SRI LANKA STANDARD 1100 : PART 2 : 1995

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METHODS OF TEST FOR HEAVY METALS IN FOOD

PART 2 : ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD FOR THE DETERMINATION OF LEAD

SRI LANKA STANDARDS INSTITUTION

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Sri Lanka Standard
METHODS OF TEST FOR HEAVY METALS IN FOOD
PART 2 : ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD FOR THE DETERMINATION OF LEAD

FOREWORD

This standard was approved by the Sectoral Committee on Agriculture and Food Technology - 2 and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 1995-11-23.

This part is one of the series of standards on determination of heavy metals in food using atomic absorption spectrophotometric method.

In reporting the result of a test or an analysis made in accordance with this standard, if the final value, obtained or calculated is to be rounded off, it shall be done in accordance with CS 102.

In the preparation of this standard, the valuable assistance derived from the following publication is gratefully acknowledged:

Official Methods of Analysis of the Association of Official Analytical Chemists (AOAC), 15th edition, 1990, 972.25

1 SCOPE

This part of the standard prescribes an atomic absorption spectrophotometric method for the determination of lead in food.

2 REFERENCES

CS 102 Presentation of numerical values
SLS 242 Methods for the destruction of organic matter

3 PRINCIPLE

Organic matter is digested and lead which is released is co-precipitated with strontium sulfate. Soluble sulfate salts are decanted. The precipitate is converted to carbonate salt and dissolved in acid and lead is determined by atomic absorption spectrophotometer at 217.0 nm or 283.3 nm.

4 REAGENTS

Unless specified otherwise, reagents of analytical grade and distilled water or water of equivalent purity shall be used.

4.1 Strontium solution, 2 per cent

Dissolve 6 g of $SrCl_2.6H_20$ in 100 ml of water.

4.2 Ternary acid mixture

Add 20 ml of sulfuric acid (rel.den = 1.84) to 100 ml of water and mix. Add 100 ml of nitric acid (rel.den. = 1.42) and 40 ml of 70 per cent perchloric acid, and mix.

4.3 Nitric acid, $c(HNO_3) = 1$ mol/l

Add 128 ml of nitric acid (rel.den. = 1.42) to 500 ml to 800 ml of water and dilute to 2 litres.

4.4 Lead stock solution, 1000 µg/ml

Dissolve 1.5985 g of recrystarlized lead nitrate [Pb $(NO_3)_2$] in about 500 ml of nitric acid **(4.3)** in one litre volumetric flask and dilute to volume with nitric acid **(4.3).**

4.5 Ammonium carbonate, saturated solution (approximately 20 per cent)

5 APPARATUS

Usual laboratory equipment and the following:

5.1 Mechanical grinder, the inside and blades of which are coated with polyethylene

5.2 Kjeldahl flasks, 500 - ml capacity

5.3 Glass beads

5.4 Stirring motor - with eccentric coupling for stirring centrifuge tubes

5.5 Atomic absorption spectrophotometer, with air-acetylene flame, suitable for measurements at wavelength 217 nm or 283.3 nm.

6 PROCEDURE

6.1 Preparation of the test sample

Mix the laboratory sample well. If necessary, grind the sample using the mechanical grinder (5.1).

Frozen or deep-frozen products shall be previously thawed in a closed container, and the liquid formed during thawing shall be added to the product before blending.

6.2 Preparation of test solutions

6.2.1 *Sample solution*

Weigh, to the nearest 0.001 g, about 10 g of the test sample (6.1) into the 500-ml Kjeldahl flask (5.2) and add 1 ml of strontium solution (4.1), and several glass beads (5.3). Add 15 ml of ternary acid mixture (4.2) and stand for 2 hours. Heat under hood or water vacuum manifold system until flask contains only sulfuric acid and inorganic salts.

NOTE

Take care to avoid sample loss from foaming when heat is first applied and soon after sample chars. Remove the flask from the heat and swirl it before continuing digestion. Add nitric acid (4.3) if necessary.

Cool the digest for few minutes. Wash while still hot (See Note) into 40 ml to 50 ml tapered bottom centrifuge tube (5.4) and swirl.

NOTE

Digest should be cool enough to add about 15 ml of water safely, but hot enough to boil when water is added.

Cool and centrifuge for 10 minutes at 350 x g. Decant the liquid into waste beaker (film like precipitate on the surface may be discarded). Dislodge precipitate by vigorously stirring with stirring motor (5.4). To complete transfer, add 20 ml of water and 1 ml of 0.5 mol/l sulfuric acid to the original flask and heat. Do not omit this step even if it appears, that the transfer is complete in the first wash. Wash hot contents of original digestion flask into centrifuge tube (5.4) containing precipitate. Swirl to mix, cool, centrifuge, and decant the liquid into waste beaker.

Dislodge precipitate by stirring vigorously. Add 25 ml of ammonium carbonate solution (4.5) and stir until all the precipitate is dispersed. Stand for one hour and centrifuge. Decant the liquid into waste beaker. Repeat the ammonium carbonate treatment.

After decanting, invert the centrifuge tube on a paper towel and drain all the liquid. Add 5 ml of nitric acid (4.3).

NOTE

Use large volume of nitric acid if more than 25μg of lead is expected in the sample.

Stir vigorously to expel carbondioxide or use ultrasonic bath for 2 minutes to 3 minutes. Stand 30 minutes, and centrifuge if precipitate remains.

6.2.2 Blank solution

Prepare a blank solution, using the same conditions as sample solution (6.2.1), but omitting the test sample .

6.3 Determination

6.3.1 Preparation of lead standard solution series

6.3.1.1 Dilute 10 ml of lead stock solution (4.4) to 100 ml with nitric acid (4.3) to obtain a solution containing 100 μg of lead per millilitre.

6.3.1.2 Place 1ml, 3ml, 5ml, 10ml, 15ml and 25 hl aliquots of this solution (6.3.1.1) into 100 ml volumetric flasks. Dilute to the mark with nitric acid (4.3). These solutions contain 1μg,3μg, 5μg, 10μg, 15μg and 25 μg of lead per millilitre respectively.

6.3.2 Spectrophotometric measurements and preparation of the calibration graph

Set instrument to previously established optimum conditions, using air-acetylene oxidizing flame and 217 nm or 283.3 nm resonant wavelength. Determine the absorbance of the sample, blank and standard solutions within the optimum working range (10 per cent to 80 per cent transmittance). Flush burner with nitric acid (4.3) and check zero point between readings. Prepare the calibration graph of absorbance against concentrations of lead standard solutions. Determine the lead content of the sample from calibration graph.

6.4 Calculation

$$\text{Lead content, mg per kg} = \frac{C \times V}{m}$$

where,

C is the lead content of the sample, in micrograms per millilitre, read from the calibration graph;
V is the volume in millilitres, of nitric acid (4.3)used to prepare the sample solution; and
m is the mass, in grams , of the test sample.

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SRI LANKA STANDARDS INSTITUTION

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