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METHOD FOR THE DETERMINATION OF ZINC

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BUREAU OF CEYLON STANDARDS

METHOD FOR THE DETERMINATION OF ZINC

S. L. S. 302 : 1974

Gr.4

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SRI LANKA STANDARD METHOD FOR THE DETERMINATION OF ZINC

FOREWORD

This Sri Lanka Standard was prepared by the Drafting Committee on Chemical test methods. It was approved by the Agricultural and Chemicals Divisional Committee of the Bureau of Ceylon Standards and was authorized for adoption and publication by the Council of the Bureau on 31st October, 1974.

In the preparation of this standard the assistance obtained from the following publication in the preparation of this document is acknowledged:

Society for Analytical Chemistry—
Official Standardised & recommended methods of analysis.
Analytical Methods Committee,
Society for Analytical Chemistry,
London, 1967.

1. SCOPE

This standard prescribes methods for the determination of zinc.

2. METHOD 1 (For zinc contents greater than 5mg per litre)

2.1 **Principle of method**—After destruction of organic matter, zinc, together with certain other heavy metals, is extracted with diphenylthiocarbazone (dithizone) at pH 4.5; zinc, together with bismuth and cadmium, is then extracted with dilute acid, and after evaporation to dryness, the iodine which is quantitatively liberated when the residue is treated with potassium iodide in the presence of potassium ferricyanide is titrated with thiosulphate. This reaction is specific for zinc.

2.2 **Range** — For zinc contents greater than 5mg per litre.

2.3 **Applicability** — The method is generally applicable.

2.4 Reagents

(i) All water shall be glass distilled or de-ionised, and be free from zinc.

(ii) Ammonium acetate solution—A 38.6 per cent $\frac{m}{v}$ solution in water.

(iii) Hydrochloric acid-Sp.gr. 1.16 to 1.18.

(iv) Dithizone extraction solution — Dissolve without heating, 0.15g of diphenylthiocarbazone (dithizone) in 100ml of chloroform, transfer to a separating funnel, add 100ml of water and 5ml of ammonia solution, sp.gr. 0.88 to 0.89, shake vigorously, allow to separate, and discard the chloroform layer. Wash the aqueous solution with two 5ml portions of chloroform, and discard the chloroform layer. To the aqueous solution add 200ml of analytical reagent grade chloroform, followed by sufficient hydrochloric acid so that on shaking and allowing to separate, the aqueous layer is colourless. Transfer the lower chloroform layer to a brown glass bottle.

(v) Diluted hydrochloric acid (1+1) — Mix equal volumes of hydrochloric acid and water.

Diluted hydrochloric acid (1+10) — Mix 1 volume of hydrochloric acid with 10 volumes of water.

(vi) Perchloric acid—A 60 per cent $\frac{m}{m}$ solution in water.

(vii) Hydrogen peroxide solution — A 30 per cent $\frac{m}{v}$ solution in water (100) volume.

(viii) Glacial Acetic Acid.

(ix) Ammonium hydrogen fluoride.

(x) Potassium iodide solution — A 5 per cent $\frac{m}{v}$ solution in water. Prepare freshly as required.

(xi) Starch indicator solution — Mix 1 g of soluble starch with 5ml of cold water, and then add 95ml of boiling water. Heat to boiling, and cool. Filter before use through a fast filter paper (Whatman No. 41). Store in a glass bottle in the dark, and prepare freshly every 5 days.

(xii) Sodium thiosulphate 0.1N.

(xiii) Sodium thiosulphate, 0.002N—Make an appropriate dilution of the 0.1N solution with freshly boiled and cooled water. Prepare freshly each day, and store in the dark.

(xiv) Potassium ferricyanide solution — A 5 per cent $\frac{m}{v}$ solution in water. Prepare freshly as required.

2.5 Procedure

2.5.1 Destruction of Organic Matter — Destroy the organic matter in a measured volume of the sample as described under SLS 242*

2.5.2 Separation of zinc — Transfer a suitable volume (not more than 40ml, and containing, preferably, 0.1 to 1mg of zinc) of the solution prepared as described above under 'Destruction of Organic Matter' to a separating funnel, add 10ml of ammonium acetate solution and 5ml of dithizone extraction solution, shake vigorously, allow to separate, and transfer the lower chloroform layer to a second separating funnel (see Note 1). Add to the chloroform solution 6ml of ammonium acetate solution 3ml of diluted hydrochloric acid (1+1) and 10ml of water shake, allow to separate, and transfer the lower chloroform layer to a third separating funnel. Wash the chloroform solution with 20ml of water, and transfer the chloroform layer to a fourth separating funnel. Repeat the extraction of the aqueous layer in the first separating funnel with further 5ml portions of dithizone extraction solution, following the same washing procedure and using the aqueous liquids remaining in the other separating funnels, until the dithizone extraction solution does not change in

