
International Standard



6637

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

Fruits, légumes et produits dérivés — Détermination de la teneur en mercure — Méthode par absorption atomique sans flamme

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6637 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in August 1982.

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

Australia	Iran	Philippines
Austria	Ireland	Poland
Canada	Israel	Romania
Czechoslovakia	Kenya	South Africa, Rep. of
Egypt, Arab Rep. of	Korea, Dem. P. Rep. of	Spain
Ethiopia	Korea, Rep. of	Tanzania
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India	New Zealand	Yugoslavia

No member body expressed disapproval of the document.

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

1 Scope and field of application

This International Standard specifies a method for the determination of the mercury content of fruits, vegetables and derived products.

2 Principle

Decomposition of organic matter in a sulfuric-nitric acid medium. Reduction of mercury(II) to metallic mercury by the action of tin(II) chloride. Entrainment of the mercury vapour by a current of air and determination by flameless atomic absorption in an enclosed apparatus.

3 Reagents

All the reagents shall be of recognized analytical quality and shall, with the exception of the standard mercury chloride solutions (3.6 and 3.7), be free from mercury. The water used shall be distilled water free from mercury, or water of equivalent purity.

3.1 Sulfuric acid ($\rho_{20} = 1,84$ g/ml).

3.2 Nitric acid ($\rho_{20} = 1,38$ g/ml).

3.3 Nitric acid, 5 % (V/V) solution.

3.4 Tin(II) chloride, 100 g/l solution.

3.5 Urea, 400 g/l solution.

3.6 Mercury(II) chloride, standard solution corresponding to 1 g of mercury per litre.

In a 500 ml one-mark volumetric flask, dissolve 0,676 8 g of mercury(II) chloride in the nitric acid solution (3.3) and dilute to the mark with the same nitric acid solution.

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

3.7 Mercury(II) chloride, standard solution corresponding to 100 µg of mercury per litre.

At the time of use, prepare a 1/10 000 (V/V) dilution of the standard mercury(II) chloride solution (3.6) in the nitric acid solution (3.3).

1 ml of this standard solution contains 0,1 µg of mercury.

4 Apparatus

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

Usual laboratory equipment, and

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

4.2 Decomposition apparatus (see figure 1).

The apparatus is made of borosilicate glass and comprises four elements joined by conical ground joints, as follows:

- Element (A) is a modified Soxhlet extractor of capacity 200 ml, fitted with a stop-cock and a side tube linking this element directly to the round-bottomed flask (D);
- Element (B) is a dropping funnel of capacity 75 ml, fitted to the second neck of the round-bottomed flask (D);
- Element (C) is a condenser 35 cm long, fitted to the top of the Soxhlet extractor (A);
- Element (D) is a round-bottomed flask, of capacity 500 ml, fitted below the Soxhlet extractor (A), and in the second neck (30 mm from the first neck) of which is fitted the dropping funnel (B).

When the stop-cock of the Soxhlet extractor (A) is open, the apparatus is in the reflux position; when it is closed, the Soxhlet extractor (A) enables the condensed water and acid vapours to be retained.

4.3 Flameless atomic absorption apparatus (mercury analyser system).

The apparatus comprises a spectrometric system and determination flasks. The arrangement of the apparatus is shown in figure 2.

4.3.1 The spectrometric system comprises a mercury vapour lamp, the beam of which passes through an absorption cell. The variation in energy transmitted through the cell is measured by a phototube sensitive to ultraviolet radiation. A filter placed in front of the phototube isolates radiation of wavelength 253,7 nm. The equipment also has a read-out device.

The absorption cell shall have an internal diameter of 25 mm and a length of 115 mm and shall have silica windows.

4.3.2 The determination flask is equipped with a bubbler and is connected to a closed circuit in which the metallic mercury is released and entrained by circulation of air thus ensuring, by recycling, uniform distribution of the mercury in the circulating air. The absorption cell (see 4.3.1) is interposed in this circuit.

4.3.3 Any other atomic absorption apparatus having the required sensitivity may also be used.

4.4 Pipettes and burettes, of appropriate capacities.

4.5 One-mark volumetric flasks, of capacity 100 ml.

4.6 Analytical balance.

5 Procedure

5.1 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 (4.1).

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.

5.2 Test portion

5.2.1 Liquid products

Transfer, by means of a pipette (4.4), 10 ml of the test sample (5.1) to the round-bottomed flask (D) of the decomposition apparatus (4.2).

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.

5.2.2 Doughy, solid or dehydrated products

Transfer a mass of the test sample (5.1) corresponding to about 5 g of product, weighed to the nearest 0,01 g, to the round-bottomed flask (D) of the decomposition apparatus (4.2) and add 5 to 10 ml of water.

5.3 Decomposition

5.3.1 Decomposition of the test portion

5.3.1.1 Place a few glass beads in the flask (D), and connect the flask to the rest of the decomposition apparatus (4.2). By means of the dropping funnel (B), add, drop by drop, 5 ml of the nitric acid (3.2). Start a fast flow of water through the condenser (C) and turn the stop-cock of the Soxhlet extractor (A) to the reflux position; place a metal sheet with a hole of diameter approximately 5 cm below the flask, and heat with a small flame.

Allow the reaction to proceed very gently so as to avoid any loss of mercury through the transfer of particles supplied by the nitrous vapours in the condenser. Continue decomposition under reflux for about 30 min until the liquid has a uniform appearance. If the mixture turns brown, add several drops of nitric acid (3.2) through the dropping funnel (B) until the colour is discharged. Allow to cool.

