Animal feeding stuffs: Methods of sampling and analysis - Determination of iodine in animal feed by ICP-MS



EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

		This Estonian standard EVS-EN 17050:2017 consists of the English text of the European standard EN 17050:2017.	
Standard on jõustunud avaldamisega EVS Teatajas	sellekohase teate	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 06.09.2017.		Date of Availability of the European standard is 06.09.2017.	
Standard on kätt Standardikeskusest.	esaadav Eesti	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 <u>www.evs.ee</u>; telefon 605 5050; e-post <u>info@evs.ee</u>

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

EUROPEAN STANDARD NORME EUROPÉENNE

EN 17050

EUROPÄISCHE NORM

September 2017

ICS 65.120

English Version

Animal feeding stuffs: Methods of sampling and analysis - Determination of iodine in animal feed by ICP-MS

Aliments pour animaux - Méthodes d'échantillonnage et d'analyse - Dosage de l'iode dans les aliments pour animaux par spectrométrie de masse à plasma induit par haute fréquence (ICP-MS) Futtermittel - Probenahme- und Untersuchungsverfahren - Bestimmung von Iod in Futtermitteln mittels Anionenaustausch ICP-MS

This European Standard was approved by CEN on 28 June 2017.

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, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, 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: Avenue Marnix 17, B-1000 Brussels

Cont	tents	Page
Europ	oean foreword	4
1	Scope	5
2	Normative references	
3	Principle	
4	Reagents	
+ 4.1	General	
4.2	Tetra methyl ammonium hydroxide (TMAH (CH ₃) ₄ N+OH-) solution	
4.3	Diluted tetra methyl ammonium hydroxide (TMAH) solution	
4.4	Stock solutions	
4.4.1	General	
4.4.2	Iodine stock solution (KIO ₃); $\rho = 1000$ mg/l, purity: mass fraction w > 99,5 %	
4.4.3	Tellurium stock solution, $\rho = 1~000$ mg/l	
4.5	Standard solutions	
4.5.1	Iodine standard solution, ρ = 10 mg/l	6
4.5.2	Tellurium standard solution (internal standard)	
4.6	Iodine calibration solutions	
4.6.1	General	
4.6.2	Calibration solution 1 - ρ = 5 μ g/l	
4.6.3	Calibration solution $2 - \rho = 20 \mu g/l$	
4.6.4	Calibration solution $3 - \rho = 50 \mu\text{g}/1$	7
4.7	Zero member compensation solution	
5	Apparatus and equipment	
5.1	General	
5.2	Containers	
5.3	Plastic syringes	
5.4	Membrane filters	
5.5	Ultracentrifuge	
5.6	Drying oven	8
5.7	Inductively coupled plasma mass spectrometer (ICP-MS)Tubes	8 0
5.8		
6	Sampling	
6.1	General	
6.2	Sampling procedure	8
7	Procedure	8
7.1	Sample preparation	
7.2	Iodine extraction	
7.3	Preparation of sample and blank solutions	9
7.4	Determination of iodine by ICP-MS	
7.4.1	General	
7.4.2	Preparation of apparatus	
7.4.3	Calibration	
7.4.4	Determination	10
8	Calculation	10

9	Limit of quantification (LOQ)	10
10	Precision	10
10.1	Inter-laboratory test	10
10.2	Repeatability	
10.3	Reproducibility	
11	Test report	
Annex	x A (informative) Results of the inter-laboratory test	12
Biblio	ography	15
	ography	
	9,	
	\sim	
	O,	
	4	
	· (O)	
		J

European foreword

This document (EN 17050:2017) 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 March 2018, and conflicting national standards shall be withdrawn at the latest by March 2018.

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.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

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.

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, Former Yugoslava Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Iteland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This European standard specifies a method for the determination of iodine in animal feeding stuffs by inductively coupled plasma mass spectrometry (ICP-MS) following extraction with an alkaline solution.

This method was successfully tested in the range of 0,70 to 631 mg/kg in following animal feeds: seaweed meal, mineral premixture, fish meal, plant based ingredient, marine based compound feed and a synthetic iodine solution.

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 15111, Foodstuffs - Determination of trace elements - Determination of iodine by ICP-MS (inductively coupled plasma mass spectrometry)

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

EN ISO 6497, Animal feeding stuffs - Sampling (ISO 6497)

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

3 Principle

This standard describes a method for the determination of iodine in animal feeding stuffs. A representative test portion of the sample is treated with a strong alkaline solution of tetra methyl ammonium hydroxide (TMAH) and incubated at 90°C for three hours. Hereby iodine is extracted into solution and is determined by use of inductively coupled plasma mass spectrometry (ICP-MS).

The method principles are the same as used in EN 15111.

WARNING — The 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.

4 Reagents

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

4.1 General

The concentration of iodine in the reagents and water used shall be low enough not to affect the results of the determination.

NOTE Different qualities of tetra methyl ammonium hydroxide (TMAH) are available and it is important to check that the iodine content is sufficiently low and does not affect the analysis.

4.2 Tetra methyl ammonium hydroxide (TMAH (CH₃)₄N+OH⁻) solution

Use mass concentration ρ = 250 g/l, (mass fraction w = 25 %), suitable for trace analysis with an iodine content of less than 1 μ g/l.