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METHOD FOR THE DETERMINATION OF CADMIUM

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BUREAU OF CEYLON STANDARDS

METHOD FOR THE DETERMINATION OF CADMIUM

S. L. S. 303 : 1974

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SRI LANKA STANDARD METHOD FOR THE DETERMINATION OF CADMIUM

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 authorised for adoption and publication by the Council of the Bureau on 31st October 1974.

The assistance obtained from the following publications in the preparation of this document is acknowledged:

Society for analytical Chemistry — Official Standardised and Recommended Methods of Analysis. Analytical Methods Committee. Society for Analytical Chemistry, London, 1967.

1. SCOPE

This Sri Lanka Standard prescribes methods for the determination of cadmium.

2. METHOD I

2.1 **Principle** — After destruction of the organic matter, cadmium is determined colorimetrically as its red complex with dithizone.

2.2 Range

For cadmium contents up to 50 μg.

2.3 Applicability

The method is generally applicable, but copper and nickel interfere unless complexed with cyanide.

2.4 Reagents

All water must be glass distilled or de-ionised and be free from cadmium.

(i) **Sodium hydroxide solution** — A 20 per cent $\frac{m}{v}$ solution in water.

(ii) **Strong standard cadmium solution** — Dissolve 0.10 g of cadmium metal in 50 ml of a 10 per cent $\frac{m}{v}$ solution of nitric acid in water, boil the solution to expel oxides of nitrogen, and dilute to 1 litre at 28°C with water.

(iii) **Dilute standard cadmium solution** — Dilute 10.0 ml of strong standard cadmium solution to 100 ml at 28°C with water; 1 ml=10µg of cadmium, Cd. Prepare freshly as required.

(iv) **Potassium sodium tartrate solution** — A 25 per cent $\frac{m}{v}$ solution of potassium sodium tartrate, $C_4 H_4 0_6 KNa.4H_2 0$, in water.

(v) **Sodium hydroxide-potassium cyanide solution A** — Dissolve 40 g of sodium hydroxide and 1 g of potassium cyanide in 100 ml of water.

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

(vii) **Hydroxylamine hydrochloride solution** — A 20 per cent $\frac{m}{v}$ solution in water. Remove interfering metals from this solution by extracting with successive 5 ml portions of a 0.01 per cent $\frac{m}{v}$ solution of diphenylthiocarbazone (dithizone) in carbon tetrachloride until an extract is obtained which remains green, and then remove the excess of dithizone by extracting with successive 10-ml portions of carbon tetrachloride until a colourless extract is obtained; finally remove the excess of carbon tetrachloride by warming in a beaker, cool, and filter.

(viii) **Dithizone stock solution** — 0.1 per cent $\frac{m}{v}$ solution of diphenyl thiocarbazone (dithizone) in carbon tetrachloride. Filter, and store in a refrigerator.

(ix) **Dithizone extraction solution** — Extract 15ml of dithizone stock solution with two 50-ml portions of water containing 2 ml of 10 N ammonia, filtering if necessary, the combined ammoniacal extracts. Acidify the extracts with dilute hydrochloric acid (about 1 per cent) and extract the precipitated dithizone with 100 ml of carbon tetrachloride, washing the extract with two successive 10-ml portions of water, and filtering the carbon tetrachloride solution through a dry filter-paper. Prepare freshly as required.

(x) **Tartaric acid solution** — A 2 per cent $\frac{m}{v}$ solution in water.

(xi) **Sodium hydroxide — potassium cyanide solution B**— Dissolve 40 g of sodium hydroxide and 0.05 g of potassium cyanide in 100 ml of water.

2.5 Procedure

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

2.5.2 **Destruction of organic matter** — Destroy the organic matter in a measured volume of sample as described in SLS 242*.

2.5.3 **Preliminary treatment of digest** — Transfer an aliquot (containing not more than 50μg of cadmium) of solution prepared as described in Clause 2.5.2 to a separating funnel, neutralise with sodium hydroxide solution, and dilute to about 25 ml with water.

2.5.4 **Determination of cadmium** — Preparation of calibration graph-Measure appropriate amounts of dilute standard cadmium solution, covering the range 0 to 50μg of cadmium, into a series of separating funnels, neutralise with sodium hydroxide solution, and dilute to about 25 ml with water. To each, add 1 ml of potassium sodium tartrate solution, 5 ml sodium hydroxide—potassium cyanide solution A and 1 ml of hydroxylamine hydrochloride solution, mixing well after each addition. To each, add 10 ml of dithizone extraction solution, shake vigorously for 1 minute, allow to separate, and transfer the lower carbon tetrachloride layers to a second series of separating funnels, each containing 25 ml of tartaric acid solution. Repeat the extraction of the aqueous layers with further 10 ml portions of dithizone extraction solution until extracts are obtained which are no longer coloured pink, adding the lower carbon tetrachloride layers to the corresponding solutions in the second series of separating funnels. Shake the second series of separating funnels for 2 minutes, allow the layers to separate, and discard the lower carbon tetrachloride layers. To each aqueous layers, add 5 ml of carbon tetrachloride, shake for 1 minute, allow to separate, and again discard the lower carbon tetrachloride layers.

To each aqueous solution, add 0.25 ml of hydroxylamine hydrochloride solution and 10 ml of dithizone extraction solution, followed by 5 ml of sodium hydroxide potassium cyanide solution B, shake for 1 minute, allow to separate, and filter the lower carbon tetrachloride layers through toughened filter-papers (Whatman No. 541 is suitable) into a series of 50-ml calibrated flasks.

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* SLS 242: Methods for the destruction of organic matter.

2.5.5 Repeat the extraction of the aqueous layers with further 10 ml portions of dithizone extraction solution until extracts are obtained which are no longer coloured pink, filtering each extract through the filter-paper into the corresponding calibrated flask. Dilute each of the combined extracts to the mark with carbon tetrachloride and mix.

Measure the optical density of each solution in turn against carbon tetrachloride, using a 1 or 4 cm cell according to the depth of colour in a spectrophotometer at 525 nm, or in a photoelectric absorptiometer fitted with a suitable green filter. Construct a graph relating the optical densities to the number of microgrammes of cadmium Cd.

Treatment of test and blank solutions — Treat the solution in the separating funnels from the "Preliminary Treatment of Digest", as described above under "Preparation of calibration graph", commencing at the addition of 1 ml of potassium sodium tartrate solution. From the optical density of the test and blank solutions, obtain from the calibration graph the number of microgrammes of cadmium in each solution, and hence calculate the amount of cadmium in the sample (see Note)

Express the result as milligrammes of cadmium, Cd/kg of the sample.

Note: If an instrument is not available, a visual colour-comparison method may be used. Develop the colours of a series of standards containing 0 to 25 μg of cadmium, and compare them with those developed by the test and blank solutions after transferring to 50-ml Nessler cylinders.

3. METHOD II

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

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