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*SLS 242 Methods for the destruction of Organic Matter.

colour on shaking with the liquid in the first separating funnel. To the combined chloroform solution collected in the fourth separating funnel, add 10ml of diluted hydrochloric acid (1+10), shake well, allow to separate, transfer the lower chloroform layer to another separating funnel. Transfer the acid aqueous solution to a 60 or 100ml beaker, wash the first separating funnel with 10ml of water, and add the washing to the beaker. Repeat the extraction of the chloroform solution with a further 10ml portion of diluted hydrochloric acid (1+10), again wash the separating funnel with 10ml of water after transferring the acid extract to the beaker, and also add the washing to the beaker. Evaporate the contents of the beaker to dryness, add 0.25ml (5 drops) of perchloric acid and 0.25ml (5 drops) of hydrogen peroxide solution, and evaporate to dryness again on a hot plate. Repeat the treatment with perchloric acid and hydrogen peroxide solution until all the organic matter has been destroyed and a white residue remains. Wash down the sides of the beaker with water, and again evaporate to dryness.

2.5.3 **Determination of zinc** — To the residue from the 'Separation of zinc' add 0.1ml of glacial acetic acid and about 0.01g (a 'speck') of ammonium hydrogen fluoride, followed by 2ml of potassium iodide solution and 0.1ml (2 drops) of starch indicator solution if blue colour appears on the addition of starch indicator solution, add 0.002 N sodium thiosulphate until the colour is just discharged. Add, stirring with a glass rod, about 0.5ml of potassium ferricyanide solution, and titrate with 0.002 N sodium thiosulphate, carrying out the titration if possible in artificial light, although this is not essential (see Note 2); 1ml of 0.002 N sodium thiosulphate=0.196mg of zinc, Zn.

Express the result as milligrammes of zinc, Zn, per litre of sample.

Notes

Note 1: If a reddish purple colour does not appear in the chloroform layer, there is too little zinc present for this method to be applied.

Note 2: The blue starch iodide complex may be absorbed on the precipitated zinc ferrocyanide, and if this occurs the precipitate serves as an indicator.

3. METHOD 2 (For zinc contents upto 5mg per litre).

3.1 Principle of method — After destruction of the organic matter, copper is removed if necessary, and zinc is separated from other metals by extraction with dithizone at pH 4.7. Excess of dithizone is removed with sodium sulphide, and the zinc is determined colorimetrically as its complex with dithizone.

3.2 Range — For zinc contents up to 5mg per litre.

3.3 Applicability — The method is generally applicable.

3.4 Reagents — All water shall be twice redistilled from glass apparatus, and preferably stored in polythene bottles.

(i) **Carbon tetrachloride** — Redistilled.

(ii) **Dithizone stock solution** — A 0.05 per cent $\frac{m}{v}$ solution of diphenylthiocarbazone (dithizone) in carbon tetrachloride.

(iii) **Dithizone extraction solution** — Extract 10ml dithizone stock solution with two successive 50ml portions of diluted ammonia (50ml of water containing 2ml of 10N ammonia), filtering if necessary the combined ammoniacal extracts. Acidify the extracts with diluted hydrochloric acid (about 1 per cent $\frac{m}{v}$), and extract the precipitated dithizone with 100ml of carbon tetrachloride, washing the extract with two successive 10ml portions of water, and filtering the carbon tetrachloride solution through a dry filter paper. Prepare freshly as required.

(iv) **Strong standard zinc solution** — Dissolve 0.4398g of zinc sulphate, $ZnSO_4 . 7H_2O$, in water, dilute to 1 litre at 20°C with water, and mix well.

(v) **Dilute standard zinc solution** — Dilute 10.0ml of strong standard zinc solution to 1 litre at 20°C with water, and mix; 1 ml = 1 μg of zinc, Zn.

(vi) Hydrochloric acid, N.

(vii) **Buffer solution** — Dissolve 136g of sodium acetate, $C_2H_3O_2$ Na. $3H_2O$, in water, add 58ml of glacial acetic acid, and dilute to 500ml with water. Remove the zinc from this solution by extracting with successive small portions of dithizone stock solution, shaking the mixture for about 2 minutes each time, until the extract is obtained which remains green, and then filter the aqueous layer through cottonwool to remove any carbon tetrachloride.

(viii) **Sodium thiosulphate solution** — 25 per cent $\frac{m}{v}$ solution in water.

(ix) **Sodium sulphide solution** — Dissolve 4g of analytical reagent grade sodium sulphide, Na_2 S. $9H_2O$, in water, and dilute to 100 ml with water. Dilute 1ml of this solution to 100ml with water. Prepare freshly as required.

(x) Anhydrous sodium sulphate.

3.5 Procedure

3.5.1 Reagent blank — Carry out a blank test by the entire procedure, using the same amounts of reagents as are used in the test and omitting only the sample.

