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METHODS OF TEST FOR HEAVY METALS IN FOOD

PART 3 : ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD FOR THE DETERMINATION OF TIN

SRI LANKA STANDARDS INSTITUTION

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Sri Lanka Standard
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METHOD FOR THE DETERMINATION OF TIN

SLS 1100 : Part 3 : 1995

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Sri Lanka Standard
METHODS OF TEST FOR HEAVY METALS IN FOOD
PART 3 : ATOMIC ABSORPTION SPECTROPHOTOMETRIC
METHOD FOR THE DETERMINATION OF TIN

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 methods.

This part of the standard consists of two sections as follows :

Section 1 : Determination of tin in food other than canned foods.
Section 2 : Determination of tin in canned foods.

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, 980.19 and 985.16

1 SCOPE

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

2 REFERENCES

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

SECTION 1 : DETERMINATION OF TIN IN FOOD OTHER THAN CANNED FOODS

1 PRINCIPLE

The sample is digested and tin is determined by atomic absorption spectrophotometry at 235.5 nm using nitrous oxide - acetylene flame.

2 REAGENTS

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

2.1 Nitric acid, rel. den. = 1.42

2.2 Sulfuric acid, rel. den. = 1.84

2.3 Ammonium chloride, saturated solution

2.4 Methyl alcohol

2.5 Tin standard solution, 1 000 μg/ml

Dissolve 1.000 g of pure tin in 150 ml of hydrochloric acid (rel. den. = 1.19) and dilute to one litre with distilled water.

3 APPARATUS

Usual laboratory equipment and the following:

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

3.2 Kjeldahl flasks, 800 ml capacity, made from borosilicate glass or silica

3.3 Glass beads, made from borosilicate glass

3.4 Atomic absorption spectrophotometer, with nitrous oxide-acetylene burner head, and reading device capable of 10 x scale expansion. Stannous (Sn) hollow cathode and electrodeless discharge lamps are both suitable.

4 PROCEDURE

4.1 Preparation of the test sample

Mix the laboratory sample well. If necessary, grind the sample using the mechanical grinder (3.1).

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

4.2 Preparation of test solutions

4.2.1 *Sample solution*

Weigh, to the nearest 0.001 g, about 20 g of the test sample (4.1) into Kjeldahl flask (3.2) and wash down inside neck of the flask with small amount of water. Add 3 to 4 boiling chips or glass beads (3.3), 60 ml of nitric acid (2.1) and 20 ml of sulfuric acid (2.2). Bring the mixture to boiling point at medium heat and continue boiling until white fumes appear. If solution turns dark, add 10 ml of nitric acid (2.1) and reheat until white fumes appear. Repeat as necessary until solution is clear and colourless or straw coloured. Allow the solution to cool partially. Add about 60 ml of water rapidly enough to cause boiling, but not so rapidly that solution is lost by spurting. Boil off water until dense white fumes reappear. Remove the solution from heat, and let cool partially. Repeat the addition of water and boiling to white fumes. Then turn off heat and allow the solution to cool.

Transfer the solution quantitatively to a 100-ml volumetric flask with two 10 ml portions of water. Add 5 ml of saturated ammonium chloride solution (2.3) and cool the solution completely. Carefully add 50 ml of methyl alcohol (2.4). Do not let solution become too hot during the addition of methyl alcohol. If necessary cool under water.

After solution has cooled to room temperature, dilute to the volume with water. Mix the solution.

4.2.2 *Blank solution*

Prepare a blank solution, using the same conditions as sample solution, (4.2.1), but omitting the test sample.

4.3 Determination

4.3.1 For each sample and blank, add 1.00 ml of water to 25-ml volumetric flask to second 25-ml volumetric flask add 1.00 ml of standard tin solution (2.5). Dilute both to volume with sample solution. (or blank solution).

4.3.2 Set up atomic absorption spectrophotometer (3.4) according to manufacturer's specifications for organic solvents. Use 235.5 nm Sn line and nitrous oxide -acetylene flame. Optimize for maximum Sn absorption. Depending on signal to noise ratio, scale expansion up to 10x may be used. Set nonabsorbing condition with water. Read blank, (4.3.1) sample (4.3.1) and spiked solution (4.3.1) in turn, aspirating water between readings. Record absorbance for all solutions.

4.4 Calculation

4.4.1

$$X = \frac{A_1 - A_2}{c}$$

4.4.2 Tin content, milligrams per kilogram of the product =

$$\frac{(A_2 - A_o)\ 25 \times 100}{X \times m \times 24}$$

where,

A_1 is the absorbance of the spiked solution (4.3.1);
A_2 is the absorbance of the sample solution (4.3.1);
A_o is the absorbance of the blank solution (4.3.1);
c is the concentration, in milligrams per litre, of spiked solution (4.3.1); and
m is the mass, in grams of the test sample (4.1).

SECTION 2 - DETERMINATION OF TIN IN CANNED FOODS

1 PRINCIPLE

The sample is digested with nitric acid and then with hydrochloric acid. Aqueous potassium chloride is added to the sample and standards to reduce positive instrument interference. Tin is determined by atomic absorption spectrophotometry at 235.5 nm with oxidizing nitrous oxide - acetylene flame.

2 REAGENTS

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

2.1 Nitric acid, rel. den. = 1 .42

2.2 Hydrochloric acid, rel. den. = 1.19

2.3 Potassium chloride solution, 10 mg/ml

Dissolve 1.91 g of potassium chloride in water and dilute to 100 ml.

2.4 Tin stock solution, 1 mg/ml

Dissolve 1.000 g of reagent grade tin in about 200 ml of hydrochloric acid (rel. den. = 1.19) and add about 200 ml of water. Cool to ambient temperature, and dilute to one litre with water.

3 APPARATUS

Usual laboratory equipment and the following :

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

3.2 Erlenmeyer flasks, 250 ml capacity

3.3 Atomic absorption spectrophotometer, with nitrous oxide - acetylene flame, wavelength 235.5 nm.

3.4 Oven

4 PROCEDURE

4.1 Preparation of the test sample

Mix the laboratory sample well. If necessary, grind the sample using the mechanical grinder (3.1).

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

4.2 Preparation of test solutions

4.2.1 *Sample solution*

Weigh, to the nearest 0.001 g, about 30 g to 40 g of juices or drinks, 20 g of foods containing 50 per cent to 75 per cent water, 5 g to 10 g of solids or semisolids and 2 g to 4 g of oil or fat into erlenmeyer flask (3.2) and dry it in oven (3.4) at 120 °C. Add 30 ml of nitric acid (2.1) to flask and within 15 minutes, heat gently in hood to initiate digestion, avoiding excessive frothing. (Do not add nitric acid to samples unless there is time to complete this stage of digestion in the same day.) Gently boil until 3 ml to 6 ml of digest remains or until sample just begins to dry on bottom. Do not allow the sample to char. Remove the flask from heat. Without delay, add 25 ml of hydrochloric acid (2.2), and heat gently for about 15 minutes until sample bumping from evolution of chlorine stops. Increase heat, and boil until 10 ml to 15 ml of volume remains, using similar flask with 15 ml of water to estimate the volume. Add about 40 ml of water. Swirl and pour into 100-ml volumetric flask rinsing once with about 10 ml of water. When hydrochloric acid is present in digest, samples may stand overnight or longer. Pipet 1.0 ml of potassium chloride solution (2.3) into volumetric flask. Cool to ambient temperature and dilute to volume with water, adding additional water to approximate compensate for volume of fat (if any) in the flask. Mix well. Filter about 30 ml to 50 ml through dry, medium porosity paper into dry, polypropylene or polyethylene screw-cap bottle.

4.2.2 *Blank solution*

Prepare a blank solution, using the same conditions as sample solution (4.2.1), but omitting the test sample. Do not filter blank solutions.

