INTERNATIONAL STANDARD

ISO 6561-1

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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

Reference number
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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.

7.5 Determination

7.5.1 Furnace programme

The furnace shall allow four independent thermal stages:

a) drying the solution;

b) thermal decomposition;

c) atomization;

d) increase to maximum temperature to purge the furnace.

The proposed conditions are as follows:

— drying at 100 °C for 25 s;

— instantaneous change to the stage of thermal decomposition at 450 °C for 30 s;

— atomization in 7 s at 1 900 °C, after a progressive increase to this temperature. During this stage, the apparatus shall record the maximum absorbance and the variation in absorbance. The duration over which atoms exist in the furnace may be increased by decreasing the rate of circulation of nitrogen (“mini flow”) or by stopping it altogether (“gas stop”);

— increase to the maximum temperature (2 700 °C) to purge the furnace with nitrogen at the end of the procedure.

7.5.2 Preparation of the calibration graph

Prepare calibration solutions having cadmium concentrations of 5 µg/l, 10 µg/l and 20 µg/l by suitably diluting the cadmium standard solution (4.10). Inject successively into the furnace, programmed in accordance with 7.5.1, by means of micropipette (5.8) fitted with a tip, 50 µl of each of these calibration solutions. Determine the absorbance from the heights of the peaks registered. Calculate the mean value of the absorbance from the results of three injections into the furnace. The absorbances thus determined correspond, respectively, to 0,000 25 µg, 0,000 5 µg and 0,001 µg of cadmium. Plot a calibration graph having, for example, the values of absorbances as ordinates and the corresponding cadmium concentrations as abscissae.

7.5.3 Determination of test solution

Inject successively into the furnace, programmed in accordance with 7.5.1, by means of a micropipette (5.8) fitted with a tip, three times the adequate volume of the decomposed sample solution obtained in 7.3. Note the corresponding absorbances. Calculate the mean value of the absorbance and, from the calibration graph, read the quantity of cadmium contained in the 50 µl of injected test solution.

7.5.4 Determination of the blank test solution

Inject successively into the furnace, programmed in accordance with 7.5.1, by means of a micropipette (5.8) fitted with a tip, three times the adequate volume of the blank test solution (7.4). The absorbance shall be zero or less than 0,005. If necessary, subtract the mean of the three values of absorbance of the blank test solution from the mean absorbance of the test solution determined in 7.5.3 before referring to the calibration graph (7.5.2) to obtain the cadmium content of the solution.

8 Expression of the results

8.1 Method of calculation and equations

8.1.1 Liquid products

The cadmium content of the sample, expressed in milligrams per litre of product, is equal to

$$m_1 \times 100 \tag{1}$$

where m_1 is the mass, in micrograms, of cadmium contained in the injected volume of the test portion, read from the calibration graph.

8.1.2 Semi-solid and solid products

The cadmium content of the sample, expressed in milligrams per kilogram of product, is equal to

$$\frac{m_1 \times 1\,000}{m_0} \tag{2}$$

where

m_0 is the mass, in grams, of the test portion (7.2.2);

m_1 is the mass, in micrograms, of cadmium contained in the injected volume of the test portion, read from the calibration graph.

8.2 Other method of expression of results

To express the cadmium content on the dry basis, modify the equations accordingly.

9 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases be greater than 10 % of the arithmetic mean of the two results.

10 Test report

The test report shall specify:

a) all information necessary for the complete identification of the sample;

b) the sampling method used, if known;

c) the test method used, with reference to this part of ISO 6561;

d) all operating details not specified in this part of ISO 6561, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

e) the test result(s) obtained, or, if the repeatability has been checked, the final quoted result obtained.