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METHODS OF TEST FOR RUBBER LATEX

PART 7 DETERMINATION OF TOTAL COPPER

[RL : 7]

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METHODS OF TEST FOR RUBBER LATEX

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[RL : 7]

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Indian Standard

METHODS OF TEST FOR RUBBER LATEX

PART 7 DETERMINATION OF TOTAL COPPER

[RL : 7]

0. FOREWORD

0.1 This Indian Standard (Part 7) was adopted by the Indian Standards Institution on 27 February 1987, after the draft finalized by the Rubber Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Test methods for rubber latex had been originally covered in the following Indian Standards:

a) *For Natural Rubber Latex*:

IS : 3708 (Part 1)-1966*

IS : 3708 (Part 2)-1968†

b) *For Styrene Butadiene Rubber Latex*:

IS : 4511 (Part 1)-1967‡

Since some of the test methods covered in above standards were common, the concerned committee had decided some years ago to unify and publish a separate series of methods of test which would be applicable to all types of latices — natural as well as synthetic. Accordingly, the following six text methods had been covered under IS : 9316:

IS : 9316 Methods of test for rubber latex:

Part 1-1979 Determination of surface tension

*Methods of test for natural rubber latex: Part 1 Dry rubber content, sludge content density, total alkalinity, KOH-number, mechanical stability, volatile fatty acid number *p*H, total nitrogen, total copper, total iron, total manganese and total ash.

†Methods of test for natural rubber latex, Part 2.

‡Methods of tests for styrene-butadiene rubber (SBR)latices: Part 1 Determinatior of dry polymer, *p*H, density, residual styrene, bound styrene and soap content.

Part 2-1979 Determination of viscosity
Part 3-1979 Determination of coagulum content
Part 4-1979 Determination of total solids content
Part 5-1979 Drawing of samples
Part 6-1982 Determination of *p*H

0.2.1 As a result of further rethinking on the subject, it has now been decided to re-designate the test methods common to natural and synthetic rubber latices as RL series; test methods for natural rubber latex as NRL series and test methods for styrene-butadiene rubber latex as SBRL series. Consequently, test methods for rubber latex have been rationalized into the following three series:

a) IS : 9316 Unified methods of test applicable to both natural and synthetic rubber latices — RL series;

b) IS : 3708 Methods of test applicable to natural rubber latex — NRL series; and

c) IS : 4511 Methods of test applicable to styrene-butadiene rubber latex — SBRL series.

0.3 The existing Indian Standards under IS : 3708 (Parts 1* and 2†), IS : 4511 (Part 1‡) and IS : 9316 (Parts 1 to 6) are being gradually replaced by separate standards under the above three series, designated by the appropriate NRL, SBRL, or RL series respectively.

0.3.1 This method, originally covered under NRL : 13 of IS : 3708 (Part 1)-1966, is now being covered in this standard.

0.4 In order to faciliate cross-reference, it has been decided to retain the original discrete NRL, SBRL and RL series numbers assigned to various test methods, in IS : 3708 (Parts 1* and 2†), IS : 4511 (Part 1‡) and IS : 9316 (Parts 1 to 6) in the revised Parts of IS : 3708, and IS : 4511 and IS : 9316 respectively.

0.4.1 For proper referencing of the existing test methods and the new methods under revision, a statement showing corresponding methods is given in Appendix A.

---

*Methods of test for natural rubber latex: Part 1 Dry rubber content, sludge content, density, total alkalinity, KOH-number, mechanical stability, volatile fatty acid number, *p*H, total nitrogen, total copper, total iron, total manganese and total ash.

†Methods of test for natural rubber latex, Part 2.

‡Methods of tests for styrene-butadiene rubber (SBR) latices: Part 1 Determination of dry polymer, *p*H, density, residual styrene, bound styrene and soap content.

0.5 In preparing the above series, the need to align the test methods with the corresponding ISO Standards DIS/DP wherever available has also been taken into account for updating the test methods. In the preparation of this standard, assistance has been derived from ISO/R 1654-1971 (E) 'Raw rubber and rubber latex — Determination of copper', issued by the International Organization for Standardization (ISO).

