International Standard **ISO** 6633

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

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

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Foreword

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Fruits, vegetables and derived products — Determination of lead 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 lead content of fruits, vegetables and derived products.

2 Principle

Decomposition of organic matter in a nitric acid medium at high temperature and under pressure. Determination of the lead(II) cation by flameless atomic absorption spectrometry after the addition of orthophosphoric acid.

3 Reagents

All reagents shall be of recognized analytical grade and, in particular, shall be free from lead [with the exception of the standard lead solution (3.3)]. The water used shall be water double distilled in a borosilicate glass apparatus or water of equivalent purity.

3.1 Nitric acid (ϱ_{20} = 1,38 g/ml).

3.2 Nitric acid, solution.

Dilute 1 part by volume of nitric acid (3.1) with 9 parts by volume of water.

3.3 Orthophosphoric acid, 85 % solution (ϱ_{20} = 1,71 g/ml), or other equivalent matrix modifier used in the same quantities.

3.4 Lead, standard solution corresponding to 1 g of Pb per litre.

Dissolve 1,598 5 g of lead nitrate in 1 % (V/V) nitric acid solution and dilute to 1 000 ml.

Store in a borosilicate glass bottle with a glass stopper.

1 ml of this standard solution corresponds to 1 mg of Pb.

4 Apparatus

NOTE — The capsule (4.2) and all glassware should be washed before use with hot concentrated nitric acid and should be rinsed with double distilled water.

Usual laboratory equipment, and

4.1 Mechanical grinder, the inner walls and blades of which shall be coated with PTFE.[1]

4.2 Uniseal type decomposition capsule, consisting of a cylindrical crucible, the rim of which is coated with PTFE, of capacity 23 ml, in a stainless steel jacket with a screw top. A circular PTFE seal, which presses against the top of the crucible, ensures the airtightness of the capsule (see the figure).

4.3 Thermostatically controlled oven, capable of being maintained at 80 °C.

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

4.5 Funnel.

4.6 Haemolysis tubes.

4.7 Pipettes, of capacities 2; 5; 10; and 20 ml.

4.8 Eppendorf micropipettes, of capacities 10 and 100 µl, with Standartys Eppendorf tips.

NOTE — Some micropipettes are 10 % or more inaccurate. Unless they have been calibrated for this procedure, it is preferable that the same pipette be used for the test solution, the calibration solutions and the blank test solution.

4.9 Atomic absorption spectrometer, with a standard graphite furnace (without pyrolytic coating) and a corrector for non-specific absorptions (deuterium lamp) and a multipotentiometric recorder.[2]

— Source : lead hollow cathode lamp

— Measurement wavelength : 283,3 nm

— Purging gas : argon, or, if this is not available, nitrogen

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1) Polytetrafluoroethylene.

2) The use of a L'vov platform with the furnace is recommended.

4.10 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 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

Take, by means of a pipette (4.7), 5 ml of the test sample (5.1) and transfer it to the crucible (see 4.2).

NOTE — If the liquid contains alcohol, it is preferable to remove it beforehand by boiling and, after cooling, to make up the volume to the initial volume by adding water.

5.2.2 Pasty, solid or dried products

Weigh, to the nearest 0,01 g, into the crucible (see 4.2), a test portion of about 1 g, expressed as fresh product.

5.3 Decomposition

Add to the test portion 10 ml of the nitric acid (3.1), place the PTFE seal on the rim of the crucible and place the crucible in its stainless jacket. Fit the top. Transfer to the oven (4.3), maintained at 80 °C, and leave for 24 h.

Remove the capsule from the oven, allow to cool in a refrigerator, open the jacket and wash the condensed drops on the seal into the crucible by washing down with a few millilitres of water. Transfer the solution, pouring through the funnel (4.5), to a 50 ml one-mark volumetric flask (4.4) and rinse the crucible several times with water. Dilute to the mark. Mix by shaking.

NOTE — If a more sensitive lead determination is required, make up the solution in a 25 ml volumetric flask.

5.4 Determination

5.4.1 Preparation of solutions to be injected

5.4.1.1 Calibration solutions

Transfer, by means of a pipette (4.7), 10 ml of the standard lead solution (3.4) to a 1 000 ml one-mark volumetric flask and dilute to the mark with water.

Take 2; 5; 10; and 20 ml aliquot portions of this solution and transfer to four 1 000 ml one-mark volumetric flasks. Dilute each to the mark with nitric acid (3.2). (These solutions correspond, respectively, to 0,02; 0,05; 0,10; and 0,20 mg of lead per litre.)

Transfer 500 µl of each of these solutions to four haemolysis tubes (4.6) and add to each 10 µl of the orthophosphoric acid (3.3).

5.4.1.2 Test solution

Transfer to a haemolysis tube (4.6) 500 µl of the solution obtained in 5.3 and add 10 µl of the orthophosphoric acid (3.3).

5.4.2 Programming the furnace

Programming the furnace allows the following three operations to be performed :

— drying of the solution

— thermal decomposition

— atomization

The conditions are as follows :

— drying at 110 °C for 30 s

— gradual increase of temperature to 700 °C (in 45 s)

— thermal decomposition at 700 °C for 30 s

— atomization at 2 300 °C for 10 s. During this stage, the apparatus records the variation of absorbance. The purging gas circulation is interrupted ("gas stop") in order to prolong the stay of the atoms in the furnace

— raising the temperature to 2 700 °C to purge the oven at the end of the operation

5.4.3 Spectrometric measurements

5.4.3.1 Preparation of the calibration graph

Inject into the furnace programmed in accordance with 5.4.2, by means of a micropipette (4.8), successively three times 10 µl of each of the calibration solutions (5.4.1.1). Determine the absorbance of each from the heights of the peaks recorded. Calculate the mean absorbance from the values obtained.

