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Animal feeding stuffs - Determination of calcium, sodium, phosphorus, magnesium, potassium, iron, zinc, copper, manganese, cobalt, molybdenum, arsenic, lead and cadmium by ICP-AES

Futtermittel - Bestimmung von Calcium, Natrium, Phosphor, Magnesium, Kalium, Eisen, Zink, Kupfer, Mangan, Cobalt, Molybdän, Arsen, Blei und Cadmium mittels ICP-AES

Aliments des animaux - Détermination des teneurs en calcium, sodium, phosphore, magnésium, potassium, fer, zinc, cuivre, manganèse, cobalt, molybdène, arsenic, plomb et cadmium par ICP-AES

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65.120 Krmila Animal feeding stuffs

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ICS 65.120

English Version

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phosphorus, magnesium, potassium, iron, zinc, copper,
manganese, cobalt, molybdenum, arsenic, lead and cadmium by
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This European Standard was approved by CEN on 30 June 2007.

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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.

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Foreword

This document (EN 15510:2007) 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.

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of an identical text or by endorsement, at the latest by February 2008, and conflicting national standards shall be withdrawn at the latest by February 2008.

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 United Kingdom.

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1 Scope

This European Standard specifies inductively coupled plasma atomic emission spectroscopy (ICP-AES) method for the determination of:

- minerals calcium, sodium, phosphorus, magnesium and potassium and the elements iron, zinc, copper, manganese, cobalt, molybdenum in animal feeding stuffs,
- elements arsenic, lead and cadmium in minerals on their own, in pre-mixtures or mixtures for use in animal nutrition.

The method detection limit 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. The limit of quantification should be 3 mg/kg or lower.

NOTE This method can also be used for the determination of minerals in products with high mineral content (> 5%), yet for this purpose, other more precise analytical techniques are available.

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

ISO 6498, *Animal feeding stuffs – Preparation of test samples*

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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 that 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 that comply with the definition of animal feeding stuffs given in regulation EU 178/2002'

4 Principle

For the determination of the minerals calcium, sodium, phosphorus, magnesium and potassium and the elements iron, zinc, copper, manganese, cobalt, molybdenum, a test portion of the sample is ashed and dissolved in hydrochloric acid (in the case of organic feeding stuffs) or wet digested with hydrochloric acid (in the case of mineral compounds).

For the determination of the elements arsenic, cadmium and lead, a test portion of the sample is wet digested with nitric acid.

The concentration of the elements calcium, sodium, phosphorus, magnesium, potassium, iron, zinc, copper, manganese, cobalt, molybdenum, arsenic, cadmium and lead is determined by inductively coupled plasma atomic emission spectrometry (ICP-AES) using external calibration or standard addition technique.

WARNING – Use of this European Standard can involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this European Standard 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), having a density of approximately (HNO₃) 1,42 g/ml.

5.3 Dilute nitric acid, to be prepared by mixing 1 volume of nitric acid (5.2) with 1 volume of water.

5.4 Nitric acid solution of 5 % (m/v), to be prepared: pipette 160 ml of dilute nitric acid (5.3) into a 1 000 ml volumetric flask (6.7) and fill to the mark with water.

5.5 Nitric acid solution of 2 % (v/v), to be prepared: pipette 20 ml of nitric acid (5.2) into a 1 000 ml volumetric flask (6.7) and fill to the mark with water.

5.6 Hydrochloric acid, concentrated, not less than 30 % (mass fraction), having a density of approximately (HCl) 1,15 g/ml.

5.7 Dilute hydrochloric acid, to be prepared by mixing 1 volume of hydrochloric acid (5.6) with 1 volume of water.

5.8 Hydrochloric acid solution of 1 % (m/v), to be prepared: pipette 60 ml of dilute hydrochloric acid (5.7) into a 1 000 ml volumetric flask (6.7) and fill to the mark with water.

5.9 Element stock solutions

Ca, Na, P, Mg, K, Fe, Zn, Cu, Mn, Co, Mo, Cd, Pb, As

$c = 1\ 000\ \text{mg/l}$.

The user should 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.

Stock solutions are not to be used after the expiry date.

NOTE Element stock solutions with concentrations different from 1 000 mg/l may also be used.

5.10 Standard solutions

5.10.1 General

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 in multi-element standard solutions also need to be considered (Annex B.2.2). The examples given below also consider the measuring range of various inductively coupled plasma atomic emission spectrometers and the expected concentration of the element in animal feeding stuffs.

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

Other combinations of elements at different concentrations can be used, provided that the element stock solutions (5.9) 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.

