भारतीय मानक

**कागज और लुगदी आधारित पैकेजिंग सामग्री के परीक्षण के तरीके**

**भाग 3**

**आर्सेनिक, संपूर्ण कॉपर, संपूर्ण आयरन, पानी में घुलनशील कॉपर और पानी में घुलनशील आयरन** (*दूसरा पुनरीक्षण*)

Indian Standard

**Methods of Test for Paper and Pulp Based Packaging Materials**

**Part 3**

**Arsenic, Total Copper, Total Iron, Water Soluble Copper and Water Soluble Iron**

 *(Second Revision)*

ICS 85.080

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B U R E A U O F I N D I A N S T A N D A R D S

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January 2023 Price Group

Paper Based Packaging Materials Sectional Committee, CHD 16

FOREWORD

This Indian Standard (Part 3) (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Paper Based Packaging Materials Sectional Committee had been approved by the Chemical Division Council.

The packaging materials used in industry are many and varied. They are paper and paper products, textiles, metal and metal foils, plastics and a variety of laminates, wood, glass and ceramics, cushioning materials, strapping and hooping materials, nails, etc. Among these, paper and paper products are of major importance.

This standard (Part 3) of IS 4006 series of standards which cover methods of tests which are carried out for paper and pulp based packaging materials to evaluate their quality. Other standards of IS 4006 series are:

IS 4006 (Part 1) Methods of Test for Paper and Pulp Based Packaging Materials, Part 1

IS 4006 (Part 2) Methods of Test for Paper and Pulp Based Packaging Materials, Part 2

This standard was first published in 1978. In the first revision, spectrophotometric methods were also incorporated for the determination of copper and iron which are faster and accurate.

This revision has been brought out to bring the standard in the latest style and format of Indian Standards. Reference standard for spectrophotometric method for determination of iron has been updated.

The composition of technical committee responsible for formulation of this standard is listed in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 rules for rounding off numerical values.

*Indian Standard*

METHODS OF TEST FOR PAPER AND PULP BASED PACKAGING MATERIALS

**PART 3 ARSENIC, TOTAL COPPER, TOTAL IRON, WATER SOLUBLE COPPER AND WATER SOLUBLE IRON**

*( Second Revision )*

**1 SCOPE**

**1.1** This standard (Part 3) prescribes methods of test for the determination of the following in paper and pulp based packaging materials:

a) Arsenic content,

b) Total copper content,

c) Total iron content,

d) Water soluble copper content, and

e) Water soluble iron content.

**1.2** If there is any inconsistency between the requirements of this standard and those of the standard for an individual material, the later shall prevail.

**2 REFERENCES**

The following standards contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

|  |  |
| --- | --- |
| *IS No.* | *Title* |
| IS 1060 (Part 1) : 2022 | Methods of sampling and test for paper and allied products: Part 1 Test methods for general purpose |
| IS 1070 : 1992 | Reagent grade water — Specification  |
| IS 2088 : 1983 | Methods for determination of arsenic  |
| IS 4261 : 2001 | Glossary of terms relating to paper and pulp based packaging materials  |
| IS 7212 : 1974 | Methods of determination of copper |
| IS 15556 : 2005 | Volumetric and spectrophotometric estimation of iron |

**3 TERMINOLOGY**

**3.1** For the purpose of this standard, the definitions given in IS 4261 shall apply.

**4 SAMPLING**

**4.1** Representative samples for test shall be drawn as prescribed in **4** of IS 1060 (Part 1).

**5 QUALITY OF REAGENTS**

**5.1** Unless otherwise specified, pure chemicals and distilled water (*see* IS 1070) freshly boiled and cooled, shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

**6 ARSENIC CONTENT**

**6.1 Procedure**

**6.1.1** Weigh 5 g of the sample, cut into strips, fold and place in a Kjeldahl flask, which shall be used only for this purpose. Add 20 ml of sulphuric acid. Warm very gently till the initial reaction is over and then more strongly, adding nitric acid, 1 ml at a time, whenever white fumes indicate that an excess of nitric acid is no longer present. Care shall be taken that fuming of the acid does not start until all the organic matter is destroyed. Continue till the paper is completely oxidized. When further addition of nitric acid produces no further change in the yellow or green solution, cool, carefully dilute to 50 ml with water and re-evaporate until copious white fumes are evolved. Repeat the operation of dilution and evaporation to ensure that the nitric acid has been entirely removed.

