

**Loomasööt. Seleenini määramine
aatomabsorptsioonspektromeetria hüdriidide
genereerimise meetodiga (HD-AAS) pärast mikrolainete
digereerimist (digereerimine 65 % lämmastikhappe ja 30
% vesinikperoksiidiga)**

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

EESTI STANDARDI EESSÕNA

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ICS 65.120

English Version

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

Aliments pour animaux - Dosage du sélénium par spectrométrie d'absorption atomique par génération d'hydrures (SAAGH) après digestion par micro-ondes (extraction avec de l'acide nitrique à 65 % et du peroxyde d'hydrogène à 30 %)

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

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 16159:2012) 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of an identical text or by endorsement, at the latest by August 2012, and conflicting national standards shall be withdrawn at the latest by August 2012.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 selenium in animal feeding stuffs by hydride generation atomic absorption spectrometry (HGAAS) after microwave pressure digestion.

The method was successfully tested by an inter-laboratory study of CEN/TC 327/WG 4 in the range of 0,25 mg/kg to 74 mg/kg.

The limit of quantification is 0,5 µg/l of the test solution which corresponds to the calibration standard 2. Using a test portion of 0,5 g and a volume of the test solution of 25 ml after pressure digestion the limit of quantification is calculated as 0,125 mg/kg in the feed material.

NOTE A lower limit of quantification could be achieved – each laboratory has to prove it.

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

Selenium is determined in the test solution by hydride generation atomic absorption spectrometry (fluorescence hydride generation atomic absorption) 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.

Selenium ions of the test solution are reduced with hydrochloric acid to selenium (IV) and converted to selenium hydride (SeH₂) by sodium borohydride. This selenium hydride is transferred by a gas stream to a heated measurement cell and decomposed. The absorption at the selenium line at 196,0 nm corresponds to the amount of selenium.

NOTE Selenium (VI) is not determined by the hydridisation as described here. It is therefore necessary to adjust the digestion conditions and to exercise a pre-reduction step with hydrochloric acid to yield only selenium (IV).

Other digestion procedures with the same digestion efficiency or other measurement systems like FI-HGAAS or hydride generation inductively coupled plasma optical emission spectrometry are possible (see Annex C).

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.