5.10.2 Multi-element standard solution – Minerals in 1 % HCl

c (Ca, Na, P, Mg, K) = 40 mg/l

Pipette 40,0 ml of each element stock solution (Ca, Na, P, Mg, K) (5.9) into a 1 000 ml volumetric flask (6.7). Add 60 ml of dilute hydrochloric acid (5.7). Fill to the mark with water and transfer to a suitable storage bottle.

5.10.3 Multi-element standard solution - Fe, Zn, Cu, Mn, Co, Mo in 1 % HCl

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c (Fe, Zn, Cu, Mn, Co, Mo) = 50 mg/l

Pipette 50,0 ml of each element stock solution (Fe, Zn, Cu, Mn, Co, Mo) (5.9) into a 1 000 ml volumetric flask (6.7). Add 60 ml of dilute hydrochloric acid (5.7). Fill to the mark with water and transfer to a suitable storage bottle.

5.10.4 Multi-element standard solution – Cd, Pb, As in 5 % HNO₃

c (Cd, Pb, As) = 100 mg/l

Pipette 100,0 ml of each element stock solution (Cd, Pb, As) (5.9) into a 1000 ml volumetric flask (6.7). Add 160 ml dilute nitric acid (5.3). Fill to the mark with water and transfer to a suitable storage bottle.

6 Apparatus, usual laboratory apparatus and, in particular, the following.

6.1 Laboratory grinder

6.1.1 Use laboratory grinders that are equipped such that they do not lead to contamination of the samples.

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

6.1.3 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.

6.1.4 Mortar with pestle, free of contamination.

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

6.3 **Electric hot plate**, with temperature control.

6.4 **Ashing crucibles**, of platinum, quartz or porcelain.

6.5 **Electric muffle-furnace**, capable of being maintained at a temperature of $450\text{ °C} \pm 20\text{ °C}$.

The real temperature in the furnace has to be checked, because this temperature may be substantially different from the adjust temperature.

6.6 **Beaker**, of capacities 100 ml, 250 ml.

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6.7 **One-mark volumetric flasks**, of capacities 100 ml, 500 ml, 1 000 ml.

6.8 Inductively coupled plasma – Atomic Emission Spectrometer

The instrument shall be equipped with a radial plasma as a minimum requirement; an 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.

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

7 Sampling

Sampling is not part of the method specified in this Standard. A recommended sampling method is given in EN ISO 6497.

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

8 Preparation of the test sample

8.1 General

Prepare the test sample in accordance with ISO 6498.

- Grinding must be carried out in conditions such that the substance is not appreciably heated.
- Operation is to be repeated as many times as is necessary and it must be affected as quickly as possible in order to prevent any gain or loss of constituents (water).
- Whole ground product is placed in a flask made of e.g. polypropylene, which can be stoppered and stored in such way to prevent any change in composition.
- Before any weighing is carried out for the analysis, the whole test sample must be thoroughly mixed for reasons of homogeneity.

8.2 Animal feeding stuffs which can be ground as such

Grind the laboratory sample (usually 500 g), using a grinder (6.1.2) or mortar (6.1.4), until a particle size of 1 mm or less has been reached.

8.3 Liquid animal feeding stuffs

8.3.1 General

Liquid feeding stuffs shall be pre-dried according to the procedure described in 8.3.2 or freeze-dried according to the procedure described in 8.3.3.

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8.3.2 Pre-drying

Pre-dry the laboratory sample at $70\text{ °C} \pm 5\text{ °C}$ over at least 16 h to reduce the moisture content. The mass of the sample before and after the pre-drying is determined using an analytical balance (6.2). Grind the pre-dried sample in accordance with 8.2.

8.3.3 Freeze-drying

Freeze-dry the laboratory sample following the instructions of the freeze-drying equipment (6.9). The mass of the sample before and after the freeze-drying is determined using an analytical balance (6.2). Grind the freeze-dried sample in accordance with 8.2.

8.4 Mineral animal feeding stuffs

Mineral compounds, except mineral products containing crystalline water, e.g. $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, shall be ground using a grinder (6.1.3) or mortar until a particle size of 0,1 mm or less has been reached. Mineral products containing crystalline water should not be ground.

9 Procedure

9.1 Digestion

9.1.1 Selection of the procedure

9.1.1.1 Determination of Ca, Na, P, Mg, K, Fe, Zn, Cu, Mn, Co, Mo

If the test sample concerns a mineral compound or a product potentially containing phosphates, proceed in accordance with 9.1.2.

If the test sample contains organic substances and if it is free from phosphates rendering insoluble products on ashing, proceed in accordance with 9.1.3.

If the test sample contains organic substances and phosphates, proceed in accordance with 9.1.2.

