SLOVENSKI STANDARD
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Sadje, zelenjava in sadni ter zelenjavni proizvodi - Določevanje kadmija - 1. del: Metoda z atomsko absorpcijo z grafitno kiveto

Fruits, vegetables and derived products -- Determination of cadmium content -- Part 1: Method using graphite furnace atomic absorption spectrometry

Fruits, légumes et produits dérivés -- Détermination de la teneur en cadmium -- Partie 1: Méthode par spectrométrie d'absorption atomique avec four en graphite

Ta slovenski standard je istoveten z: **ISO 6561-1:2005**

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67.080.01	Sadje, zelenjava in njuni proizvodi na splošno	Fruits, vegetables and derived products in general

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INTERNATIONAL STANDARD

ISO 6561-1

First edition
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Fruits, vegetables and derived products — Determination of cadmium content —

Part 1: Method using graphite furnace atomic absorption spectrometry

Fruits, légumes et produits dérivés — Détermination de la teneur en cadmium —

Partie 1: Méthode par spectrométrie d'absorption atomique avec four en graphite

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 6561-1 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 3, *Fruit and vegetable products*.

This first edition of ISO 6561-1, together with ISO 6561-2:2004, cancels and replaces ISO 6561:1983, which has been technically revised.

ISO 6561 consists of the following parts, under the general title *Fruits, vegetables and derived products — Determination of cadmium content*:

— *Part 1: Method using graphite furnace atomic absorption spectrometry*

— *Part 2: Method using flame atomic absorption spectrometry*

Fruits, vegetables and derived products — Determination of cadmium content —

Part 1: Method using graphite furnace atomic absorption spectrometry

1 Scope

This part of ISO 6561 specifies a graphite furnace atomic absorption spectrometric method for the determination of the cadmium content of fruits, vegetables and derived products.

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.

ISO 5515:1979, *Fruits, vegetables and derived products — Decomposition of organic matter prior to analysis — Wet method*

3 Principle

Organic matter is decomposed by a wet method and the dissolved cadmium is determined by graphite furnace atomic absorption spectrometry.

4 Reagents

Use only reagents of recognized analytical grade, and which, with the exception of the cadmium sulfate hydrate (4.8) and the cadmium standard solutions (4.9 and 4.10), shall be free from cadmium. Use only water which has been double-distilled in borosilicate glass apparatus, or water of at least equivalent purity.

4.1 Sulfuric acid, concentrated, ρ_{20} = 1,84 g/ml.

4.2 Nitric acid, ρ_{20} = 1,38 g/ml.

4.3 Perchloric acid, ρ_{20} = 1,67 g/ml.

4.4 Sulfuric acid, dilute, 10 % (volume fraction).

4.5 EDTA (ethylenediaminetetraacetic acid, disodium salt), 0,20 mol/l solution.

4.6 Buffer solution, pH 9.

Dissolve 5,4 g of ammonium chloride in water and transfer to a 100 ml one-mark volumetric flask. Add 35 ml of 25 % (volume fraction) ammonia solution and make up to the mark with water.

4.7 Eriochrome black T, 1 % (mass fraction) mixture with sodium chloride.

4.8 Cadmium sulfate hydrate ($3CdSO_4 \cdot 8H_2O$).

The titre of the cadmium sulfate shall be verified as follows.

Weigh exactly 102,6 mg of cadmium sulfate hydrate, transfer quantitatively to a conical flask with water and shake until dissolved. Add 5 ml of the buffer solution (4.6) and about 20 mg of the eriochrome black T mixture (4.7). Titrate with the EDTA solution (4.5) until the end point is reached as indicated by a change of colour to blue.

The volume of EDTA used shall be 20 ml. If the volume differs slightly, correct the mass of cadmium sulfate used to prepare the standard cadmium solution (4.9) accordingly.

4.9 Cadmium standard solution, corresponding to 1,0 mg of cadmium per mililitre.

4.10 Cadmium standard solution, containing 0,05 mg of cadmium per litre.

Transfer, by means of a pipette, 10 ml of the cadmium standard solution (4.9) to 1 000 ml one-mark volumetric flask and dilute to the mark with water. Transfer 5 ml of this solution to another 1 000 ml one-mark volumetric flask and dilute to the mark with the dilute sulfuric acid (4.4).

1 ml of this standard solution contains 0,05 µg of cadmium.

5 Apparatus

The glassware used shall be washed beforehand with hot concentrated nitric acid and rinsed with water.

Usual laboratory apparatus and, in particular, the following.

5.1 Round-bottom flasks, of capacity 1 000 ml.

5.2 One-mark volumetric flasks, of capacity 50 ml.

5.3 One-mark pipettes or **graduated pipettes**, of appropriate capacities.

5.4 Funnels and **ashless filter papers**.

5.5 Conical flask.

5.6 Burette, of capacity 25 ml, graduated in 0,1 ml divisions.

5.7 Atomic absorption spectrometer, with a graphite furnace, a background corrector, a multipotentiometric recorder and a hollow-cathode cadmium lamp, suitable for measurements at a wavelength of 228,8 nm.

5.8 Eppendorf micropipettes, of capacities 5 µl, 10 µl, 20 µl, 25 µl and 50 µl, having standard colourless Eppendorf tips.

Some Eppendorf micropipettes are inaccurate by 10 % or more. Unless they have been especially calibrated for this procedure, it is recommended that the same pipette be used with the test solution, blank test solution and calibration solutions.

5.9 Analytical balance.

5.10 Mechanical grinder, the internal lining and blades of which are of polytetrafluoroethylene (PTFE).

6 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this part of ISO 6561. If there is no specific International Standard dealing with the product concerned, it is recommended that the parties concerned come to an agreement on the subject.

7 Procedure

7.1 Preparation of the test sample

Mix the laboratory sample well. If necessary, first remove stones, stalks and hard seed-cavity walls and pass the laboratory sample through the mechanical grinder (5.10).

Allow frozen or deep-frozen products to thaw in a closed vessel and add the liquid formed during this process to the product before mixing.

7.2 Test portion

7.2.1 Liquid products

Take, by means of pipette, 10 ml of the test sample (7.1).

It is also possible to take the test portion by mass by weighing a quantity of the test sample to the nearest 0,01 g.

7.2.2 Semi-solid and solid products

Weigh, to the nearest 0,01 g, a quantity of the test sample (7.1) corresponding to approximately 10 g of product.

7.3 Decomposition

Transfer the test portion (7.2) to a round-bottom flask (5.1). If the test portion is liquid (7.2.1) and contains ethanol, first eliminate ethanol by boiling and then allow to cool. Add 10 ml of nitric acid (4.2), heat and then carefully add 5 ml of concentrated sulfuric acid (4.1) Proceed as described in ISO 5515:1979, 6.3.1, from the second paragraph to the eighth paragraph.

When decomposition is complete, filter the sample solution, diluted with a few millilitres of water, through an ashless filter paper (5.4) that has been previously rinsed with hydrochloric acid and water. Collect the filtrate in a 50 ml one-mark volumetric flask (5.2), rinsing the round-bottom flask (5.1) and the filter paper with a few millilitres of water and collecting the rinsing in the same volumetric flask. Shake, allow to cool, and dilute to the mark. Mix by shaking.

7.4 Blank test

Carry out a blank test by repeating the decomposition (7.3), replacing the test portion by 10 ml of water.