The absorbances thus determined correspond to 0,000 2; 0,000 5; 0,001; and 0,002 µg of lead. Plot the calibration graph.

5.4.3.2 Measurement on the test solution

Inject into the furnace programmed in accordance with 5.4.2, by means of a micropipette (4.8), successively three times 10 µl of the test solution (5.4.1.2). Measure the respective absorbances and determine the mean absorbance.

5.5 Blank test

Carry out a blank test, using the same procedure for decomposition (5.3), but replacing the test portion by 5 ml of water.

Prepare the blank test solution in accordance with 5.4.1.2.

Inject into the furnace programmed in accordance with 5.4.2, by means of a micropipette (4.8), successively three times 10 µl of the blank test solution. The absorbances shall be zero or less than 0,005.

NOTE — The blank test is not necessary if the absence of lead in the reagents used has been established.

6 Expression of results

6.1 Method of calculation and formulae

6.1.1 Liquid products

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

$$1\ 000\ m_1$$

where m_1 is the mass, in micrograms, of lead in 10 µl of the solution obtained in 5.3, corrected if necessary for the blank test, and read from the calibration graph.

6.1.2 Pasty, solid or dried products

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

$$5\ 000\ \frac{m_1}{m_0}$$

where

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

m_1 is the mass, in micrograms, of lead in 10 µl of the solution obtained in 5.3, corrected if necessary for the blank test, and read from the calibration graph.

If it is wished to express the lead content on the dry basis, take the water content of the sample into account in the calculation.

6.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst on the same sample should not exceed 10 % (relative).

7 Test report

The test report shall show the method used and the results 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, together with details of any incidents likely to have influenced the results.

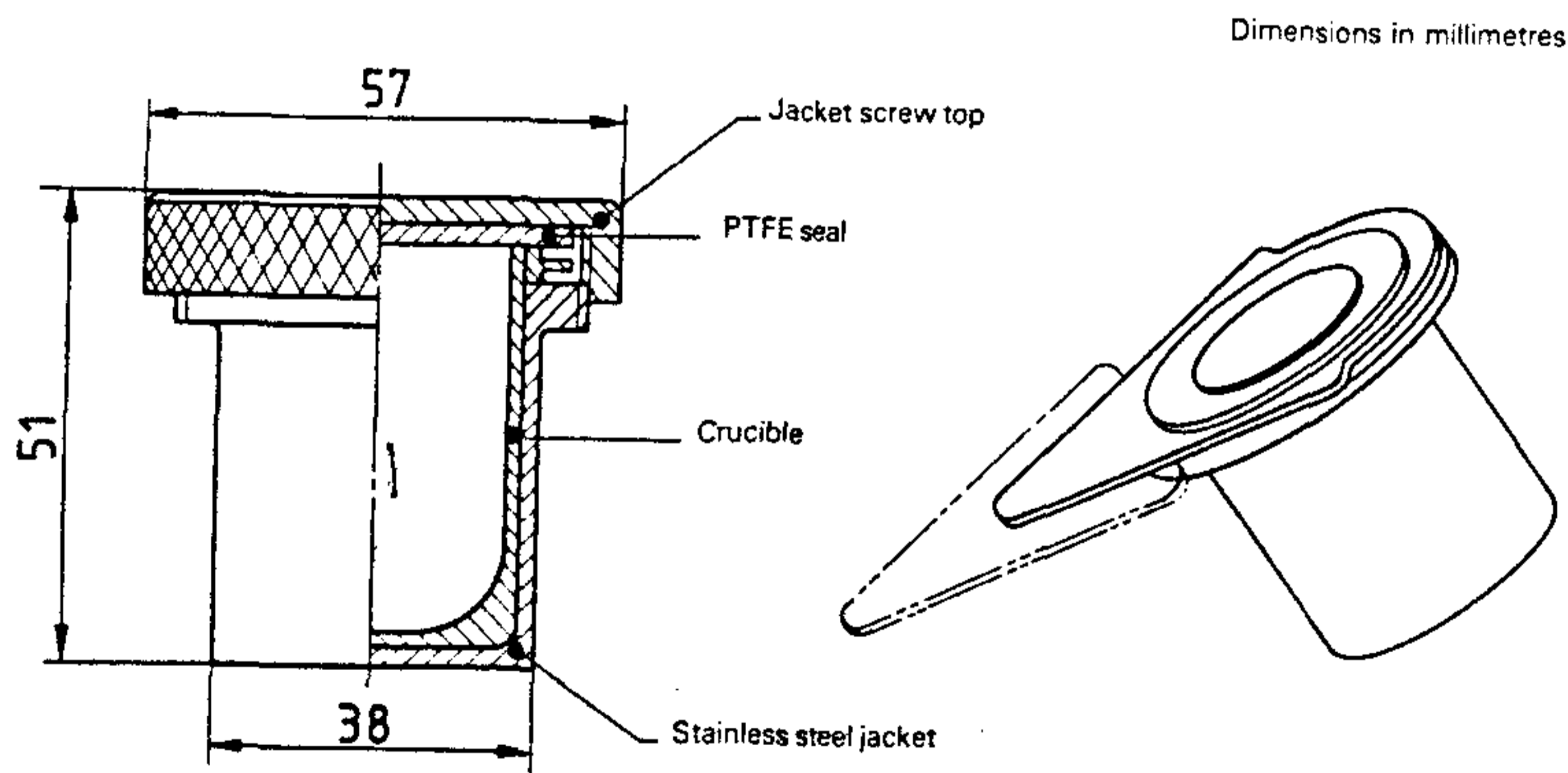The test report shall include all the information necessary for the complete identification of the sample.

Dimensions in millimetres

Figure — Decomposition capsule