भारतीय मानक
Indian Standard

IS 4027 (Part 1) : 2024

काँसे के रासायनिक विश्लेषण — पद्धतियाँ

भाग 1 इलेक्ट्रोलाइटिक विधि द्वारा तांबे और सीसा का निर्धारण

(दूसरा पुनरीक्षण)

Chemical Analysis of Bronzes — Methods

Part 1 Determination of Copper and Lead by Electrolytic Method

(Second Revision)

ICS 73.080

भारतीय मानक ब्यूरो
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October 2024 **Price Group 3**

Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

FOREWORD

This Indian Standard (Part 1) (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Methods of Chemical Analysis of Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This standard was first published in 1967 and it covered determination of copper, lead, tin, manganese, phosphorus, nickel, iron, silicon, aluminium, zinc and antimony in bronzes. Subsequently, the first revision was published in 1987 to bifurcate the standard into different parts which superseded IS 4027 : 1967.

This revision has been brought out to bring the standard in the latest style and format of the Indian Standards. This part is one of that series and covers the determination of copper and lead by electrolytic method. The other parts are as follows:

Part 2 Determination of manganese — Photometric method

Part 3 Determination of phosphorus — Volumetric method

Part 4 Determination of nickel-dimethylglyoxime photometirc method

Part 5 Determination of tin-iodimetric method

Part 6 Determination of zinc by complexometric (EDTA) method

Part 7 Determination of antimony by rhodamine B spectrophotometric method

Part 8 Determination of iron

Part 9 Determination of aluminium by atomic absorption spectrometric method

Part 10 Determination of silicon

Part 11 Determination of lead — Ethylenediamine tetraacetic acid (EDTA) — Titrimetric method

The methods of analysis prescribed in this standard may primarily serve as referee methods and may also be used by the laboratories for their day-to-day work. Due consideration has been given in the preparation of this standard to the facilities available in the country for such analysis.

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be same as that of the specified value in this standard.

Indian Standard

CHEMICAL ANALYSIS OF BRONZES — METHODS

PART 1 DETERMINATION OF COPPER AND LEAD BY ELECTROLYTIC METHOD

(Second Revision)

1 SCOPE

This standard (Part 1) prescribes a method for determination of copper and lead in the ranges as specified in the relevant Indian Standards on bronzes.

NOTE — The method is not applicable when manganese is present in bronzes and also when lead is of the order of 0.1 percent.

2 REFERENCES

The standards given below contains provisions which through reference in this text, constitutes provisions of this standard. At the time of publication the edition indicated was valid. All standards are subject to revision and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standard.

IS No.	*Title*
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 1817 : 1961	Methods of sampling non-ferrous metals for chemical analysis

3 SAMPLING

Samples shall be drawn and prepared in accordance with IS 1817.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070) shall be employed in the test.

5 DETERMINATION OF COPPER AND LEAD BY THE ELECTROLYTIC METHOD

5.1 Outline of the Method

The sample is dissolved in nitric acid, and copper and lead are electrolytically deposited and weighed.

NOTE — This method is not applicable for too low as well as higher content of lead.

5.2 Apparatus

5.2.1 *Electrodes*

The following platinum electrodes are recommended, but strict adherence to the shape and size of the electrodes is not essential. For agitation of electrolyte in order to decrease the time of deposition, one of the types of rotating forms of electrodes, generally available, may be employed.

5.2.2 *Cathode*

It may he formed either from plain or from perforated sheet or from wire gauge.

5.2.2.1 Gauge cathodes preferably made from gauge containing 400 meshes/cm^2 should be used. The wire used for making gauge should be approximately 0.20 mm in diameter. Cathodes should be stiffened by doubling the gauge for about three millimetres on the top and the bottom or by reinforcing the gauge at the top and bottom with platinum ring or band.

5.2.2.2 The diameter of the cylinder should be approximately 30 mm and the height 50 mm. The stem should be made from platinum alloy wire, such as platinum-iridium, platinum-rhodium or platinum-ruthenium having diameter of approximately 1.5 mm. It should be flattened and welded throughout the entire length of the gauge. The overall height of the cathode should be approximately 130 mm.

5.2.3 *Anode*

When the amount of lead in the sample is less than 4.0 mg, a spiral anode should be used. It should be made from 1.0 mm or larger platinum wire formed into spiral of seven turns with a height of approximately 50 mm and diameter of 12 mm, the overall height being 130 mm.