3.5.2 Destruction of organic matter — Destroy the organic matter as given in SLS 242*.

3.5.3 Preliminary treatment of digest — Determine the acidity of 10ml aliquot of the solution prepared as described above under 'Destruction of Organic Matter' and adjust the acidity of another aliquot, containing not more than 5μg of zinc, to 0.1N; Note the volume of the adjusted solution.

3.5.4 Removal of copper — To the adjusted solution, add 5ml of dithizone extraction solution, shake for 2 minutes and allow to separate; if the colour of the lower carbon tetrachloride layer is red, continue the addition of dithizone extraction solution, 0.5ml at a time, with shaking as above, until an excess is present, as indicated by the appearance of a green

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*SLS 242 Methods for the destruction of Organic Matter.

tint. Allow to separate, and discard the lower carbon tetrachloride layer. Repeat the extraction with further portions of dithizone extraction solution twice more, discarding the lower carbon tetrachloride layer each time.

3.5.5 Determination of zinc

3.5.5.1 **Preparation of calibration graph** — Measure appropriate amounts of dilute standard zinc solution, covering the range 0 to 5 μg of zinc, into a series of separating funnels, and treat each solution by the following procedure.

Add 1ml of N hydrochloric acid, dilute to 10ml with water, add 5ml of buffer solution, and 1ml of sodium thiosulphate solution. Add, from a burette, 4.0ml of dithizone extraction solution, shake for 2 minutes, allow to separate, and filter the lower carbon tetrachloride layer through a small plug of cotton wool into a second separating funnel. Wash the aqueous layer with 1ml of carbon tetrachloride, and filter the washing through the same plug of cotton wool into the second separating funnel. Repeat the extraction and washing of the aqueous layer, and discard it (see Note). To the combined carbon tetrachloride solutions in the second separating funnel, add 10ml of sodium sulphide solution, shake, allow to separate, and transfer the lower carbon tetrachloride layer to a third separating funnel. Wash the aqueous layer in the second separating funnel with 1ml of carbon tetrachloride, add the washing to the solution in the third separating funnel, and discard the aqueous layer. Repeat the extraction of the carbon tetrachloride solution in the third separating funnel with further successive 10ml portions of sodium sulphide solution and the washing with carbon tetrachloride until the colour of the aqueous layer is no longer yellow. Shake the carbon tetrachloride solution with about 1g of anhydrous sodium sulphate and filter through a dry 90mm fast filter paper (Whatman No. 41) into a 25ml calibrated flask. Wash the separating funnel and the filter paper with a few millilitres of carbon tetrachloride, add the washing to the solution in the flask, dilute to the mark at 20°C with carbon tetrachloride, and mix.

Measure the optical density of each solution in turn against carbon tetrachloride in a 10mm cell in a spectrophotometer at 532nm, or in a photoelectric absorptiometer using a green filter. After correcting for the optical density of the solution to which no standard zinc solution has been added, construct a graph relating the optical densities to the number of microgrammes of zinc, Zn.

3.5.5.2 **Treatment of test and blank solution** — To the aqueous solution from the 'Removal of Copper', (Clause 3.5.4) add half its volume of buffer solution, mix and treat as described above under 'Preparation of calibration graph', (Clause 3.5.5.1) commencing at the addition of 1ml of sodium thiosulphate solution. From the optical density of the test and blank solutions, obtain from the calibration graph the number of microgrammes of zinc in each solution, and hence calculate the amount of zinc in the sample.

Express the result as mg/kg of Zn.

Note : The carbon tetrachloride layer will be green if the zinc content does not exceed 5 μg and the concentration of the dithizone extraction solution is satisfactory ; this can easily be checked by extracting a known amount of zinc with the dithizone extraction solution.

4. METHOD 3

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

BUREAU OF CEYLON STANDARDS

The Bureau of Ceylon Standards (BCS) is the national standards organisation of Sri Lanka and was established by the Hon. Minister of Industries & Fisheries, as provided for by the Bureau of Ceylon Standards Act, No. 38 of 1964.

The principal objects of the Bureau as set out in the Act are to promote standards in industry and commerce, prepare national Standard Specifications and Codes of Practice and operate a Standardisation Marks Scheme and provide testing facilities, as the need arises.

The Bureau is financed by Government grants and the sale of its publications. Financial and administrative control is vested in a Council appointed in accordance with the provisions of the Act.

The detailed preparation of Standard Specifications is done by Drafting Committees composed of experts in each particular field assisted by permanent officers of the Bureau. These Committees are appointed by Divisional Committees, which are appointed by the Council. All members of the Drafting and Divisional Committees render their services in an honorary capacity. In preparing the Standard Specifications, the Bureau endeavours to ensure adequate representation of all view points.

In the International field the Bureau represents Sri Lanka in the International Organisation for Standardisation (ISO) and will participate in such fields of Standardisation as are of special interest to Sri Lanka.

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