9.1.1.2 Determination of Cd, Pb, As in minerals

For the determination of Cd, Pb and As in minerals, proceed in accordance with 9.1.4.

9.1.2 Extraction with 1% HCl

Weigh about 1 g of the prepared test sample to the nearest 1 mg into a beaker of 250 ml (6.6).

Add 30 ml dilute hydrochloric acid (5.7). Add about 100 ml of water.

Cover the beaker (6.6) with a watch-glass and boil for 30 min on a hot plate (6.3).

Allow to cool. Transfer the liquid into a 500 ml volumetric flask (6.7), rinsing the beaker and the watch-glass several times with water.

Leave to cool, dilute to the mark with water.

After homogenising, filter through a dry folded filter paper into a dry conical flask. Use the first portion of the filtrate to rinse the glass ware and discard that part. If the determination is not carried out immediately, the conical flask with the filtrate shall be stoppered.

Carry out a blank determination at the same time as the extraction, with only the reagents and follow the same procedure as for the samples.

Proceed in accordance with 9.2.

When the expected concentration of the element is lower than 100 mg/kg, proceed as described in 9.1.2, but use 12 ml dilute hydrochloric acid (5.7) and 70 ml of water, and transfer the liquid into a 100 ml volumetric flask (6.7).

9.1.3 Dry ashing – 1% HCl

Weigh 5 g of the prepared test sample to the nearest 1 mg in an ashing crucible (6.4).

Ash in the furnace (6.5), set at a temperature of 450 °C, until white or grey ash is obtained (a small quantity of carbon does not interfere).

Transfer the ash to a 250 ml beaker (6.6) with 30 ml of dilute hydrochloric acid (5.7). Add 100 ml of water.

Cover the beaker (6.6) with a watch-glass and boil for 30 min on a hot plate (6.3).

Allow to cool. Transfer the liquid into a 500 ml volumetric flask (6.7), rinsing the beaker and the watch-glass several times with water.

Leave to cool, dilute to the mark with water.

After homogenising, filter through a dry folded filter paper into a dry conical flask. Use the first portion of the filtrate to rinse the glassware and discard that part. If the determination is not carried out immediately, the conical flask with the filtrate shall be stoppered.

Carry out a blank determination at the same time as the extraction, with only the reagents and follow the same procedure as for the samples.

Proceed in accordance with 9.2.

When the expected concentration of the element is lower than 100 mg/kg, proceed as described in 9.1.3, but use 12 ml dilute hydrochloric acid (5.7) and about 70 ml of water, and transfer the liquid into a 100 ml volumetric flask (6.7).

9.1.4 Extraction with 5% HNO₃

Weigh about 2 g of the prepared test sample to the nearest 1 mg into a beaker of 100 ml (6.6).

Add 16 ml dilute nitric acid (5.3). Add about 70 ml of water.

Cover the beaker (6.6) with a watch-glass and boil for 30 min on a hot plate (6.3).

Allow to cool. Transfer the liquid into a 100 ml volumetric flask (6.7), rinsing the beaker and the watch-glass several times with water.

Leave to cool, dilute to the mark with water.

After homogenising, filter through a dry folded filter paper into a dry conical flask. Use the first portion of the filtrate to rinse the glassware and discard that part. If the determination is not carried out immediately, the conical flask with the filtrate shall be stoppered.

Carry out a blank determination at the same time as the extraction with only the reagents and follow the same procedure as for the samples.

Proceed in accordance with 9.2. <https://standards.iteh.ai/catalog/standards/sist/97e4eb3c-3a0a-4fc0-bc0a-e80f6f5e59b/sist-en-15510-2007>

9.2 Calibration

9.2.1 General

Calibration shall be performed by means of external calibration or standard addition technique. It is important that the measurements are made in the linear range of the instrument. Appropriate matrix matching of the calibration solutions shall be performed if an (external) calibration method is used (see Annex B).

9.2.2 External calibration

The calibration is performed with at least two calibration solutions of which one is a blank calibration solution. In all cases linearity should be checked on regular basis. If linearity is guaranteed, calibrate with at least two calibration solutions, if linearity is not guaranteed, calibrate with at least three equidistant calibration solutions (B.3.2).

9.2.3 Standard addition technique

The standard addition curve should consist of at least two points one of which is an addition (B.3.4). For those elements whose concentration is near the limit of quantification, the standard addition curve should consist of at least four points of which three are additions. If three additions are used, the concentration of the highest standard should be three to five times the concentration in the sample solution.

9.2.4 Example of calibration with one addition after dry ashing – 1% HCl

EXAMPLE Determination of copper in a mixed feed with expected concentration 200 mg/kg Cu.