SLOVENSKI STANDARD
SIST EN 15550:2008

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Krma - Določevanje kadmija in svinca z atomsko absorpcijsko spektrometrijo (GF-AAS) z grafitno kiveto po razklopu pod tlakom

Animal feeding stuffs - Determination of cadmium and lead by graphite furnace atomic absorption spectrometry (GF-AAS) after pressure digestion

Futtermittel - Bestimmung von Cadmium und Blei mittels Graphitrohrofen-Atomabsorptionsspektrometrie (GF-AAS) nach Druckaufschluss

Aliments des animaux - Détermination de la teneur en cadmium et en plomb par spectrométrie d'absorption atomique a four graphite (GF-AAS) apres digestion sous pression

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English Version

Animal feeding stuffs - Determination of cadmium and lead by graphite furnace atomic absorption spectrometry (GF-AAS) after pressure digestion

Aliments des animaux - Détermination de la teneur en cadmium et en plomb par spectrométrie d'absorption atomique à four graphite (GF-AAS) après digestion sous pression

Futtermittel - Bestimmung von Cadmium und Blei mittels Graphitrohrofen-Atomabsorptionsspektrometrie (GF-AAS) nach Druckaufschluss

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (EN 15550:2007) 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of an identical text or by endorsement, at the latest by April 2008, and conflicting national standards shall be withdrawn at the latest by April 2008.

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.

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1 Scope

This European Standard specifies a method for the determination of the elements cadmium and lead in animal feeding stuffs by graphite furnace atomic absorption spectrometry (GF-AAS) after pressure digestion.

The method limit of quantification for each element is dependent on the sample matrix as well as the instrument. For cadmium a limit of quantification of 0,05 mg/kg should normally be obtained while for lead, a limit of quantification of 0,5 mg/kg should 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 ISO 3696, *Water for analytical laboratory use – Specification and test methods (ISO 3696:1987)*

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
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 ($n \geq 10$, were n = number of measures) performed under reproducibility conditions.

3.3
feed additives
substances that comply with the definition of feed additives given in the Regulation (EC) No 1831/2003 of the European Parliament and of the Council of 22 September 2003 on additives for use in animal nutrition

3.4
animal feeding stuffs
substances that comply with the definition of animal feeding stuffs given in the Regulation (EC) No 178/2002

4 Principle

For the determination of the elements cadmium and lead, a test portion of the sample is digested under pressure.

The concentration of the elements is determined by graphite furnace atomic absorption spectrometry (GF-AAS) using external calibration.

The method detection limit for each element is dependent on the sample matrix as well as the instrument, the type of atomizer and the use of chemical modifiers. A typical sample volume of 10 µl to 20 µl is used.

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

5.1 General

Use only reagents of recognized analytical grade, and water conforming to grade 2 of EN ISO 3696.

5.2 Nitric Acid, concentrated, not less than 65 % (mass fraction), $c(HNO_3)$ = 14,4 mol/l, having a density of approximately $\rho(HNO_3)$ = 1,42 g/ml.

5.3 Hydrogen peroxide, mass fraction not less than 30 %.

5.4 Element stock solutions

Cd, Pb

c = 1 000 mg/l

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

Stock solutions should not be used after expiration dates.

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

5.5 Calibration solutions

Prepare a range of standards including a blank calibration solution, which covers the linear range of the element to be determined by diluting the element stock solutions (5.4). Appropriate matrix matching of the calibration solutions shall be performed (see Annex B), e.g. adjust the acid concentration of the standards to the acid concentration of the samples. Dilute to volume with water.

5.6 Matrix modifier (e.g. Palladium nitrate/magnesium nitrate modifier)

$Pd(NO_3)_2$ solution (Pd-nitrate solution) is commercially available (mass concentration 10 g/l). Dissolve 0,259 g of $Mg(NO_3)_2$ $6H_20$ (Mg-nitrate solution) in 100 ml of water. Mix the Pd-nitrate solution with twice as much Mg-nitrate solution. 10 µl of the mixed solution is equal to 15 µg Pd and 10 µg $Mg(NO_3)_2$. It is advisable to use this solution for not longer than one week.

The combination of Pd and $Mg(NO_3)$ is regarded as a “universal” modifier that could be used for a lot of elements. Other matrix modifiers may be used as well, e.g. palladium nitrate modifier and ammonium dihydrogen phosphate modifier.

5.7 Purge and protective gas

Argon, Ar purity not less than 99,99% by volume.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Laboratory grinder

6.1.1 Use laboratory grinders that are equipped so that samples cannot be contaminated.

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 with 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 a low level of contamination. Apparatus are available that 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 Graphite furnace atomic absorption spectrometer, with background correction, e.g. Zeeman, supplied with auto sampler, an appropriate gas (5.7) supply and hollow cathode lamps or EDL-lamps for lead and cadmium.

NOTE It is necessary to place an exhaust venting system over the furnace to remove any smoke and vapours that might be harmful.

6.5 Graphite tubes, pyrolytically coated and preferably with platforms.

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.

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 done 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 effected 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.

8.3.2 Pre-drying

Pre-dry the laboratory sample at 70 °C ± 5 °C during at least 16 h to reduce the moisture content. The mass of the sample before and after the pre-drying is to be 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 (6.6). The mass of the sample before and after the freeze-drying is to be 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. $MgCl_2 \cdot 6H_2O$, shall be ground using a grinder (6.1.3) or mortar (6.1.4), 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 General

Use pressure digestion. Proceed in accordance with 9.1.2.