**6.1.2** Determine the arsenic content, using the working solution prepared in **6.1.1**, as prescribed in IS 2088.

**6.1.3** In case of dispute the spectrophotometric method using silver diethyldithiocarbamate as prescribed in IS 2088, shall be used as the referee method for determination of arsenic content.

**7 DETERMINATION OF TOTAL COPPER**

**7.1 General**

Two methods have been prescribed for the determination of total copper. Method A is the routine method and Method B is the referee method.

**7.2 Method A**

**7.2.1** *Reagents*

**7.2.1.1** *Citric acid*

**7.2.1.2** *Ammonium hydroxide solution* — relative density 0.90.

**7.2.1.3** *Sodium diethyldithiocarbamate* — 0.2 percent solution, freshly prepared.

**7.2.1.4** *Standard copper solution* — 1 ml = 0.01 mg of copper.

**7.2.2** *Procedure*

**7.2.2.1** Prepare a working solution by wet ashing 5 g of the sample as specified in **6.1.1** for determination of arsenic. Dilute to 100 ml.

**7.2.2.2** Take 20 ml or other suitable aliquot of the working solution and add 1 g of citric acid and one or two drops of thymol blue indicator. Make alkaline with ammonia solution. Make up to 50 ml in a Nessler cylinder. Add 5 ml of 0.2 percent solution of sodium diethyldithiocarbamate. Match any yellow colour produced against a standard solution of copper which has been treated in the same way. Make a blank test on the reagents.

**7.3 Method B**

Determine copper by the spectrophotometric method as prescribed in **7** of IS 7212.

**8 DETERMINATION OF TOTAL IRON**

**8.1 General**

Two methods have been prescribed for the determination of total iron. Method A is the routine method and Method B is the alternate referee method.

**8.2 Method A**

 **8.2.1** *Reagents*

**8.2.1.1** *Hydrochloric acid* — 20 percent v/v.

**8.2.1.2** *Thioglycollic acid* — 5 percent solution.

**8.2.1.3** *Ammonium hydroxide solution* — relative density 0.90.

**8.2.1.4** *Standard iron solution*

Prepared from ammonium ferrous sulphate oxidized with potassium permanganate solution and hydrochloric acid (1 ml = 0.005 mg of iron).

**8.2.2** *Procedure*

Prepare ash 5 g of the sample in a platinum dish at a temperature not exceeding 600 °C. Evaporate the residue with 2 ml of 20 percent hydrochloric acid on a water-bath. Make up to 100 ml in a measuring flask. Transfer 25 ml or other suitable aliquot to a 50 ml Nessler cylinder. Add 5 ml of 5 percent aqueous solution of thioglycollic acid; make alkaline with ammonia solution. Dilute to 50 ml with water. Allow to stand for 5 min. Match the resulting colour against a series of standard iron solutions treated in the same way. Make a blank test on the reagents used.

**8.3 Method B**

Determine iron by the spectrophotometric method as prescribed in IS 15556.

**9 DETERMINATION OF WATER-SOLUBLE COPPER**

**9.1 Reagents**

**9.1.1** *Sulphuric Acid* — relative density 1.84.

**9.1.2** *Sodium Nitrate*

**9.1.3** *Sulphuric Acid* — 20 percent of v/v.

**9.2** **Preparation of Working Solution**

**9.2.1** Weigh exactly 5 g of the sample and cut into strips about 2 cm wide and of any convenient length. Fold these in zig-zag fashion so. as to have a little bundle 2 cm high when arranged on edge side by side in a 250 m1 beaker, thus preventing packing but exposing a maximum stable surface to the action of the solvent, which is distilled water. Extract with 4 successive 100 ml portions each for 30 min at 85 °C to 95°C to give a total volume of 400 ml and taking a total time of 2 h.

**9.2.2** Filter each washing immediately through a filter using a sintered glass filter. This washing can be rapidly evaporated in a weighed flat bottomed dish of platinum, porcelain, or silica, while the next extraction proceeds.