0.6 The estimation of copper in natural rubber latex is of importance in avoiding danger of active contamination leading to degradation in the final rubber. Copper in certain forms is known to catalyse the oxidative breakdown of natural rubber although the mechanism by which degradation is brought about is not fully understood. It is recognized also that other forms of copper may be present without degradation taking place, but no generally accepted method is available for distinguishing between the active and inactive forms. At present, therefore, there is no alternative to determining the total amount of copper in rubber.

0.6.1 Little is known concerning the influence of copper on the catalytic oxidation of synthetic rubbers, although it is widely accepted that its effect is less severe than is the case with natural rubber. Possibly for this reason the determination of copper in synthetic rubbers is less frequently carried out; nevertheless this Indian Standard is applicable to most of the commonly used synthetic elastomers.

0.7 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 : 2-1960*.

1. SCOPE

1.1 This standard (Part 7) prescribes a method for the quantitative determination of total copper content in uncompounded natural rubber latex and uncompounded synthetic rubber latex which do not contain chlorine.

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

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

*Rules for rounding off numerical values (*revised*).
†Specification for water for general laboratory use (*second revision*).

3. OUTLINE OF THE METHOD

3.1 Five grams of the dried latex solids are ashed in a silica crucible. The ash is extracted with a hydrochloric-nitric acid mixture and the solution made alkaline with ammonium hydroxide. Any iron present is complexed with ammonium citrate. The aqueous solution is then shaken with solution in chloroform of zinc diethyldithiocarbamate to form and extract the yellow copper complex. The optical density of this solution is measured photometrically and is proportional to the concentration of copper.

4. APPARATUS

4.1 Electrophotometer, Absorptiometer, or Spectrophotometer — capable of measuring optical density at approximately 435 nm.

4.2 Matched Absorption Cells — 10 to 50 mm in path length.

4.3 Silica Crucibles — nominal capacity 50 or 80 ml.

4.4 Muffle Furnace — capable of maintaining a temperature of 550 ± 25°C

5. REAGENTS

5.1 Light Magnesium Oxide

5.2 Sodium Sulphate Anhydrous

5.3 Hydrochloric Acid — Nitric Acid Mixture

2 volumes of hydrochloric acid (*see* IS : 265-1976*)

1 volume of nitric acid (*see* IS : 264-1976†), and

3 volumes of water.

5.4 Citric Acid Solution — Dissolve 50 g of citric acid (solid) in 100 ml of water.

5.5 Ammonia Solution — density 0·890 g/ml.

*Specification for hydrochloric acid (*second revision*).
†Specification for nitric acid (*second revision*).

5.6 Litmus Paper

5.7 Zinc Diethyldithiocarbamate Reagent — Dissolve 1 g of solid zinc diethyldithiocarbamate in one litre of chloroform. If zinc diethyldithiocarbamate is not available the reagent may be prepared as follows:

Dissolve 1 g of sodium diethyldithiocarbamate in water and add 2 g of zinc sulphate. Extract the resulting zinc diethyldithiocarbamate by shaking with 100 ml of chloroform and separate the chloroform solution. Dilute to one litre. Store in an amber coloured bottle; this reagent is stable for at least six months.

5.8 Standard Copper Solution — Weigh 0·393 g of copper sulphate pentahydrate ($CuSO_4.5H_2O$) into a small beaker and dissolve in water. Add 3 ml of concentrated sulphuric acid. Transfer the solution to a 1 000-ml one-mark volumetric flask and dilute to the mark with water to form the stock solution. Pipette 10 ml of this stock solution into a 100-ml one-mark volumetric flask and dilute to the mark with water. This solution contains the equivalent of 0·01 mg of copper per millilitre and should be freshly prepared from the stock solution when required.

6. PROCEDURE

6.1 Preparation of Sample — For the determination of copper in latex, take a portion of thoroughly mixed latex containing about 5 g of total solids and dry to constant mass as described in total solids determination [*see* IS : 9316 (Part 4)-1979*]. Homogenize the rubber so obtained by passing it a few times between the cold rolls of a laboratory mill to produce a thin sheet. At all stages of sample preparation, care should be taken to avoid contamination.

