International Standard 6561

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Fruits, vegetables and derived products — Determination of cadmium content — Flameless atomic absorption spectrometric method

Fruits, légumes et produits dérivés — Détermination de la teneur en cadmium — Méthode par spectrométrie d'absorption atomique sans flamme

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Foreword

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It has been approved by the member bodies of the following countries :

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The member body of the following country expressed disapproval of the document on technical grounds :

Australia

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Fruits, vegetables and derived products — Determination of cadmium content — Flameless atomic absorption spectrometric method

1 Scope and field of application

This International Standard specifies a flameless atomic absorption spectrometric method for the determination of the cadmium content of fruits, vegetables and derived products.

2 Reference

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

3 Principle

Decomposition of organic matter by the wet method and determination of dissolved cadmium by flameless atomic absorption spectrometry.

4 Reagents

All reagents shall be of recognized analytical quality, and, with the exception of the cadmium sulfate (4.8) and the standard cadmium solutions (4.9 and 4.10), shall be free from cadmium. The water used shall be water which has been double-distilled in an apparatus of borosilicate glass, or water of equivalent purity.

4.1 Sulfuric acid (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, 10 % (V/V) solution.

4.5 EDTA (ethylenediaminetetraacetic acid, disodium salt), 0,020 0 mol/l solution.

4.6 Buffer solution, pH 9.

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

4.7 Eriochrome black T, 1 % (m/m) mixture with sodium chloride.

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

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

Weigh exactly 102,6 mg of the cadmium sulfate, 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 20,00 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 g of cadmium per litre.

Dissolve 2,282 0 g of the cadmium sulfate (4.8) in water. Transfer quantitatively to a 1 000 ml one-mark volumetric flask and dilute to the mark with water.

1 ml of this standard solution contains 1 mg of cadmium.

Store in a borosilicate glass bottle fitted with a ground glass stopper.

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

Transfer, by means of a pipette, 10 ml of the standard cadmium solution (4.9) to a 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 sulfuric acid solution (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 equipment, and

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 and 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 — 10 — 20 — 25 and 50 µl, having standard colourless Eppendorf tips.

NOTE — 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 Procedure

6.1 Preparation of the test sample

Mix the laboratory sample well. If necessary first remove stones 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.

6.2 Test portion

6.2.1 Liquid products

Take, by means of a pipette, 10 ml of the test sample (6.1).

NOTE — It is also possible to take the test portion by mass by weighing, to the nearest 0,01 g, 10 g of the test sample.

6.2.2 Semi-solid and solid products

Weigh, to the nearest 0,01 g, a quantity of the test sample (6.1) corresponding to approximately 10 g of product.

6.3 Decomposition

Transfer the test portion (6.2) to a round-bottom flask (5.1). If the test portion is liquid (6.2.1) and contains ethanol, first eliminate the ethanol by boiling, and then allow to cool. Add 10 ml of the nitric acid (4.2), heat, and then carefully add 5 ml of the sulfuric acid (4.1). Proceed as described in ISO 5515, sub-clause 6.3.1 from the second paragraph to the eighth paragraph.

When decomposition is complete, filter the sulfuric solution, diluted with a few millilitres of water, through an ashless filter paper (5.4), which 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 rinsings in the same volumetric flask. Shake, allow to cool, and dilute to the mark. Mix by shaking.

6.4 Blank test

Carry out a blank test by repeating the decomposition (6.3), replacing the test portion by 10 ml of water.

6.5 Determination

6.5.1 Furnace programme

The furnace shall allow four independent thermal stages :

— drying of the solution;

— thermal decomposition;

— atomization;

— 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.

6.5.2 Preparation of the calibration graph

Prepare calibration solutions having cadmium concentrations of 5 — 10 and 20 µg/l by suitably diluting the standard cadmium solution (4.10). Inject successively into the furnace, programmed in accordance with 6.5.1, by means of a micropipette (5.8) fitted with a tip, 3 times 50 µl of each of these calibration solutions. Determine the absorbances from the heights of the peaks registered. Calculate the mean value of the absorbance from the results of the 3 injections into the furnace. The absorbances thus determined correspond, respectively, to 0,000 25, 0,000 5 and 0,001 µg of cadmium. Plot a calibration graph

having, for example, the values of absorbance as ordinates and the corresponding cadmium contents as abscissae.

6.5.3 Determination on the test solution

Inject successively into the furnace, programmed in accordance with 6.5.1, by means of a micropipette (5.8) fitted with a tip, 3 times 50 µl of the decomposed sulfuric solution obtained in 6.3 (test solution). 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.

6.5.4 Determination on the blank test solution

Inject successively into the furnace, programmed in accordance with 6.5.1, by means of a micropipette (5.8) fitted with a tip, 3 times 50 µl of the blank test solution (6.4). The absorbances 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 6.5.3 before referring to the calibration graph (6.5.2) to obtain the cadmium content of the test solution.

6.6 Number of determinations

Carry out two determinations on the same test sample (6.1).

7 Expression of results

7.1 Method of calculation and formulae

7.1.1 Liquid products

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

$$m_1 \times 100$$

where m_1 is the mass, in micrograms, of cadmium contained in 50 µl of the test solution, read from the calibration graph.

7.1.2 Semi-solid and solid products

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

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

where

m_0 is the mass, in grams, of the test portion (6.2.2);

m_1 is the mass, in micrograms, of cadmium contained in 50 µl of the test solution, read from the calibration graph.

7.1.3 Result

Take as the result the arithmetic mean of the values obtained in the two determinations (6.6), provided that the requirement for repeatability (see 7.2) is fulfilled.

7.2 Repeatability

The difference between the values obtained in the two determinations (6.6), carried out simultaneously or in rapid succession by the same analyst on the same test sample, shall not exceed 10 % of the mean.

7.3 Other method of expression of results

If it is wished to express the cadmium content on the dry basis, modify the formulae accordingly.

8 Test report

The test report shall show the method used and the result obtained, indicating clearly the method of expression used. It shall also mention any operating details not specified in this International Standard, or regarded as optional, as well as any incidents which may have affected the results.

The test report shall include all the information necessary for the complete identification of the sample.

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