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Djurfoder – Metoder för provtagning och analys – Bestämning av jod med jonbyteskroatografi-ICP-MS i djurfoder

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

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The European Standard EN 17050:2017 has the status of a Swedish Standard. This document contains the official version of EN 17050:2017.

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Denna standard är framtagen av kommittén för Livsmedel och foder, SIS/TK 435.

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 17050

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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.

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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.

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

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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of an identical text or by endorsement, at the latest by March 2018, and conflicting national standards shall be withdrawn at the latest by March 2018.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 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 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 $(CH_3)_4N+OH^-$) solution

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

4.3 Diluted tetra methyl ammonium hydroxide (TMAH) solution

Dilute TMAH solution for preparing the zero member compensation and calibration solutions with a concentration to suit that of the sample solution (see 7.3). Prepare a 0,5 % TMAH solution by diluting e.g. 1,0 ml of TMAH solution (4.2) to 50 ml with water.

4.4 Stock solutions

4.4.1 General

Commercially available standard stock solutions of iodine and tellurium are recommended. As an alternative the solutions described below may be used.

4.4.2 Iodine stock solution (KIO_3); ρ = 1 000 mg/l, purity: mass fraction w > 99,5 %

Dissolve 1,686 4 g of potassium iodate in water and dilute to 1 000 ml with water.

4.4.3 Tellurium stock solution, ρ = 1 000 mg/l

Dissolve 1,250 8 g of tellurium dioxide (TeO_2) in 4 mol/l hydrochloric acid (HCl) and dilute to 1 000 ml with water.

4.5 Standard solutions

4.5.1 Iodine standard solution, ρ = 10 mg/l

Pipette 1 ml of the iodine stock solution (4.4.2) into a 100 ml volumetric flask and dilute to the mark with water. This solution is stable for about four weeks and is used to prepare the calibration solutions in 4.6.

4.5.2 Tellurium standard solution (internal standard)

Prepare e.g. a 10 mg/l standard tellurium solution by pipetting 1 ml of the tellurium stock solution (4.4.3) into a 100 ml volumetric flask and dilute to the mark with water. This solution is stable for about four weeks.

Tellurium is satisfactory as an internal standard for determining iodine since it has a mass in a comparable range and ionization energy similar to that of iodine. The original tellurium content in the sample to be analysed shall be negligible. If that is not the case, another suitable internal standard shall be used (e.g. rhodium (Rh)).

4.6 Iodine calibration solutions

4.6.1 General

The concentrations of the calibration solutions specified below are given as example and may be modified to suit the sensitivity of the apparatus and the concentration range to be covered for the analysis in question. The linear range of the detector system should not be exceeded.

The internal standard added to the calibration solutions shall have a concentration high enough to reach a stable detector count rate. The calibration, zero member compensation and sample solutions shall contain exactly the same amount of internal standard. The internal standard may be added online. The TMAH concentrations in the calibration solutions shall be approximately equal to that of the sample solutions. The calibration solutions shall be prepared freshly every working day.

4.6.2 Calibration solution 1 - ρ = 5 µg/l

Fill a 50 ml volumetric flask with about 30 ml of diluted TMAH solution (4.3). Pipette 2,5 ml of tellurium standard solution (4.5.2) into the flask and mix. Add 25 µl of iodine standard solution (4.5.1) to this mixture and fill to the mark with diluted TMAH solution (4.3).

4.6.3 Calibration solution 2 - ρ = 20 µg/l

Fill a 50 ml volumetric flask with about 30 ml of diluted TMAH solution (4.3). Pipette 2,5 ml of tellurium standard solution (4.5.2) into the flask and mix. Add 100 µl of iodine standard solution (4.5.1) to this mixture and fill to the mark with diluted TMAH solution (4.3).

4.6.4 Calibration solution 3 - ρ = 50 µg/l

Fill a 50 ml volumetric flask with about 30 ml of diluted TMAH solution (4.3). Pipette 2,5 ml of tellurium standard solution (4.5.2) into the flask and mix. Add 250 µl of iodine standard solution (4.5.1) to this mixture and fill to the mark with diluted TMAH solution (4.3).

4.7 Zero member compensation solution

The zero member compensation solution contains water and the same amount of TMAH and internal standard as the sample solution. Fill a 50 ml volumetric flask with about 30 ml of diluted TMAH solution (4.3). Pipette 2,5 ml of tellurium standard solution (4.5.2) into the flask, mix and fill to the mark with diluted TMAH solution (4.3).

5 Apparatus and equipment

5.1 General

To minimize the blank, all apparatus that comes into direct contact with the sample and the solutions used shall be carefully pre-treated with a diluted TMAH solution (e.g. as in 4.3) and then rinsed with water.

5.2 Containers

Containers shall be gastight sealable, glass or quartz, of capacity 30 ml to 100 ml, e.g. screw-thread Erlenmeyer flask, 100 ml with plastic screw caps and PTFE-protected seal.

As alternative, plastic containers that can be gas-tightly sealed and are able to withstand a temperature of not less than 110 °C (e.g. made of polypropylene, high-density polyethylene or polyfluorine (such as PFA)) may be used.

NOTE If plastic vessels are repeatedly used, there is a risk of higher blanks, in particular if samples having elevated iodine contents are extracted in them.

5.3 Plastic syringes

Plastic syringes of capacity 5 ml to 25 ml, preferably with a bayonet-like connection (e.g. Luer lock).

5.4 Membrane filters

Membrane filters should be used as dispensable syringe attachment.

— Membrane filter, having a pore size of 5 µm, with a connection fitting the syringes in 5.3, not necessary if ultracentrifuge (5.5) is used;

— Membrane filter, having a pore size of 0,45 µm or less, with a connection fitting the syringes in 5.3. Membrane filters with included pre-filters may be used as well.