International Standard



INTERNATIONAL ORGANIZATION FOR STANDARDIZATION®MEXCHAPOCHAR OPPAHUSALUAR TO CTAHCAPTUSALUN®ORGANISATION INTERNATIONALE DE NORMALISATION

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

First edition - 1984-06-01

Descriptors : food products, fruit and vegetable products, tests, determination of content, mercury, atomic absorption spectroscopic method.

Foreword

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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
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India	New Zealand	Yugoslavia

No member body expressed disapproval of the document.

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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 ($\varrho_{20} = 1,84 \text{ g/ml}$).
- **3.2** Nitric acid ($\varrho_{20} = 1,38 \text{ g/ml}$).
- **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:

a) 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);

b) Element (B) is a dropping funnel of capacity 75 ml, fitted to the second neck of the round-bottomed flask (D);

c) Element (C) is a condenser 35 cm long, fitted to the top of the Soxhlet extractor (A);

d) 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.