

SRI LANKA STANDARD 1332 : Part 8 : 2010
ISO 6633 : 1984

METHODS OF TEST FOR
FRUITS AND VEGETABLE PRODUCTS
PART 8 – DETERMINATION OF LEAD CONTENT –
FLAMELESS ATOMIC ABSORPTION
SPECTROMETRIC METHOD

SRI LANKA STANDARDS INSTITUTION

Sri Lanka Standard
METHODS OF TEST FOR FRUITS AND VEGETABLE PRODUCTS
PART 8 – DETERMINATION OF LEAD CONTENT – FLAMELESS ATOMIC
ABSORPTION SPECTROMETRIC METHOD

SLS 1332 : Part 8 : 2010
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Sri Lanka Standard
METHODS OF TEST FOR FRUITS AND VEGETABLE PRODUCTS
PART 8 – DETERMINATION OF LEAD CONTENT – FLAMELESS ATOMIC
ABSORPTION SPECTROMETRIC METHOD

NATIONAL FOREWORD

This Sri Lanka standard was approved by the Sectoral Committee on Agricultural and Food Products and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 2010-03-25.

In order to accommodate the large number of test methods within the scope of one standard, this standard is published in several parts.

This part of the standard is identical with ISO 6633 :1984 , Fruits, vegetables and derived products- Determination of lead content – Flameless atomic absorption spectrometric method, published by the International Organization for Standardization (ISO).

Terminology and Conventions:

The text of the International Standard has been accepted as suitable for publication, without deviation, as a Sri Lanka Standard. However, certain terminology and conventions are not identical with those used in Sri Lanka Standards. Attention is therefore drawn to the following:

- a) Wherever the words “International Standard” appear referring to this standard should be interpreted as “Sri Lanka Standard”.
- b) The comma has been used throughout as a decimal marker. In Sri Lanka Standards it is the current practice to use the full point on the base line as the decimal marker.
- c) Wherever page numbers are quoted, they are ISO page numbers.

The test temperature adopted in Sri Lanka is 27 ± 2 °C and relative humidity 65 ± 5 per cent is recommended.

International Standard



6633

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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

First edition — 1984-09-15

UDC 634.1/635.6 : 543.422 : 546.815

Ref. No. ISO 6633-1984 (E)

Descriptors : agricultural products, fruit and vegetable products, fruits, vegetables, chemical analysis, determination of content, lead, atomic absorption method.

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 6633 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*.

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 ($\rho_{20} = 1,38$ 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 ($\rho_{20} = 1,71$ 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 μ l, 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

1) Polytetrafluoroethylene.

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

ISO 6633-1984 (E)**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 $10\ \mu\text{l}$ 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 $10\ \mu\text{l}$ 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 $10\ \mu\text{l}$ 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.

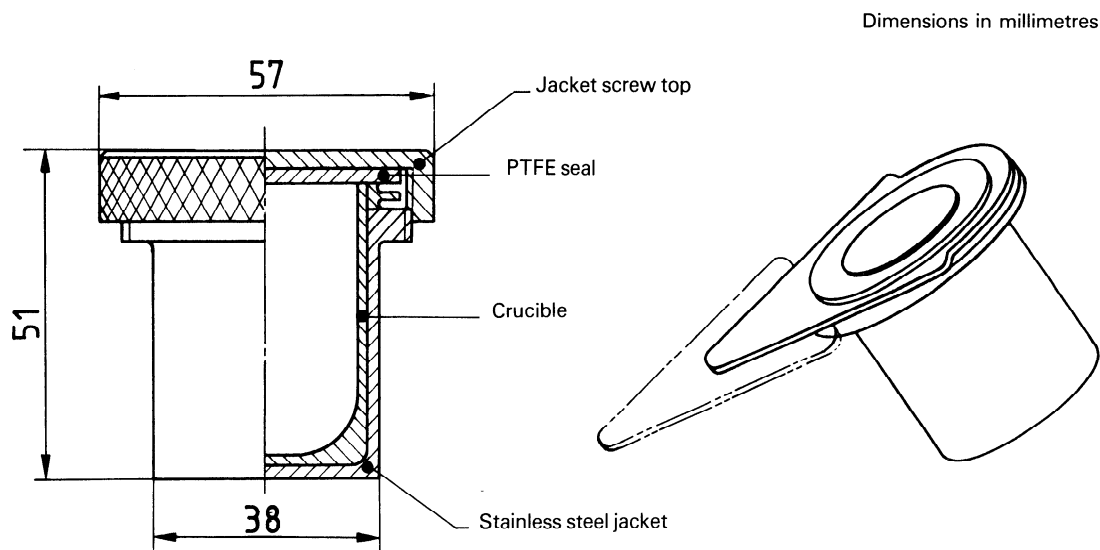
6.2 Repeatability

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.



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SRI LANKA STANDARDS INSTITUTION

The Sri Lanka Standards Institution (SLSI) is the National Standards Organization of Sri Lanka established under the Sri Lanka Standards Institution Act No. 6 of 1984 which repealed and replaced the Bureau of Ceylon Standards Act No. 38 of 1964. The Institution functions under the Ministry of Science & Technology.

The principal objects of the Institution as set out in the Act are to prepare standards and promote their adoption, to provide facilities for examination and testing of products, to operate a Certification Marks Scheme, to certify the quality of products meant for local consumption or exports and to promote standardization and quality control by educational, consultancy and research activity.

The Institution is financed by Government grants, and by the income from the sale of its publications and other services offered for Industry and Business Sector. Financial and administrative control is vested in a Council appointed in accordance with the provisions of the Act.

The development and formulation of National Standards is carried out by Technical Experts and representatives of other interest groups, assisted by the permanent officers of the Institution. These Technical Committees are appointed under the purview of the Sectoral Committees which in turn are appointed by the Council. The Sectoral Committees give the final Technical approval for the Draft National Standards prior to the approval by the Council of the SLSI.

All members of the Technical and Sectoral Committees render their services in an honorary capacity. In this process the Institution endeavours to ensure adequate representation of all view points.

In the International field the Institution represents Sri Lanka in the International Organization for Standardization (ISO), and participates in such fields of standardization as are of special interest to Sri Lanka.