5.2.3.1 When the amount of lead in the sample is more than 4.0 mg, the gauge anode should be used. It should be made of the same material and of the same general design as platinum gauge cathode

mentioned under 5.2.2. It should be approximately 12 mm in diameter and 50 mm in height, the overall height being 130 mm.

5.3 Reagents

5.3.1 *Dilute Nitric Acid* — 1 : 1 (*v/v*)

5.3.2 *Hydrobromic Acid* — 48 percent

5.3.3 *Urea* — solid

5.3.4 *Sulphamic Acid* — solid

5.3.5 *Hydrogen Sulphide Solution*

Saturate dilute sulphuric acid (1 : 99) with hydrogen sulphide gas. Prepare fresh as needed.

5.3.6 *Ethanol* — 95 percent (*v/v*)

5.4 Procedure

5.4.1 Weigh 2.500 g of sample, dissolve in 25 ml of dilute nitric acid and evaporate to syrupy consistency. Add 50 ml of hot water and allow to stand on a steam bath for one hour. If there is any opalescence or precipitate, add paper pulp, filter and wash several times with acidulated water. Reserve the filtrate.

5.4.1.1 Transfer the residue to silica crucible and ignite. Add 10 ml to 12 ml of hydrobromic acid, evaporate to dryness and ignite. Take up residue with dilute nitric acid and boil to expel brown fumes. Add to the filtrate reserved under 5.4.1. Repeat the hydrobromic acid treatment till tin is completely volatilized.

5.4.1.2 Adjust the volume of the solution to 150 ml. Add 0.5 g of urea or 0.1 g of sulphamic acid and boil for a few minutes. Insert the tared electrodes, cover the beaker with split watch-glasses. Electrolyse with a current of 5 A/dm^2, with constant stirring. When the solution is colourless, wash down the cover glasses, electrodes and sides of the beaker, raising the level of liquid slightly. Continue passing the current noting whether or not copper is being plated on the newly exposed surface of platinum cathode. If no copper appears, transfer about one millilitre of the solution to a spot plate and test for copper with a few drops of freshly prepared acidified hydrogen sulphide solution.

5.4.1.3 As soon as the deposition is complete, lower the beaker slowly while washing the cathode with water without stopping the current. Remove the cathode, rinse it with water and then dip in two successive baths of ethanol. Dry for three minutes to five minutes in an oven at 105 ºC, cool and weigh the deposit immediately as metallic copper. Remove the anode, rinse thoroughly with water, and dry the anode in an oven at 100 °C for 30 minutes. The deposit, being fragile, should be handled with care. Cool the anode and weigh as lead peroxide.

5.5 Calculation

$$\text{Copper, percent} = \frac{A}{C} \times 100$$

$$\text{Lead, percent} = \frac{B \times 86.62}{C}$$

where

A = mass, in g, of the copper deposit;

B = mass, in g, of the lead peroxide deposit; and

C = mass, in g, of the sample taken.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

Organization	*Representative(s)*
CSIR - National Metallurgical Laboratory, Jamshedpur	DR SANCHITA CHAKRAVARTY **(*Chairperson*)**
Arcelor Mittal Nippon Steel, Mumbai	SHRI MANOJ GUPTA SHRI KIRIT TAILOR (*Alternate*)
Bhabha Atomic Research Centre, Mumbai	MS SANJUKTA A. KUMAR SHRI M. V. RANA (*Alternate*)
CSIR - National Metallurgical Laboratory, Jamshedpur	DR ASHOK K. MOHANTY (*Alternate*)
Defence Metallurgical Research Laboratory, Ministry of Defence, Hyderabad	SHRI S. S. KALYAN KAMAL
Directorate General of Quality Assurance, Ministry of Defence, New Delhi	SHRI A. MITRA SHRI D. KARTIKEY (*Alternate*)
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Tata Steel Limited, Kolkata	DR JATIN MOHAPATRA DR RAVIKRISHNA CHATTI (*Alternate*)
BIS Directorate General	SHRI SANJIV MAINI, SCIENTIST 'F'/SENIOR DIRECTOR AND HEAD (METALLURGICAL ENGINEERING) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary
SHRI ASHISH PRABHAKAR WAKLE
SCIENTIST 'C'/DEPUTY DIRECTOR
(METALLURGICAL ENGINEERING), BIS

Bureau of Indian Standards

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

This Indian Standard has been developed from Doc No.: MTD 34 (21051).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi