SRI LANKA STANDARD 315:1976
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METHOD FOR
THE DETERMINATION OF TIN

BUREAU OF CEYLON STANDARDS

METHODS FOR THE DETERMINATION OF TIN

SLS 315 : 1976

(Attached AMD 412)

Gr.3

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AMD 412

AMENDMENT NO: 01 TO SLS 315 : 1976
METHOD FOR THE DETERMINATION OF TIN

SRI LANKA STANDARDS INSTITUTION

Amendment No: 01 approved on 2010-10-15 to SLS 315 : 1976

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METHOD FOR THE DETERMINATION OF TIN

FOREWORD

Insert the following under 4th paragraph of **FOREWORD**.

“Introduction of the atomic absorption spectrophotometric method for the determination of Tin is given in Appendix **A**.”

Insert the following at the end of Clause **3.4**.

“APPENDIX A
ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD

The atomic absorption spectrophotometric method is to be adopted if and when it could be used as a routine method.”

…………….

SRI LANKA STANDARD

METHODS FOR THE DETERMINATION OF TIN

FOREWORD

This Sri Lanka Standard has been prepared by the Drafting Committee of the Bureau on Chemical Test Methods. It was approved by the Agricultural and Chemicals Divisional Committee of the Bureau of Ceylon Standards and was authorised for adoption and publication by the Council of the Bureau on 1976-02-04.

In the preparation of this standard the assistance derived from the Society for Analytical Chemistry is acknowledged.

1 SCOPE

This standard prescribes methods for the determination of tin.

2 METHOD FOR THE DETERMINATION OF AMOUNTS OF TIN NOT GREATER THAN 30 µg.

2.1 Principle of method

After destruction of the organic matter by wet oxidation with nitric and sulphuric acids, nitric perchloric and sulphuric acids or 50 per cent hydrogen peroxide in the presence of sulphuric acid,the residual sulphuric acid is diluted to four times its volume with water to give an approximately 9 N concentration of the acid.

Tin is selectively separated from this solution by treating it with potassium iodide and extracting tin (IV) iodide into toluene. Tin (IV) is then returned to aqueous solution by shaking the toluene extract with a solution of sodium hydroxide. After acidification, and removal of free iodine from the solution, the tin (IV) is determined spectrophotometrically as its coloured complex with catechol violet, the solution being buffered to pH 3.8 with acetate.

2.2 Reagents

All reagnets should be of analytical grade.

2.2.1 *Water*

Purify glass-distilled water further by passing it through a mixture of strongly acidic cation-exchange resin and strongly basic anion-exchange resin.

2.2.2 *Sulphuric acid, approximately 9 N*

Cautiously mix 250 ml of sulphuric acid, sp. gr. 1.84, with 500 ml of water, cool to room temperature and dilute to 1 litre with water.

2.2.3 *Potassium iodide, approximately 5 N*

Dissolve 83 g of potassium iodide in water to produce 100 ml. Prepare freshly each day.

2.2.4 *Toluene (low in benzene)*

2.2.5 *Sodium hydroxide, approximately 5 N and approximately 0.1 N.*

2.2.6 *Hydrochloric acid, approximately 5 N.*

2.2.7 *Ascorbic acid solution*

A freshly prepared 5 per cent m/v aqueous solution.

2.2.8 *Catechol violet solution*

A 0.05 per cent, m/v aqueous solution. Prepare freshly each week.

2.2.9 *Sodium acetate trihydrate solution*

A 20 per cent, m/v aqueous solution.

2.2.10 *Ammonia solution, approximately 5 N.*

2.2.11 *Tin (IV) stock solution*

Dissolve 0.1000 g of pure granulated tin in 20 ml of sulphuric acid, sp.gr. 1.84, by heating until fumes appear. Cool, cautiously dilute with 150 ml of water, and cool again. Add 65 ml of sulphuric acid, sp.gr. 1.84, cool and transfer to a 500-ml calibrated flask. Dilute to the mark with water.

1 ml of solution = 200 µg of tin

2.2.12 *Tin (IV) dilute standard solution*

Dilute 5.0 ml tin (IV) stock solution to 100 ml with water in a calibrated flask. Prepare freshly each day.

1 ml of solution = 10 µg of tin.

2.3 Preparation of calibration graph

Transfer by pipette, or small capacity burette, suitable volumes of dilute standard tin solution, to cover the range 0 µg to 30 µg of tin, to a series of 50-ml beakers and treat each as follows: dilute to 7 ml with water, add 1 ml of 5 N sodium hydroxide and mix. Add 2.5 ml of 5 N hydrochloric acid, mix, add 2.0 ml of catechol violet solution, mix again and add 5 ml of sodium acetate solution (see Note 1). Adjust the pH of the solution with 5 N ammonia solution to 3.8 ± 0.1 units, with the aid of a pH meter. Transfer to a 25 ml calibrated flask, dilute to the mark with water, mix thoroughly, and set aside for 30 minutes. Measure the optical density of the solution in a 10 mm cell at a wavelength of 552 nm, with the solution containing no added tin in the reference cell. Construct a graph relating the amount of tin to the optical density (see Note 2). The graph should be rectilinear and pass through the origin.

2.4 Procedure

Dilute the sulphuric acid solution containing not more than 30 µg of tin to approximately 9 M. Cool and transfer it to a separating funnel. For each 25 ml of solution add 2.5 ml of 5 M potassium iodide, mix, and add 10 ml of toluene. Insert the stopper, shake the funnel vigorously for 2 minutes allow the layers to separate, and discard the aqueous phase. Wash the toluene layer, without shaking it with 5 ml of a solution prepared by mixing 25 ml of 9 N sulphuric acid and 2.5 ml of 5 M potassium iodide, and discard the washings. The toluene layer will be coloured pink with extracted iodine.

Add 5 ml of water to the toluene extract and then 5 N sodium hydroxide dropwise, with shaking, until the toluene layer is colourless. Add 2 drops of 5 N sodium hydroxide in excess (usually a total of 8 drops to 10 drops is required). Insert the stopper, shake the funnel for 30 seconds, allow the phases to separate, and transfer the aqueous layer into a 50 ml beaker. Shake the toluene layer with 3 ml of 0.1 N sodium hydroxide for 30 seconds, allow the layers to separate, and add the aqueous layer to the contents of the 50-ml beaker. Retain the organic (toluene) phase.

Acidify the aqueous solution in the beaker with 2.5 ml of 5 N hydrochloric acid, and decolorise the liberated iodine by the dropwise addition of ascorbic acid solution. Add 2.0 ml of catechol violet solution, and mix. Wash the toluene retained from above, without shaking, with 5 ml of sodium acetate solution. Add the washings to the contents of the beaker, mix, and adjust the pH of the solution to 3.8 ± 0.1 units with 5 N ammonia solution by means of a pH meter. Transfer the solution to a 25-ml calibrated flask, and complete the determination of tin as described above under "Preparation of Calibration Graph". Calculate the amount of tin present by reference to the calibration graph.

NOTES

1 The order of addition of reagents is important, and the stated order should be strictly adhered to.

2 When a new bottle or batch of catechol violet is used a fresh calibration graph should be prepared.

3 METHOD FOR THE DETERMINATION OF AMOUNTS OF TIN FROM 30 µg TO 150 µg

3.1 Principle of method

The organic matter in the sample is destroyed by wet oxidation with nitric and sulphuric acids, with nitric, perchloric and sulphuric acid, or with 50 per cent m/v hydrogen peroxide (tin free) and sulphuric acid. The residue is diluted with water and extracted with a solution of dithizone in carbon tetrachloride to remove any copper present. The aqueous solution is then allowed to react with zinc dithiol in the presence of thioglycollic acid, and sodium lauryl sulphate, which acts as a dispersing agent for the tin-dithiol complex. The extinction of the red-coloured suspension is measured at 535 nm.

3.2 Reagents

All reagents should be of analytical-reagent quality unless stated otherwise.

3.2.1 *Dithizone solution*

Prepare a 0.02 per cent m/v solution in carbon tetrachloride. This solution should be recently prepared or stored in a refrigerator.

3.2.2 *Sodium lauryl sulphate solution*

Prepare as 1 per cent m/v aqueous solution using sodium lauryl sulphate of B.P. quality.

3.2.3 *Sulphuric acid, 20 per cent, v/v*

To 50 ml of water cautiously add 20 ml of concentrated sulphuric acid (sp. gr. 1.84). Cool and dilute to 100 ml.

3.2.4 *Tin stock solution*

Dissolve 0.100 g of pure granulated tin in 20 ml of sulphuric acid (sp. gr. 1.84) by heating until fumes appear. Cool, cautiously dilute with 150 ml of water, and cool again. Add 65 ml of sulphuric acid (sp. gr. 1.84), again cool, and transfer to a 500-ml calibrated flask. Dilute to the mark with water.

3.2.5 *Tin standard solution*

Dilute 10 ml of tin stock solution to 100 ml with water. Prepare freshly each day. (1 ml of solution = 20 µg of tin).

3.2.6 *Zinc dithiol*

Dissolve 0.2 g of zinc dithiol in 1 per cent sodium hydroxide solution containing a few drops of ethanol. Add 1 ml of thioglycollic acid and dilute to 100 ml with 1 per cent sodium hydroxide solution. Prepare immediately before use.

3.3 Procedure

Destroy the organic matter as in SLS 242*.

**SLS 242* *Standard methods for the destruction of organic matter.*

When oxidation is complete, dilute the solution with 10 ml of water and boil gently to fuming. Allow the solution to cool, add a further 10 ml of water and boil gently to fuming. Transfer the final clear solution to a calibrated flask of such volume that the diluted solution contains no more than the equivalent of 4 ml of concentrated sulphuric acid per 100 ml.

Transfer, by pipette, 10 ml of this solution containing between 30 µg and 150 µg of tin into a separating funnel, add 5 ml of dithizone solution, and shake the funnel. Allow the layers to separate, and discard the lower dithizone layer. Continue the extraction with successive 5-ml portions of dithizone solution until the extracts remain green. Wash the aqueous solution with two successive 5-ml portions of carbon tetrachloride, and discard the washings.

Transfer the aqueous phase to a 20-ml calibrated flask and add 20 per cent sulphuric acid, so that the final solution contains the equivalent of between 0.7 ml and 1 ml of concentrated sulphuric acid. Add 1 ml of sodium lauryl sulphate solution, mix, and then add 1 ml of zinc dithiol reagent.

Dilute to the mark with water, mix thoroughly, and immerse in a boiling water bath for exactly 1 minute. Allow the solution to cool at room temperature for 20 minutes to 30 minutes, and then measure the extinction of the solution at a wavelength of 535 nm, by using 10-mm cells with a reagent blank solution in the comparison cell.

Read the number of micrograms of tin equivalent to the observed extinction from a previously prepared calibration graph, and calculate the tin content of the sample.

3.4 Preparation of calibration graph

Transfer aliquots of standard tin solution to cover the range 30 µg to 150 µg of tin to a series of 20-ml calibrated flasks. Add 5 ml of 20 per cent sulphuric acid, mix and proceed as described above beginning at "Add 1 ml of sodium lauryl sulphate solution ...."

Measure the extinctions of the solutions, and construct a graph relating the extinctions to the number of micrograms of tin.

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