SLOVENSKI STANDARD
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Sadje, zelenjava in sadni ter zelenjavni proizvodi - Določevanje kadmija - 2. del: Metoda s plamensko atomsko absorpcijo

Fruits, vegetables and derived products -- Determination of cadmium content -- Part 2: Method using flame atomic absorption spectrometry

Fruits, légumes et produits dérivés -- Détermination de la teneur en cadmium -- Partie 2: Méthode par spectrométrie d'absorption atomique avec flamme

Ta slovenski standard je istoveten z: **ISO 6561-2:2005**

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ISO 6561-2

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

Part 2:
Method using flame atomic absorption spectrometry

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

Partie 2: Méthode par spectrométrie d'absorption atomique avec flamme

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Foreword

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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 6561-2 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 3, *Fruit and vegetable products*.

This first edition of ISO 6561-2, together with ISO 6561-1:2004, 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 2:
Method using flame atomic absorption spectrometry

1 Scope

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

NOTE The method of cadmium determination in fruit, vegetables and derived products is based on AOAC Official Methods of Analysis [1].

2 Principle

This method is based on the decomposition of organic matter with HNO_3, H_2SO_4 and H_2O_2, extraction of cadmium by dithizone-$CHCl_3$ at pH 9, and determination of cadmium by flame atomic absorption spectrometry.

3 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified, and water which has been distilled twice in borosilicate glass apparatus, or water of at least equivalent purity.

3.1 Nitric acid, concentrated (ρ_{20} = 1,38 g/ml).

3.2 Sulfuric acid, concentrated (ρ_{20} = 1,84 g/ml).

3.3 Hydrochloric acid, dilute, 0,2 mol/l.

Place 16,5 ml of concentrated hydrochloric acid (ρ_{20} = 1,19 g/ml) into a 1 000 ml one-mark volumetric flask and dilute to the mark with water. Mix.

3.4 Hydrogen peroxide, concentrated (50 %).

3.5 Citric acid monohydrate.

3.6 Sodium hydroxide, 0,05 mol/l solution.

3.7 Thymol blue indicator.

Triturate 0,1 g of thymol blue indicator in an agate mortar with 4,3 ml of sodium hydroxide solution (3.6). Dilute to 200 ml in a flask with water.

3.8 Ammonia solution, 28 % to 30 % solution.

3.9 Chloroform.

3.10 Dithizone, 1,0 mg/ml solution.

Place 0,2 g of dithizone in a 200 ml volumetric flask and add chloroform to the mark.

3.11 Dithizone, 0,2 mg/ml solution.

Dilute dithizone solution (3.10) 1 + 4 with chloroform (3.9). Prepare fresh daily.

3.12 Cadmium standard solution, corresponding to a cadmium concentration of 1,0 mg/ml.

4 Apparatus

Before use, wash all glassware with 8 mol/l nitric acid, followed by a thorough rinse with water.

Cover beakers with watch glasses during all operations.

Usual laboratory apparatus and, in particular, the following.

4.1 Mechanical grinder, the inside and the blades of which are coated with polytetrafluoroethylene (PTFE).

4.2 Round-bottom flasks, of 1 500 ml capacity.

4.3 Beaker, of 400 ml capacity.

4.4 One-mark volumetric flasks, of capacities 50 ml, 100 ml and 1 000 ml.

4.5 Pipettes, of appropriate capacity.

4.6 Separators, 125 ml and 250 ml capacities.

4.7 Atomic absorption spectrometer, provided with an air/acetylene burner (10 cm), suitable for measurements at a wavelength of 228,8 nm.

4.8 Burner or **heating mantle**.

4.9 Hot plate.

4.10 Analytical balance.

5 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.

6 Procedure

6.1 Preparation of test sample

Mix well the laboratory sample, if necessary, first remove any seeds, stalks and hard seed-cavity walls, and then grind in the mechanical grinder (4.1).

Frozen or deep-frozen products shall be previously thawed in a closed vessel, and the liquid formed during this process shall be added to the product before mixing.

6.2 Test portion

Weigh, to the nearest 0,01 g, 50 g of the test sample (6.1) into a 1 500 ml round-bottom flask (4.2).

6.3 Decomposition

If the test portion contains ethanol, first remove the ethanol by evaporation.

Add several boiling chips or beads and carefully add 25 ml of nitric acid (3.1). Cover and warm gently using a burner or heating mantle (4.8) to initiate reaction.

When reaction subsides, add 25 ml of nitric acid (3.1), warm again and continue until 100 ml of nitric acid (3.1) has been added. Alternatively, add carefully 100 ml of nitric acid (3.1) to the test portion at once, and allow it to stand at room temperature overnight.

Heat until most NO fumes have evolved; control excessive frothing by cooling or quenching with water from a washbottle.

Add 20 ml of concentrated sulfuric acid (3.2) to the solution. Dilute to approximately 300 ml with water and evaporate using a burner or heating mantle (4.8) until charring begins. When charring becomes extensive, cautiously add hydrogen peroxide (3.4), 1 ml at a time. Let the reaction subside before adding the next portion of oxidant, and never add more than 1 ml at a time. Continue the addition of hydrogen peroxide until the solution is colourless.

Heat vigorously to SO_3 fumes, adding more hydrogen peroxide (3.4) as required, to remove char. Heat vigorously to expel excess of hydrogen peroxide. Cool colourless digest to room temperature.

6.4 Blank test

Carry out a blank test, using the same decomposition procedure as in 6.3, but replacing the test portion with a suitable amount of water corresponding to the amount of test portion taken for analysis (6.2).

6.5 Extraction

WARNING — The method described in this subclause requires the use of chloroform, a toxic and ozone-depleting substance. Avoid inhalation of and exposure to this solvent. Work in a fume cupboard when handling this solvent and solutions thereof. Dispose of the waste chloroform and solutions properly.

Add 2 g of citric acid (3.5) to cooled digest of a sample or blank and cautiously dilute to about 25 ml with water. Add 1 ml of thymol blue indicator (3.7) and adjust to pH 8,8 by slowly adding ammonia solution (3.8), while cooling in ice bath, until the solution changes from yellowish green to greenish blue. Transfer quantitatively to a 250 ml separator (4.6) with water, and dilute to about 150 ml.

Cool the solution, and extract with two 5-ml portions of concentrated dithizone solution (3.10), shaking 1 min to 2 min each time. Continue extraction with 5-ml portions of dilute dithizone solution (3.11) until the dithizone extract shows no change in colour. Combine the dithizone extracts in the 125 ml separator (4.6); wash with