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METHOD FOR THE DETERMINATION OF COPPER

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BUREAU OF CEYLON STANDARDS

METHOD FOR THE DETERMINATION OF COPPER

S.L.S. 301 : 1974

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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.

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SRI LANKA STANDARD METHOD FOR THE DETERMINATION OF COPPER

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.

In the preparation of this standard the assistance derived from the following publication is acknowledged :

IUPAC Determination of copper content in foodstuffs - photometric method. Butterworths, London 1959.

1. SCOPE

This standard prescribes methods for the determination of copper.

2. METHOD 1

2.1 Principle — The material is digested by means of sulphuric and nitric acids. The residue is diluted with water, the disodium salt of ethylenediamine tetra acetic acid (EDTA) and citrate are added and the pH adjusted to 8.5 with dilute ammonium hydroxide. By adding sodium diethyldithio-carbamate ("sodium carbamate") the copper complex.

$$(C_2H_5)_2 = N - C \begin{matrix} S \\ S \end{matrix} Cu/2$$

is formed. This golden brown compound is extracted from the water phase by means of carbon tetrachloride. The optical density of the carbon tetrachloride layer is measured with a spectrophotometer at 435 nm.

Many metals react with sodium carbamate, but if EDTA and citrate are present all interference, except that from bismuth and tellurium, is eliminated by chelation.

Copper carbamate is destroyed by cyanide, while bismuth carbamate is not and tellurium carbamate is partly destroyed by cyanide. If, therefore, the carbon tetrachloride layer is shaken with cyanide solution and does not turn colourless, bismuth or tellurium carbamate or both are present.

If bismuth or tellurium are shown to be present, another aliquot part of the sample solution is extracted in the same way, but the solution of the carbamates in carbon tetrachloride is washed with sodium hydroxide solution. Copper carbamate is stable during this treatment while the bismuth and tellurium carbamates are destroyed.

2.2 Reagents

2.2.1 All reagents used shall be of analytical reagent quality.

(i) Ammonium hydroxide 6N may be prepared by passing gaseous ammonia through water or by purifying ammonia as described for EDTA—citrate solution.

(ii) Carbon tetrachloride.

(iii) EDTA—Citrate solution—Dissolve 20 g of ammonium citrate and 5 g of the disodium salt of ethylenediamine tetra-acetic acid in water and dilute to 100 ml with water. Add 0.1 ml of the carbamate solution and extract with carbon tetrachloride. Repeat the extraction process till the organic layer is colourless.

(iv) Nitric acid concentrated.

(v) Potassium cyanide solution 5% $\frac{m}{v}$

(vi) Sodium carbamate solution. Dissolve 1 g of sodium diethyl-dithiocarbamate in 100 ml of water and filter the solution if it is not clear. Store in the dark in a refrigerator. The solution used should not be older than 7 days.

(vii) Sodium hydroxide solution 1 N.

(viii) Standard copper solution (use either *a* or *b*).

(*a*) Place 0.200 g of pure copper wire of foil in a 125 ml conical flask. Add 15 ml of 4N nitric acid, cover with a watch glass and allow to dissolve, warming to complete solution. Boil to expel oxides of nitrogen, cool and dilute to 200 ml with water (1ml = 1 mg of copper). Dilute this solution with 2N sulphuric acid to a working standard solution, containing 2 μg of copper per ml, on the day when it is to be used.

(*b*) Dissolve 0.3926 g of copper sulphate ($CuSO_4.5H_2O$ analytical reagent quality) in 2N sulphuric acid and dilute to 1 litre. Dilute this solution, containing 100μg of copper per ml, with 2N sulphuric acid to a working standard solution, containing 2μg of copper per ml, on the day when it is to be used.

(ix) Sulphuric acid sp. gr. 1.84.

(x) Thymol blue solution — Dissolve 0.1 g of thymol blue in 2.15 ml of 0.1N sodium hydroxide and dilute to 100 ml with water.

(xi) Water — Redistil distilled water in a still constructed throughout of heat resistant borosilicate glass. De-ionised water is also suitable.

2.2.2 Control of reagents — If the total blank with 10 ml of sulphuric acid and 30 ml of nitric acid amounts to more than 1.0 μg of copper per 25 ml aliquot of the dilution, the copper content of each reagent is checked. If necessary nitric acid is purified by distillation.

2.3 Glassware

All glassware, and the reagent bottles shall be of heat resistant borosilicate glass. Glassware should be washed before use with dilute nitric acid and water.

2.4 Laboratory

The laboratory should be as dust-free as possible, and entirely devoted to the determination of traces of metals. Apparatus should be covered against dust during analysis.

2.5 Procedure

2.5.1 Digestion — The sample solution shall be prepared as described in SLS 242*.

Add two drops of thymol blue solution and ammonium hydroxide until the solution is green or bluish green.

Cool and add 1 ml of the carbamate solution and (from a burette) 15 ml of carbon tetrachloride. Stopper the funnel and shake vigorously for 2 minutes. Allow the layers to separate. Place a piece of cotton wool in the stem of the separating funnel and run the carbon tetrachloride layer into a 25 ml measuring cylinder. Repeat extraction procedure with another 5 ml portion of carbon tetrachloride. Combine the extracts and remove traces of moisture with anhydrous sodium sulphate. Transfer to a dry cell of the photometer. Do not expose the extract unnecessarily to light greater than 150 lux but measure immediately the optical density at 436 nm.

Read the number of microgrammes of copper equivalent to the observed optical density of the test and blank solutions from a calibration graph previously prepared according to Clause 7, and so obtain the content of copper in the sample. Express the result in mg Cu per kg of sample (ppm).

2.5.3 **Qualitative test for bismuth and tellurium** — Transfer the carbon tetrachloride from the cell to a 25 ml test tube or to a stoppered separating funnel, add 10 ml of potassium cyanide solution and shake. If the carbon tetrachloride layer turns colourless, bismuth and tellurium carbamates are absent.

*S.L.S. 242 "Methods for the destruction of organic matter"

2.5.4 Copper determination if bismuth or tellurium are present— Repeat the determination in another 25 ml of sample solution. Transfer the carbon tetrachloride extract to a stoppered 25 ml test tube or to a separating funnel, add 10 ml of 1N sodium hydroxide and shake. After settling remove the sodium hydroxide layer as completely as possible. Repeat washing with another 10 ml sodium hydroxide 1N.

Measure the optical density of the copper carbamate solution as described.

2.6 Calibration

Transfer to a separating funnel 10 ml of EDTA—citrate solution, and the amounts of standard copper solution in sulphuric acid as stated in Table I.

TABLE I

Working copper standard solution 1 ml = 2μg Cu	0	1	2.5	5	10	15	20	25 ml
2N sulphuric acid	25	24	22.5	20	15	10	5	0 ml

Proceed as described in clause 2.5.2 second and third paragraphs. Construct a graph relating optical densities to microgrammes of copper.

3. METHOD 2

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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