**9.2.3** Evaporate to dryness on a water-bath and heat for 2 h in an air-oven at 105 °C ± 1 °C.

**9.2.4** Dissolve in hot water free from iron and copper, and transfer to a 50 m1 beaker. Evaporate to dryness and char gently. Add 20 ml of concentrated sulphuric acid and heat carefully until fuming strongly, then add sodium nitrate gradually until the last traces of carbonaceous matter have been cleared away. About 2 g of sodium nitrate will normally be needed. When perfectly clear, cautiously evaporate the acid to dryness, the mineral residue being allowed to cool. Take with 10 ml of 20 percent sulphuric acid, warm and dilute with water making up to 50 ml of working solution.

NOTE — This method of preparation of the sample is necessary in order to avoid loss of volatile water-soluble copper and iron-salts. Preparation of sample by the more obvious method of `ashing' tends to give a low result.

**9.3 Procedure**

Take 20 ml or other suitable aliquot of the working solution and proceed as described in **7.2**.

**9.4** Alternatively, the spectrophotometric method as prescribed in **7** of IS 7212 may be followed.

**10 DETERMINATION OF WATER SOLUBLE IRON**

**10.1** Take 5 ml or other suitable aliquot of the working solution prepared in **9.2.4** in 50 ml Nessler cylinder and proceed as described in **8.2**.

**10.2** Alternatively, determination of iron by suitable spectrophotometric method.

**ANNEX A**

(*Foreword*)

**COMMITTEE COMPOSITION**

Paper Based Packaging Materials Sectional Committee, CHD 16

| *Organization* | *Representative(s)* |
| --- | --- |
|
| Indian Institute of Packaging, Mumbai |  Dr Tanweer Alam (***Chairperson***) |
| B&A Packaging(I) Limited, Kolkata  | Shri Amal Kumar MohantyShri Tapan Kr Chand (*Alternate*) |
| Century Pulp & Paper Limited | Shri Sanjay Kumar Yadav Shri Sunesh Yadav (*Alternate*) |
| CPPRI, Saharanpur  |  Dr M. K. GuptaDr Sanjay Tyagi (*Alternate*) |
| Dr Reddy Laboratories | Shri Avinash TalwarShri Vinay Kr Singh (*Alternate*) |
| Federation of Corrugated Box Manufacturers of India, Mumbai | Shri Satish TyagiShri K. P. Singh (*Alternate*) |
| Federation of paper convertors of India  | Shri Mukesh GuptaShri Sushil Kumar Singh (*Alternate*) |
|  Hersheys India Private Limited  | Ms Deepa Mandar Naik |
| Indian Agro & Recycled Paper Mills Association, New Delhi | Dr B. P. Thapliyal Dr Anil Naithani (*Alternate*) |
| Indian Institute of Packaging  | Shri Madhab Chakraborty |
| Indian Institute of Technology, Roorkee  | Dr Dharm Dutt Dr Kirtiraj K. Gaikwad (*Alternate* I)Dr Vibhor Kumar Rastogi (*Alternate* II) |
| Indian Paper Manufacturers Association, New Delhi | Shri Biswaranjan Dash Shri Rohit Pandit (*Alternate*) |
| ITC, Bhadrachalam  | Shri Chidambara VinayagamShri P. N. Sridharr (*Alternate*) |
| ITC Life Sciences, Bangalore  | Shri Ajith KumarDr Kamal (*Alternate*) |
| J K Paper  | Shri Umakant Patil Shri Aayush Raj Srivastava (*Alternate*) |
| Nestle India Limited  | Shri Barun BanerjeeShri Deepak Singh (*Alternate*) |
| Package Design Research and Test Lab, Moradabad | Shri L. M. GuptaShri Mayank Gupta (*Alternate*) |
| Parksons Packaging | Shri Srikanth RamamurthyShri Pawan Kumar Singh *(Alternate*) |
| Prem Industries  | Shri Alok GoelShri H. P. Singh (*Alternate*) |
| Safepack Industries Limited | Shri Rajendra TapadiaShri Anand Tapadia (*Alternate*) |
| Siegwerk | Shri Jatin ThakkarPriyanka Baweja (*Alternate*) |
| Stora Enso | Shri Sanjeev Khandelwal |
|  BIS Directorate General | Shri A. K. Lal, Scientist ‘E’/Director and Head (Chemical) [Representing Director General (*Ex-officio*)] |

*Member Secretary*

Ms Shrishti Dixit

Scientist ‘D’/Joint Director

(Chemical), BIS