SRI LANKA STANDARD 1100 : PART 1 : 1995

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METHODS OF TEST FOR HEAVY METALS IN FOOD

PART 1 : ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD FOR THE DETERMINATION OF ZINC

SRI LANKA STANDARDS INSTITUTION

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SLS 1100 : Part 1 : 1995

Gr. 5

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Sri Lanka Standards are subject to periodical revision in order to accommodate the progress made by industry. Suggestions for improvement will be recorded and brought to the notice of the Committees to which the revisions are entrusted.

This standard does not purport to include all the necessary provisions of a contract.

SRI LANKA STANDARD
METHODS OF TEST FOR HEAVY METALS IN FOOD
PART 1 : ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD FOR THE DETERMINATION OF ZINC

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 publications is gratefully acknowledged:

i) ISO 6636/2 - Fruits, vegetables and derived products - Determination of zinc content - Part 2 Atomic absorption spectrometric method

ii) Official Methods of Analysis of the Association of Official Analytical Chemists (AOAC) - 15th edition, 1990, 969.32

1 SCOPE

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

2 REFERENCES

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

3 PRINCIPLE

Dry or wet ashing of the sample. Dissolve the residue in acid and dilute to optimum working range. Determining the zinc content by atomic absorption spectrophotometry at 213.8 nm using air-acetylene flame.

4 REAGENTS

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

4.1 Nitric acid, rel. den. =1.42

4.2 Sulfuric acid, rel. den.= 1.84

4.3 Hydrochloric acid,
Mix one volume of concentrated hydrochloric acid (rel. den.=1.19) with one volume of water.

4.4 Hydrochloric acid, approximately 3.7 g/l solution.
In a 1 000-ml one-mark volumetric flask, dilute 8.3 ml of concentrated hydrochloric acid (rel. den. 1.19) to the mark with water and mix.

4.5 Zinc stock solution
In a conical flask, dissolve 1 g of pure zinc metal in 10 ml of hydrochloric acid solution (4.3). Transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water, and mix.

5 APPARATUS

Usual laboratory equipment and the following :

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

5.2 Platinum dishes, diameter 70 mm.

5.3 Kjeldahl flasks, 250-ml capacity

5.4 Centrifuge

5.5 Boiling water bath

5.6 Muffle furnace, capable of being controlled at 525 ± 25 °C.

5.7 Atomic absorption spectrophotometer, with air-acetylene flame, suitable for measurements at a wavelength of 213.8 nm.

5.8 Heating device, use a IR lamp or heat gently over a flame.

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*

Decomposition may be carried out by dry ashing or wet digestion method.

6.2.1.1 Decomposition by the dry ashing

Weigh, to the nearest 0.001g, about 10 g of the test sample (6.1) into the clean platinum dish (5.2). Place it on the boiling water bath (5.5), regulating the temperature of the bath so as to minimize the risk of loss of material by spattering. Evaporate to dryness. Char the sample using a heating device (5.8) with low heat and continue the decomposition in the muffle furnace (5.6), controlled at 525 ± 25 °C.

Dissolve the ash in a few drops of nitric acid (4.1), evaporate on the boiling water bath (5.5), then transfer to the muffle furnace (5.6), and leave until white ashes are obtained. Dissolve the ash in 1 ml to 2 ml of the hydrochloric acid solution (4.3). Transfer the contents of the dish quantitatively to a centrifuge tube (5.4), rinsing the dish with about 20 ml of the hydrochloric acid solution (4.4), centrifuge. Transfer the supernatant liquid to a 50-ml volumetric flask. Add a further 10 ml of the hydrochloric acid solution (4.4) to the contents of the centrifuge tube (5.4), centrifuge. Transfer the supernatant liquid to the same flask. Repeat this procedure using 10 ml of water and make up the volume in the volumetric flask to the mark with water. Mix the solution.

6.2.1.2 Decomposition by the wet digestion method

Weigh, to the nearest 0.001 g, about 10 g of the test sample (6.1) into 250 ml kjeldahl flask (5.3). If the sample is liquid, evaporate to a small volume. If the sample contains ethanol, eliminate it beforehand by boiling and allow to cool. Add 10 ml of the nitric acid (4.1), heat and carefully add 5 ml of the sulfuric acid (4.2).

In some cases it may be useful to effect a preliminary digestion, by leaving the mixture in contact in the flask for a period (over-night for example).

Place the flask containing the mixture on the heating device (5.8) and heat cautiously to avoid excessive frothing. If necessary, interrupt heating and begin again only when vigorous frothing has ceased.

As soon as possible, bring the liquid to the boil and continue boiling until it begins to turn brown.

