

Animal feeding stuffs: Methods of sampling and analysis
- Determination of inorganic arsenic in animal feed by
anion-exchange HPLC-ICP-MS

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

See Eesti standard EVS-EN 17374:2020 sisaldab Euroopa standardi EN 17374:2020 ingliskeelset teksti.	This Estonian standard EVS-EN 17374:2020 consists of the English text of the European standard EN 17374:2020.
Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas.	This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation.
Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 01.07.2020.	Date of Availability of the European standard is 01.07.2020.
Standard on kättesaadav Eesti Standardikeskusest.	The standard is available from the Estonian Centre for Standardisation.

Tagasisidet standardi sisu kohta on võimalik edastada, kasutades EVS-i veebilehel asuvat tagasiside vormi või saates e-kirja meiliaadressile standardiosakond@evs.ee.

ICS 65.120

Standardite reprodutseerimise ja levitamise õigus kuulub Eesti Standardikeskusele

Andmete paljundamine, taastekitamine, kopeerimine, salvestamine elektroonsesse süsteemi või edastamine ükskõik millises vormis või millisel teel ilma Eesti Standardikeskuse kirjaliku loata on keelatud.

Kui Teil on küsimusi standardite autorikaitse kohta, võtke palun ühendust Eesti Standardikeskusega:
Koduleht www.evs.ee; telefon 605 5050; e-post info@evs.ee

The right to reproduce and distribute standards belongs to the Estonian Centre for Standardisation

No part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying, without a written permission from the Estonian Centre for Standardisation.

If you have any questions about copyright, please contact Estonian Centre for Standardisation:

Homepage www.evs.ee; phone +372 605 5050; e-mail info@evs.ee

ICS 65.120

English Version

Animal feeding stuffs: Methods of sampling and analysis - Determination of inorganic arsenic in animal feed by anion-exchange HPLC-ICP-MS

Aliments des animaux - Méthodes d'échantillonnage et d'analyse - Détermination de la teneur en arsenic inorganique dans les aliments pour animaux, par CLHP avec échange d'anions et spectrométrie de masse à plasma induit par haute fréquence (ICP-SM)

Futtermittel: Probenahme- und Untersuchungsverfahren - Bestimmung von anorganischem Arsen in Futtermittel mittels Anionenaustausch HPLC-ICP-MS

This European Standard was approved by CEN on 10 May 2020.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

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-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.



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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

Contents	Page
European foreword.....	3
1 Scope	4
2 Normative references	4
3 Terms and definitions	4
4 Principle	4
5 Reagents	4
6 Apparatus and equipment	6
7 Sampling	7
8 Procedure	7
8.1 Sample preparation	7
8.2 Water bath extraction	7
8.3 Determination of inorganic arsenic by HPLC-ICP-MS	8
8.3.1 General	8
8.3.2 Preparation of apparatus	8
8.3.3 Calibration	8
8.3.4 Determination of samples and blank solution	8
8.3.5 HPLC sequence	8
8.3.6 Typical HPLC-ICP-MS settings	9
8.4 Quality control	9
9 Calculation	9
9.1 Integration of peaks	9
9.2 Inorganic arsenic in test solutions	10
9.3 Calculation of inorganic arsenic in the samples	10
10 Precision	10
10.1 Inter-laboratory test	10
10.2 Repeatability	10
10.3 Reproducibility	10
11 Test report	11
Annex A (informative) Results of the inter-laboratory test	12
Bibliography	14

European foreword

This document (EN 17374:2020) 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 January 2021, and conflicting national standards shall be withdrawn at the latest by January 2021.

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 has been prepared under a standardization request given to CEN by the European Commission and the European Free Trade Association.

WARNING — The method described in this document 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.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This document specifies a procedure for the determination of inorganic arsenic in animal feeding stuffs by anion-exchange HPLC-ICP-MS following water bath extraction.

This method was successfully tested in the range of 0,149 mg/kg to 9,69 mg/kg in the following animal feed matrices: rice meal, seaweed meal, fish meal, grass meal, complete feed (marine-based), complete feed (cereal based) and a synthetic solution.

NOTE Mineral feed matrices are not included in the scope of this method. It is good to perform a determination of the total arsenic content in such matrices.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments).

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia. available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

4 Principle

This document describes a method for the determination of inorganic arsenic in animal feeding stuffs. Inorganic arsenic consists of arsenite, As(III) and arsenate, As(V). A representative test portion of the sample is treated with a diluted nitric acid and hydrogen peroxide solution in a heated water bath. Hereby the arsenic species are extracted into solution and As(III) is oxidized to As(V). The inorganic arsenic is selectively separated from other arsenic compounds using anion exchange HPLC (High Performance Liquid Chromatography) coupled online to the element-specific detector ICP-MS (Inductively Coupled Plasma Mass Spectrometry) for the determination of the mass fraction of inorganic arsenic. External calibration with solvent matrix-matched standards is used for quantification of the amount of inorganic arsenic.

5 Reagents

Use only reagents of recognized analytical grade and water conforming to grade 1 of EN ISO 3696:1995.

5.1 General

The concentration of arsenic species in the reagents and water used shall be low enough to not affect the results of the determination. Reagents should be of minimum p.a. quality where possible. Special facilities should be used in order to avoid contamination during the steps of preparation and measurement (e.g. laminar flow benches or comparable clean facilities).

When using a method of high sensitivity like ICP-MS, the control of the blank levels of water, acid and other reagents is very important. Generally ultra-pure water complying with grade 1 of EN ISO 3696:1995 (i.e. electrical conductivity below 0,1 $\mu\text{S}/\text{cm}$ at 25 °C) and acid of high purity, e.g. cleaned by sub-boiling distillation, are recommended.