

Loomasööt. Aeseeni määramine hüdroiide moodustuva aatomabsorptsioonspektromeetriga (HD-AAS) pärast mikrolainete surve digereerimist (digereerimine 65 % lämmastikhappega ja 30 % vesinikülihapendiga)

Animal feeding stuffs - Determination of arsenic by hydride generation atomic absorption spectrometry (HGAAS) after microwave pressure digestion (digestion with 65 % nitric acid and 30 % hydrogen peroxide)

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

See Eesti standard EVS-EN 16206:2012 sisaldab Euroopa standardi EN 16206:2012 ingliskeelset teksti.	This Estonian standard EVS-EN 16206:2012 consists of the English text of the European standard EN 16206:2012.
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English Version

Animal feeding stuffs - Determination of arsenic by hydride generation atomic absorption spectrometry (HGAAS) after microwave pressure digestion (digestion with 65 % nitric acid and 30 % hydrogen peroxide)

Aliments pour animaux - Dosage de l'arsenic par spectrométrie d'absorption atomique par génération d'hydrures (SAAGH) après digestion sous pression par micro-ondes (Extraction à l'acide nitrique à 65 % et au peroxyde d'hydrogène à 30 %)

Futtermittel - Bestimmung von Arsen mit Atomabsorptionsspektrometrie-Hydridtechnik (HD-AAS) nach Mikrowellen-Druckaufschluss (Aufschluss mit 65% Salpetersäure und 30% Wasserstoffperoxid)

This European Standard was approved by CEN on 30 December 2011.

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Foreword

This document (EN 16206:2012) has been prepared by Technical Committee CEN/TC 327 "Animal feeding stuffs", 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of an identical text or by endorsement, at the latest by August 2012, and conflicting national standards shall be withdrawn at the latest by August 2012.

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1 Scope

This European Standard specifies a method for the determination of total arsenic in animal feeding stuffs by hydride generation atomic absorption spectrometry (HGAAS) after microwave pressure digestion. The limit of quantification is 0,5 µg/l of the test solution. Using a test portion of 0,5 g, a volume of the test solution of 25 ml and an aliquot of 5 ml for pre-reduction the limit of quantification is 0,125 mg/kg in the feed material.

NOTE For feed materials containing organic arsenic species from compounds of marine origin (i.e. arsenobetaine and tetramethylarsine oxide) a higher digestion temperature of the microwave system up to 300 °C may be necessary in order to enable the hydridisation of these arsenic compounds and in order to determine all different kinds of arsenic species in the corresponding feeding stuffs. Alternatively, the digestion procedure of Annex C can be used if the microwave system does not reach higher temperatures up to 300 °C to ensure complete mineralization for HGAAS determination.

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 6497, *Animal feeding stuffs — Sampling (ISO 6497)*

prEN ISO 6498, *Animal feeding stuffs — Guidelines for sample preparation (ISO/DIS 6498)*

3 Principle

Arsenic is determined in the test solution by hydride generation atomic absorption spectrometry (HGAAS) after microwave pressure digestion and a pre-reduction step.

The homogenised feeding stuff test sample is digested by nitric acid and hydrogen peroxide under pressure and high temperatures in a microwave-heated pressure digestion system.

Arsenic ions of the test solution are reduced with a potassium iodide/ascorbic acid solution and hydrochloric acid to arsenic (III) and converted to arsenic hydride (AsH₃) by sodium borohydride. Arsenic hydride is transferred by a gas stream into a heated measurement cell and decomposed. The absorption at the arsenic line at 193,7 nm corresponds to the amount of arsenic.

Since arsenic (III) and arsenic (V) show a different sensitivity with the hydride technique, it is necessary to reduce arsenic (V) to arsenic (III) in order to avoid incorrect measurements.

Other digestion procedures with the same digestion efficiency are possible in order to completely mineralize all arsenic species like organic arsenic species from compounds of marine origin for HGAAS determination (see Annex C).

NOTE 1 When using e.g. perchloric acid as alternative digestion procedure to ensure complete mineralisation of all organic and inorganic arsenic species for HGAAS determination you must use NaI/L-ascorbic acid because KI results in precipitation of potassium perchlorate.

NOTE 2 Alternatively, inductively-coupled-plasma mass-spectrometry (ICP-MS) for measuring can be used where an incomplete mineralization is not of importance.

WARNING — The use of this 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 standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.