Then add, drop by drop, 1 ml to 2 ml portions of the nitric acid (4.1).

Bring to boil after every addition, but avoid vigorous heating. A small amount of nitric acid shall always remain in the mixture, as indicated by the presence of nitrous vapours.

Cease addition of portions of nitric acid when the solution no longer turns brown on addition of the acid. Continue heating until white fumes appear, indicating a high concentration of sulfuric acid and a reduction in nitric acid. If the solution turns brown again, continue the addition of nitric acid and repeat the operations described above until browning ceases.

Allow the solution to cool. The absence of colour or the presence of a light green or yellow colour indicates that the digestion is complete.

When decomposition is terminated, dilute the sulfuric solution with a few millilitres of water. Transfer the contents of flask quantitatively to a centrifuge tube (5.4), rinsing the flask with about 10 ml of water and collecting the rinsing water in the centrifuge tube (5.4). Centrifuge and transfer the supernatant liquid to a 50-ml volumetric flask. Add a further 10 ml of water to the contents of the centrifuge tube (5.4), centrifuge and transfer the supernatant liquid to the same flask. Repeat this procedure with another 10 ml of water and make up the volume in the volumetric flask to the mark with water. Mix the solution.

6.2.2 *Blank solution*

Carry out a blank test, using the same conditions for decomposition (6.2.1.1 or 6.2.1.2 as appropriate), but replacing the test sample (6.1) by 10 ml of water.

6.3 Determination

6.3.1 *Samples decomposed by the dry ashing method*

6.3.1.1 Preparation of the zinc standard solution series

Dilute the zinc stock solution (4.5) with the hydrochloric acid solution (4.4) to obtain four solutions containing 0.25 , 0.5, 1.0 and 1.5 mg of zinc per litre.

6.3.1.2 Spectrophotometric measurements and preparation of the calibration graph

Aspirate each of the solutions (6.3.1.1), in turn, into the flame of the spectrophotometer (5.7), at a rate such that the maximum absorbance is obtained for the solution having a zinc content of 1.5 mg per litre. Record the corresponding values of absorbance and draw the calibration graph.

Aspirate the test solution obtained (6.2.1.1) and the blank solution (6.2.2) into the flame of the spectrophotometer (5.7) at the same rate as in standard zinc solutions (6.3.1.1). Record the corresponding absorbances.

If the absorbance of the test solution (6.2.1.1) exceeds that of the most concentrated zinc standard solution, measure the absorbance of the test solution suitably diluted with the hydrochlotic acid solution (4.4).

The absorbance of the blank solution (6.2.2) shall be less than or equal to 0.002.

6.3.2 *Samples decomposed by the wet digestion method*

6.3.2.1 Preparation of the zinc standard solution series

a) Dilute the zinc stock solution (4.5) with water to obtain four solutions containing 2.5, 5, 10 and 15 mg of zinc per litre.

b) Place 5 ml of each of these solutions into a series of four 50-ml volumetric flasks. Add 30 ml to 35 ml of water, and then 5 ml of the sulfuric acid (4.2). Mix, allow to cool and dilute to the mark with water and mix. These solutions contain 0.25, 0.5, 1.0 and 1.5 mg of zinc per litre respectively.

6.3.2.2 Spectrophotometric measurements and preparation of the calibration graph

Aspirate each of the solutions (6.3.2.1 b), in turn, into the flame of the spectrophotometer (5.7), at a rate such that the maximum absorbance is obtained for the solution having a zinc content of 1.5 mg per litre. Record the corresponding values of absorbance and draw the calibration graph.

Aspirate the test solution (6.2.1.2) and the blank solution (6.2.2) into the flame of the spectrophotometer (5.7) at the same rate as in standard zinc solutions (6.3.2.1 b). Record the corresponding absorbances.

If the absorbance of the test solution (6.2.1.2) exceeds that of most concentrated calibration solution, measure the absorbance of the test solution suitably diluted with 10 per cent (V/V) sulfuric acid solution.

The absorbance of the blank solution (6.2.2) shall be less than or equal to 0.002.

6.4 Calculation

$$\text{Zinc content, mg. per kg} = \frac{(C_1 - C_2) \times 50}{m}$$

where,

C_1 is the zinc content of the sample, in milligrams per litre, read from the calibration graph (See Note);

C_2 is the zinc content of the blank solution, in milligrams per litre, read from the calibration graph; and

m is the mass, in grams, of the test sample.

NOTE

If the test solution was diluted, use the appropriate dilution factor in the calculation.

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SRI LANKA STANDARDS INSTITUTION

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