other elements present in multi-element standards also need to be considered.

Various combinations of elements at different concentrations can be used, provided that the element stock solutions (5.5) are diluted, with the same acid and equal concentration as the acid in the test solution, to a range of standards that covers the concentrations of the elements to be determined.

The multi-element standard solutions are considered to be stable for several months, if stored in the dark.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

NOTE For the determination of sodium in low concentrations it is advisable not to use glassware since glassware can be a source of sodium contamination.

6.1 Laboratory grinder

6.1.1 Laboratory grinder capable of grinding to a particle size of less than or equal to 1 mm, e.g. a knife mill or equivalent.

6.1.2 Laboratory grinder capable of grinding to a particle size of less than or equal to 0,1 mm, e.g. a ball mill or equivalent.

6.1.3 Mortar with pestle, free of contamination.

NOTE It should be checked that the mill used does not influence the concentration of elements in the sample (e.g. adsorption, contamination) which have to be analysed.

6.2 Analytical balance, capable of weighing to an accuracy of 1 mg.

6.3 Pressure digestion apparatus, commercially available.

The apparatus shall be tested for safety pressure vessels made of acid-resistant materials and having holders for the sample of acid-resistant material with low level of contamination by elements to be determined. Apparatus is available which uses a high-pressure incinerator with or without ambient autoclave pressure.

Instead of polytetrafluoroethylene (PTFE) holders, it is better to use graduated quartz holders, perfluoro ethylene propylene (FEP) holders or perfluoro alkoxy (PFA) holders. Quartz is advisable to be used for decomposition temperatures above 230 °C.

6.4 One-mark volumetric flasks, of capacity 1000 ml.

6.5 Inductively coupled plasma – Atomic Emission Spectrometer

The instrument shall be equipped with radial plasma as a minimum requirement; axial plasma is equally acceptable. Background correction shall also be performed when necessary. Settings of the working conditions (e.g. viewing height, gas flows, RF or plasma power, sample uptake rate, integration time, number of replicates, ...) shall be optimised according the manufacturer's instructions.

6.6 Freeze drying equipment, capable of freeze-drying liquid animal feeding stuffs.

7 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in EN-ISO 6497[1].

It is important that the laboratory receives a sample which is truly representative and has not been damaged or changed during transport or storage.