भारतीय मानक

Indian Standard

IS 4006 (Part 3) : 2024

कागज़ और लुगदी आधारित पैकेजिंग सामग्री — परीक्षण पद्धतियाँ

भाग 3 आर्सेनिक, संपूर्ण कॉपर, संपूर्ण आयरन, पानी में घुलनशील कॉपर और पानी में घुलनशील आयरनण

(दूसरा पुनरीक्षण)

Paper and Pulp Based Packaging Materials — Methods of Test

Part 3 Arsenic, Total Copper, Total Iron, Water Soluble Copper and Water Soluble Iron

(Second Revision)

ICS 85.080

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October 2024 **Price Group 4**

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 the 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 part of IS 4006 series are:

Part 1	Flexural Resistance and Deflection, Waterproofness, Water Penetration, Grease Resistance, Abrasion Loss, Blocking Resistance, Compression Resistance, Rigidity, Stiffness and Softness; and Air Permeance (*second revision*) CHD 16 (20530) F
Part 2	Odour, Ply Separation, Puncture, and Reducible Sulphur (*second revision*) CHD 16 (20531) F

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

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off 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 the same as that of the specified value in this standard.

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 standards given below contain provisions which, through reference in this text, constitute provision 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 edition of these standards:

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 (*second revision*)
IS 2088 : 2023	Methods for determination of arsenic (*third revision*)
IS 4261 : 2001	Glossary of terms relating to paper and pulp based packaging materials (*first revision*)
IS 7212 : 2023	Methods of determination of copper (*first revision*)
IS 15556 : 2005	Volumetric and spectrophotometric estimation of iron

3 TERMINOLOGY

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

4 SAMPLING

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

5 QUALITY OF REAGENTS

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 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 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) Ltd Kolkata	SHRI AMAL KUMAR MOHANTY SHRI TAPAN KR. CHAND (*Alternate*)
Century Pulp & Paper Limited	SHRI SANJAY KUMAR YADAV SHRI SUNESH YADAV (*Alternate*)
CPPRI, Saharanpur	DR M. K. GUPTA DR SANJAY TYAGI (*Alternate*)
Dr Reddy laboratories	SHRI AVINASH TALWAR SHRI VINAY KR SINGH (*Alternate*)
Federation of Corrugated Box Manufacturers of India, Mumbai	SHRI SATISH TYAGI SHRI K. P. SINGH (*Alternate*)
Federation of paper convertors of India	SHRI MUKESH GUPTA SHRI SUSHIL KUMAR SINGH (*Alternate*)
Hersheys India Pvt Ltd	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*)
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Tetra Pak India Pvt Ltd	SHRI SHARAD SHARMA SHRI JAIDEEP GOKHALE (*Alternate*)
Uflex Limited	SHRI ASHVANI SHARMA SHRI RAKESH SHARMA (*Alternate*)
BIS Directorate General	SHRI AJAY KUMAR LAL, SCIENTIST 'F'/SENIOR DIRECTOR AND HEAD (CHEMICAL) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary
SHRISHTI DIXIT
SCIENTIST D/JOINT DIRECTOR
(CHEMICAL), BIS

Bureau of Indian Standards

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