SLOVENSKI STANDARD
SIST-TS CEN/TS 15111:2005

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Živila - Določevanje elementov v sledovih - Določevanje joda v dietetični hrani z ICP-MS (masno spektrometrijo z induktivno sklopljeno plazmo)

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

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

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

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English Version

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

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Foreword

This Technical Specification (CEN/TS 15111:2005) has been prepared by Technical Committee CEN/TC 275 “Food analysis - Horizontal methods”, the secretariat of which is held by DIN.

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1 Scope

This Technical Specification specifies a method for the determination of added inorganic iodine compounds, including water-soluble iodine compounds of natural origin, in dietetic foods 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, added to dietetic foods, are extracted with a strong alkaline reagent at elevated temperature. After the removing of undissolved 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.

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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 determination.

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

mass concentration c = 250 g/l, 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 test solution (see 7.3).

Prepare e.g. 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 (KIO_3), c = 1 000 mg/l

Dissolve 1,6864 g of potassium iodate in water and dilute to 1 l.

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

Dissolve 1,2508 g of tellurium dioxide (TeO_2) in 4 mol/l hydrochloric acid and dilute to 1 l.

4.5 Standard solutions

4.5.1 Iodine standard solution, c = 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 reference 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, but 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 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 reference solutions

4.6.1 General

The reference solution concentrations specified below are given as an 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 TMAH concentrations in the reference solutions shall be approximately equal to that of the test solution.

4.6.2 Reference solution 1, c = 10 µg/l

Pipette 25 µl of standard solution (4.5.1) into a 25 ml volumetric flask and dilute to the mark with diluted TMAH solution (4.3).

4.6.3 Reference solution 2, c = 40 µg/l

Pipette 100 µl of standard solution (4.5.1) into a 25 ml volumetric flask and dilute to the mark with diluted TMAH solution (4.3).

4.6.4 Reference solution 3, c = 200 µg/l

Pipette 500 µl of standard solution (4.5.1) into a 25 ml volumetric flask and dilute to the mark with diluted TMAH solution (4.3).

The reference solutions shall be prepared freshly every day.

4.7 Iodine calibration solutions

4.7.1 General

To calibrate the analytical system, the internal standard shall be added to the reference solutions (4.6) in a concentration high enough to reach a stable detector count rate. The calibration, zero member compensation and test solutions shall contain the same amount of internal standard. The following solutions are recommended.

4.7.2 Calibration solution 1

Pipette 0,5 ml of standard tellurium solution (4.5.2) into a vessel (5.8), add 10 ml of iodine reference solution (4.6.1) and mix.

4.7.3 Calibration solution 2

Pipette 0,5 ml of standard tellurium solution (4.5.2) into a vessel (5.8), add 10 ml of iodine reference solution (4.6.2) and mix.

4.7.4 Calibration solution 3

Pipette 0,5 ml of tellurium standard solution (4.5.2) into a vessel (5.8), add 10 ml of iodine reference solution (4.6.3) and mix.

The calibration solutions shall be prepared freshly every day.

4.8 Zero member compensation solution, containing water and the same amount of TMAH and internal standard as the test solution.Pipette e.g. 0,5 ml of tellurium standard solution (4.5.2) into a vessel (5.9), and mix with 10 ml of 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 pretreated with a diluted TMAH solution (e.g. as in 4.3) and then rinsed with water.

5.2 Vessels, gastight, sealable quartz or glass, of capacity 30 ml to 100 ml.

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) and a membrane filter, as a dispensable syringe attachment, having a pore size of 5,0 µm, with a connection fitting the syringe.

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 filter, as dispensable syringe attachment, having a pore size of 0,45 µm, with a connection fitting the syringes in 5.3.

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 test solution or 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.

7.2 Iodine extraction

Weigh to the nearest 3 mg, approximately 200 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 (4.3), 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. Filter an aliquot through a 5 µm membrane filter, discarding the first 0,5 ml. The particles may also be removed by centrifuging (5.4) at 10 000 *g* for not less than 15 min.

Transfer the filtrate or the supernatant to a plastic syringe and pass it through a 0,45 µm filter, discarding the first 0,5 ml of filtrate. The extract contains 1 % TMAH.

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

NOTE The filtrate can be cloudy in the case of foods containing starch.

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 test solutions is the same as that of the zero member compensation and calibration solutions.

Prepare a sample solution e.g. by pipetting 0,5 ml of standard tellurium solution (4.5.2) into a vessel (5.8), add 5 ml of extract (7.2) and 5 ml of water and mix.