5.3.1.2 Carefully add 10 ml of a mixture of equal parts of the nitric acid (3.2) and the sulfuric acid (3.1). Heat with a small flame and add nitric acid (3.2) drop by drop if the digest turns brown. Continue heating until fibrous matter has apparently been destroyed. Close the stop-cock of the Soxhlet extractor (A) to trap the water and acids and continue heating. The decomposition liquid will become more concentrated. If the liquid turns brown, add several drops of nitric acid (3.2) in just sufficient quantity to discharge the colour. Continue heating until the nitrous vapours are eliminated and there is a concentration of white fumes above the decomposition liquid.

NOTE — Waxes and fats cannot be completely destroyed by hot acids.

5.3.1.3 Control the heating so that the white fumes do not rise more than half way up the condenser (C). The liquid shall be colourless or pale yellow. Allow to cool. Carefully entrain the water and acids collected in the Soxhlet extractor (A) into the flask (D) by opening the stop-cock. Add 5 ml of the urea solution (3.5) through the side neck, and boil under reflux for 30 min. Allow to cool.

5.3.1.4 Disconnect the apparatus and transfer the contents of the flask (D) into a volumetric flask (4.5). Ensure that no undigested waxes or fats are transferred. Rinse the condenser (C) and the Soxhlet extractor (A) twice with 15 to 20 ml of the nitric acid solution (3.3), collecting the rinsings in the flask (D) and transferring them to the volumetric flask. Carefully rinse the device twice with 10 to 20 ml of water and add the rinsings to the solution contained in the volumetric flask. Dilute to the mark with water.

5.3.2 Blank test

Proceed as described in 5.3.1, but replace the test portion by 10 ml of water.

5.4 Determination

5.4.1 Transfer the decomposition liquid into a determination flask (4.3.2). Reduce the mercury(II) to metallic mercury by adding 5 ml of the tin(II) chloride solution (3.4). Immediately connect the air bubbling device and start the device which provides the air circulation.

5.4.2 Measure the absorbance at 253,7 nm using the apparatus described in 4.3.

5.4.3 Proceed in the same manner using the liquid derived from the blank test (5.3.2), and subtract the absorbance obtained from that of the liquid derived from the test portion.

5.5 Preparation of the calibration graph

Into a series of six volumetric flasks (4.5), place 0 - 1 - 2 - 3 - 4 and 5 ml of the dilute standard mercury(II) chloride solution (3.7), corresponding to 0 - 0,1 - 0,2 - 0,3 - 0,4 and 0,5 µg of mercury. Introduce into each flask, in the following order, about 80 ml of water, 5 ml of sulfuric acid (3.1). Mix, leave to cool and make up to the mark with water. Transfer quantitatively into six determination flasks (4.3.2). Proceed as described in 5.4.1 and 5.4.2.

Plot a graph having, for example, the mercury contents, in micrograms, of the calibration solutions as abscissae and the corresponding values of absorbance as ordinates.

5.6 Number of determinations

Carry out two determinations on the same test sample (5.1).

6 Expression of results

6.1 Method of calculation and formulae

6.1.1 Test portions taken by volume

The mercury content, expressed in micrograms per litre of product as received, is equal to

$$\frac{1\,000\,m}{V}$$

where

m is the mass, in micrograms, of mercury in the test portion, read from the calibration graph (5.5);

V is the volume, in millilitres, of the test portion, i.e. 10 ml.

6.1.2 Test portions taken by mass

The mercury content, expressed in micrograms per kilogram of product as received, is equal to

$$\frac{1\,000\,m}{m_0}$$

where

m is the mass, in micrograms, of mercury in the test portion, read from the calibration graph (5.5);

m_0 is the mass, in grams, of the test portion.

6.1.3 Result

Take as the result the arithmetic mean of the values obtained in the two determinations (5.6), provided that the requirement for repeatability (see 6.2) is fulfilled.

6.2 Repeatability

The difference between the values obtained in the two determinations (5.6), carried out simultaneously or in rapid succession by the same analyst on the same test sample, shall not exceed 10 % of the mean.

6.3 Other method of expression of results

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

7 Test report

The test report shall show the method used and the result obtained. It shall also mention all operating details not specified in this International Standard, or regarded as optional, together with any incidents likely to have influenced the results.

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

Dimensions in millimetres

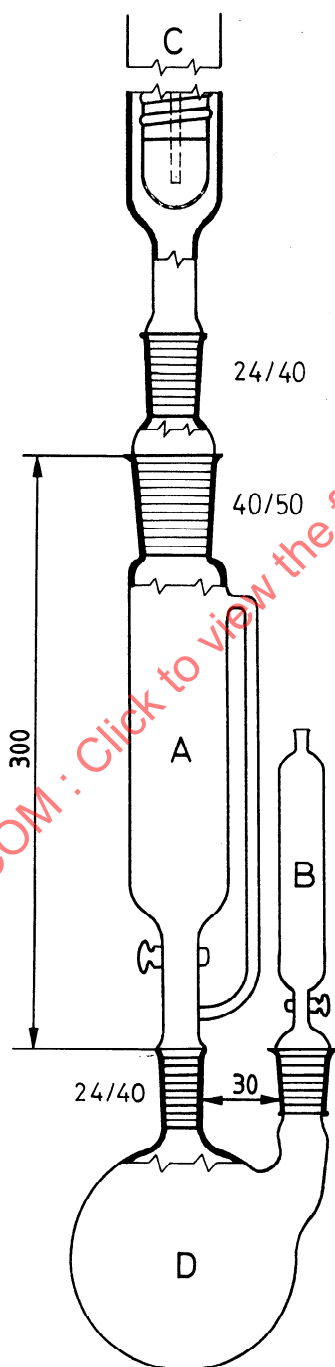


Figure 1 — Decomposition apparatus