SLOVENSKI STANDARD
SIST EN 15111:2007

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SIST-TS CEN/TS 15111:2005

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

Lebensmittel - Bestimmung von Elementspuren - Bestimmung von Iod mit der ICP-MS (Massenspektrometrie mit induktiv gekoppeltem Plasma)

Produits alimentaires - Dosage des éléments traces - Dosage de l'iode par spectrométrie d'émission avec plasma induit par haute fréquence et spectrometre de masse (ICP-SM)

Ta slovenski standard je istoveten z: EN 15111:2007

<u>**ICS:**</u>

67.050	Splošne preskusne in analizne metode za živilske proizvode	General methods of tests and analysis for food products

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 15111

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English Version

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

Produits alimentaires - Dosage des éléments traces - Dosage de l'iode par spectrométrie d'émission avec plasma induit par haute fréquence et spectromètre de masse (ICP-SM)

Lebensmittel - Bestimmung von Elementspuren - Bestimmung von Iod mit der ICP-MS (Massenspektrometrie mit induktiv gekoppeltem Plasma)

This European Standard was approved by CEN on 21 January 2007.

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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 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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Contents

Foreword

This document (EN 15111:2007) has been prepared by Technical Committee CEN/TC 275 "Food analysis - Horizontal methods", the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by September 2007, and conflicting national standards shall be withdrawn at the latest by September 2007.

This document supersedes CEN/TS 15111:2005.

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

1 Scope

This European Standard specifies an extraction method for the determination of iodine compounds in foodstuffs by inductively coupled plasma mass spectrometry (ICP-MS).

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 13804, *Foodstuffs — Determination of trace elements — Performance criteria, general considerations and sample preparation*

3 Principle

Iodine compounds are extracted with a strong alkaline reagent at elevated temperature. After removing un-dissolved components, the nebulized solution is atomized and ionized in an inductively coupled argon plasma. The ions are extracted from the plasma by a system of sampler and skimmer cones, separated in a mass spectrometer on the basis of their mass/charge ratio and determined using a pulse counting detector system.

WARNING — The use of this European Standard may involve hazardous materials, operations and equipment. This European 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 or regulatory limitations prior to use.

4 Reagents

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, e.g. different qualities of TMAH are available. It is important to check, that the iodine content is low enough for the analysis.

4.2 Tetramethylammonium hydroxide (TMAH = $(CH_3)_4N^+OH^-$) solution,

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 tetramethylammonium 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 1,0 ml of TMAH solution (4.2) to 50 ml with water.

4.4 Stock solutions

4.4.1 General

Commercial stock solutions may be used as an alternative to the solutions described below.

4.4.2   Iodine stock solution (KIO₃), 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 l 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 and dilute to 1 l 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)

Tellurium has proved satisfactory as an internal standard for determining iodine since it has a mass in a comparable range and an 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.

Prepare e.g. a 10 mg/l standard tellurium solution by pipetting 1 ml 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.

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. The linear range of the detector system shall 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 TMAH concentrations in the reference solutions shall be approximately equal to that of the sample solution.

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).

The calibration solutions shall be prepared freshly every day.

4.7   Zero member compensation solution, containing water and the same amount of TMAH and internal standard as the sample solution.

E.g. 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   Vessels, 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 an alternative, plastic vessels that can be gastightly 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 fairly high iodine contents are extracted in them.

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

5.4   Ultracentrifuge, as an alternative to 5.3, having an acceleration of not less than 10 000 g and equipped with suitable rotors or adapters for accommodating vessels that can be sealed gastight.

5.5   Membrane filters, as dispensable syringe attachment.

5.5.1   Membrane filter, having a pore size of 5 µm, with a connection fitting the syringes in 5.3, not necessary if ultracentrifuge (5.4) is used

5.5.2   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.

NOTE   Membrane filters sometimes give different blanks depending on the filter manufacturer and the batch number.

5.6   Drying oven, capable of being maintained at temperatures of (90 ± 3) °C.

5.7   ICP - mass spectrometer (ICP-MS), with inductively coupled plasma as ionization unit, quartz burner, nebulizing chamber, nebulizer, sample feed device and optionally an automatic sampler.

5.8   Vessels, for automatic sampler.

6   Sampling

6.1   General

The conditions for sampling as outlined in EN 13804 have to be followed.

6.2   Sampling procedure

To prevent any change in the iodine content between sample collection and analysis, the sample shall be stored in a tightly sealable vessel or in the original vessel and refrigerated, avoiding prolonged contact with air and exposure to light.

7   Procedure

7.1   Sample preparation

Homogenize the sample using suitable equipment and avoiding excessive heating. The particle size of non-soluble dry samples should be below 0,3 mm.

7.2   Iodine extraction

Weigh to the nearest 1 mg, approximately 100 mg to 500 mg of sample (7.1) (calculated as dry matter) into a vessel as in 5.2, add 5 ml of water and mix thoroughly to prevent any agglomeration. Add 1 ml of TMAH solution (4.2), mix thoroughly, seal the vessel tightly and place it in a drying oven preheated to (90 ± 3) °C for 3 h. After cooling, transfer the contents quantitatively to a 25 ml volumetric flask and dilute to the mark with water. For removal of coarse particles filter an aliquot through a 5 μm membrane filter, discarding the first 3 to 5 drops. The particles may also be removed by centrifuging (5.4) at 10 000 g for not less than 15 min. This procedure needs to be done only if the sample tends to clog the 0,45 μm filter very fast.

Transfer the filtrate or the supernatant to a plastic syringe and pass it through a 0,45 μm filter, discarding the first 3 to 5 drops of filtrate. The extract contains 1 % TMAH. The extraction solution should be measured within one day.

To check the method blank, perform all the extraction steps as in 7.2 with all the reagents without any sample.

NOTE   The filtrate can be cloudy in the case of foods containing starch. Some foodstuffs e.g. algae contain swelling substances making filtration very difficult. In these cases membrane filters with included pre-filters should be used. Some samples tend to foam making fill up to 25 ml difficult.

7.3   Preparation of sample and blank solutions

Add internal standard (4.5.2) to an aliquot of the extract obtained in 7.2 ensuring that the TMAH concentration of the sample solutions is the same as that of the zero member compensation and calibration solutions.

Prepare a sample solution e.g. by pipetting 500 μl of tellurium standard solution (4.5.2) into a 10 ml volumetric flask, add 5 ml extract (7.2), mix and fill up with water (dilution factor = 2).