4.3 Determination

4.3.1 *Preparation of the tin standard solution series*

Into each of five 100-ml volumetric flasks, pipet 10 ml of hydrochloric acid (2.2), 1.0 ml of potassium chloride solution (2.3) and 0 ml, 5 ml, 10 ml, 15 ml and 20 ml of tin stock solution (2.4). Dilute to volume with water. These solutions contain 0 µg, 50 µg, 100 µg, 150 µg and 200 µg of tin per millilitre respectively.

Using 200 μg per millilitre standard solution and 235.5 nm tin line, optimize spectrophotometer, burner, and flame according to manufacturer's instructions. Then increase nitrous oxide flow or decrease acetylene flow to give oxidizing flame. This reduces sensitivity but improves precision to 0± 0.0004 absorbance for blank solution and 0.201 ± 0.001 absorbance for 100 μg per millilitre standard solution. Periodically monitor the sensitivity of a standard solution. If the sensitivity decreases by more than 20 per cent, turn off flame and carefully clean the burner slot.

Zero the spectrophotometer while aspirating water. But do not adjust to zero until after determinations. (Autozero reduces precision). Aspirate water before and after each sample, standard and blank solutions. Take three readings for each solution and average. Take all absorbance readings with reference to absorbance of water.

Record the absorbance of standards and draw the calibration curve. Visually check for inaccurate standards. Two times blank - corrected absorbance for 50 μg per millilitre standard should not differ by more than 3 per cent from blank corrected absorbance for 100 μg per ml standard.

5 CALCULATION

5.1 Block standard blank with 50 μg per millilitre standard solution. Using ratio of absorbances, calculate the concentration of standard blank.

$$\text{Concentration of standard blank, } \mu g \text{ per ml} = \frac{A_0 \times 50}{A_1 - A_0}$$

where,

A_0 is the absorbance of the standard blank; and
A_1 is the absorbance of 50 μg per ml standard solution.

Add concentration of standard blank to nominal concentration of standard solutions to obtain true concentrations of standard solutions.

5.2 Measure absorbance of sample blanks as for standard blank and calculate the concentration of sample blank.

$$\text{Concentration of sample blank, } \mu g \text{ per ml} = \frac{A_0}{A_1} \times c_1$$

where,

A_0 is the absorbance of the sample blank;
A_1 is the absorbance of the 50 μg/ml standard solution; and
c_1 is the true concentration of 50 μg per ml standard solution.

Calculate the mean concentration of sample blanks.

5.3 Determine the concentration of sample solution by one of two ways.

5.3.1 Measure absorbance of sample solutions and 50 μg/ml standard solution or 100 μg/ml standard solution (depending on sample concentration level).

Block samples with standard solution and calculate the blank - corrected sample solution concentrations.

$$\text{Concentration of sample solution, } \mu g \text{ per ml} = \left[\frac{A}{A_2} \times c_2 \right] - c_3$$

where,

- A is the absorbance of the sample solution;
- A_2 is the absorbance of the standard solution used;
- c_2 is the true concentration, in μg per ml. of standard solution used; and
- c_3 is the mean value of sample blanks concentration, in μg per ml.

When high accuracy is not required or when calibration curvature is extensive, use calculation given in **5.3.2** after confirmation that sensitivity changes and baseline drift are absent during the analytical run.

5.3.2 Calibrate using the blank, 50, 100 and 150 μg per millilitre standards. Run sample blanks and samples. Calculate solution concentrations using either instrument microprocessor or calibration curve. Calculate the mean of sample blank concentrations. Calculate the blank - corrected solution concentrations by subtracting the mean value of sample blank concentration from the solution concentrations.

5.4 Calculation

$$\text{Tin content, } \mu g \text{ per g} = \frac{c_4}{m} \times 100$$

where,

- c_4 is the blank corrected solution concentration, in μg per ml (5.3.1); and
- m is the mass, in grams, of the test sample.

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SRI LANKA STANDARDS INSTITUTION

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