



BSI Standards Publication

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

National foreword

This British Standard is the UK implementation of EN 15550:2017. It supersedes BS EN 15550:2007, which is withdrawn.

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.

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English Version

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

Aliments des animaux - Méthodes d'échantillonnage et
d'analyse - Dosage du cadmium et du plomb par
spectrométrie d'absorption atomique à l'aide d'un four
graphite après minéralisation sous pression

Futtermittel - Probenahme und
Untersuchungsverfahren - Bestimmung von Cadmium
und Blei mittels Graphitrohrfurnen-
Atomabsorptionsspektrometrie (GF-AAS) nach
Druckaufschluss

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European foreword

This document (EN 15550:2017) 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of an identical text or by endorsement, at the latest by February 2018, and conflicting national standards shall be withdrawn at the latest by February 2018.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 15550:2007.

WARNING — The method described in this standard implies the use of reagents that pose a hazard to health. The standard does not claim to address all associated safety problems. It is the responsibility of the user of this standard to take appropriate measures for the health and safety protection of the personnel prior to use of the standard and to ensure that regulatory and legal requirements are complied with.

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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 was successfully tested in the range of 0,015 mg/kg to 5,65 mg/kg for Cd and 0,18 mg/kg to 40,3 mg/kg for lead in 11 animal feeds: 2 complete feeds (pig feed, sheep feed), 2 complementary feeds (2 mineral feeds), 1 mineral premixture, 4 feed materials (MgO, 2 phosphates, CaCO₃) and 2 feed additives (CuSO₄, bentonite).

For the determination of extractable lead in minerals and feeds, containing phyllosilicates (e.g. kaolinite clay) wet digestion with diluted nitric acid should be used.

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 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.

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696)*

EN ISO 6498, *Animal feeding stuffs - Guidelines for sample preparation (ISO 6498)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

limit of detection (LOD)

concentration (mass fraction) derived from the smallest measure that can be detected with reasonable certainty for a given analytical procedure

Note 1 to entry: x_{LOD} denotes the limit of detection expressed as concentration (mass fraction).

[SOURCE: ISO 11843-1, see [1]]

Note 2 to entry: For analytical systems where the validation range does not include or approach it, the detection limit does not need to be part of a validation.

[SOURCE: IUPAC 2002, see [2]]

Note 3 to entry: The various conceptual approaches to the subject depend on the estimate of precision at or near zero concentration (mass fraction) under repeatability or reproducibility conditions.

3.2

limit of quantification (LOQ)

lowest concentration (mass fraction) determined with satisfactory measurement uncertainty

Note 1 to entry: x_{LOQ} denotes the limit of quantification expressed as concentration (mass fraction)

[SOURCE: ISO 11843-1, see [1]]

Note 2 to entry: It is useful to state a concentration (mass fraction) below which the analytical method cannot operate with an acceptable precision. Such precision is arbitrarily defined as relative standard deviation of 10 %.

Note 3 to entry: Usually the limit is set as a fixed multiple (e.g. 3.3) of the detection limit.

Note 4 to entry: Measurements below LOQ are not devoid of information content and are fit for purpose.

[SOURCE: IUPAC 2002, see [2]]

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]

3.4

animal feeding stuffs

substances that comply with the definition of animal feeding stuffs given in the Regulation (EC) No 178/2002 [4]

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.

5 Reagents

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

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.

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

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