6.2 Preparation of Calibration Curve — If the magnesium oxide method of ashing is to be used, prepare a series of standard solutions, each containing 0·1 g of magnesium oxide dissolved in 10 ml of the dilute hydrochloric acid-nitric acid mixture. If the rubber is to be ashed by wrapping in filter paper and then placing directly in the muffle furnace, omit the magnesium oxide. To these solutions add portions of the standard copper solution ranging from 0 to 10 ml followed in each case by 5 ml of citric acid solution. Add ammonia solution drop by drop until the solutions are just alkaline to litmus paper. Cool the solutions, transfer individually to a separating funnel and add a further 2 ml of ammonia solution to each. Pipette 25 ml of

*Methods of test for rubber latex: Part 4 Determination of total solids.

zinc diethyldithiocarbamate reagent into each solution and shake for 2 minutes. Immediately after separation, draw the chloroform layer into a stoppered flask containing about 0·1 g of anhydrous sodium sulphate. If turbidity persists after standing for about 30 minutes, make further small additions of anhydrous sodium sulphate until the solution becomes clear.

6.2.1 Decant each chloroform solution through a plug of glass wool or a small filter paper into the cell of the electrophotometer, absorptiometer or spectrophotometer, and measure the optical density at the absorption maximum (about 435 nm). Correct the reading by subtracting the optical density of the solution containing no added copper. If the optical density is measured on a double beam or null point instrument, the cell containing the blank solution should be placed in the reference beam and the optical density of each standard solution measured against that of the solution containing no added copper. Plot the reading thus obtained for each solution against the appropriate concentration of copper to give the calibration curve, which should be checked periodically according to local conditions and the type of instrument used.

6.3 Ashing — Weigh, to the nearest 5 mg, a 5 g test portion of the dried latex film, and place in a silica crucible containing 0·1 g of magnesium oxide distributed over the base and partly up the side of the crucible. Support the crucible in a hole cut in an asbestos board so that about two-thirds of the crucible projects below the asbestos. Commence a blank determination using a similar crucible and the same amount of magnesium oxide, and give identical treatment throughout to the test and blank determinations. Heat the crucible and contents with a small gas flame until a dry carbonaceous residue remains, and then transfer the crucible to a muffle furnace at a temperature of 550 ± 25°C and heat until all carbon has been oxidized. Remove the crucible and allow to cool.

6.3.1 As an alternative to the above method of ashing, wrap the 5 g test portion, weighed to the nearest 5 mg, in a piece of ashless filter paper about 150 mm in diameter and place in a transparent silica crucible having clean unetched walls. Place the crucible and contents in furnace at 550 ± 25°C and close the door. Because of the risk of igniting flammable gases the furnace door should not be opened during the first hour. Also commence a blank determination using a similar filter paper and crucible, and give identical treatment throughout to the test blank determinations. When all the carbon has been oxidized, remove the crucible and allow it to cool.

6.4 Moisten the contents of the crucible with 0·5 to 1 ml water, then add 10 ml of the dilute hydrochloric acid-nitric acid mixture and heat

the crucible, covered with a clockglass, on a steam bath for 30 to 60 minutes. Wash the contents of the crucible into a small beaker or flask, add 5 ml of citric acid solution and then add ammonia solution drop by drop until the solution is just alkaline to litmus paper. Cool the solution, for example, by immersion in running water, tranfer to a separating funnel, add a further 2 ml of ammonia solution and then dilute to about 40 ml with water. Pipette 25 ml of zinc diethyldithiocarbamate reagent into the solution and shake the funnel for 2 minutes. After separation, immediately transfer the chloroform layer into a stoppered flask containing about 0·1 g of anhydrous sodium sulphate. If turbidity persists after standing for about 30 minutes, make further small additions of anhydrous sodium sulphate until the solution becomes clear.

6.5 Photometric Measurement of Colour — Decant the chloroform solution through a plug of glass wool or a small filter paper into the cell of the electrophotometer, absorptiometer or spectrophotometer, and measure the optical density at the wavelength used in preparing the calibration curve (about 435 nm). Correct the reading by subtracting the optical density of the blanks solution. If the optical density is measured on a double beam or null point instrument, the cell containing the blank solution should be placed in the reference beam and the optical density of the test solution measured against that of the blank. The reading thus obtained, used in conjunction with the calibration curve, gives the concentration of copper in the test solution and hence in the rubber.

NOTE — All precautions and safeguards required for carrying out of trace metal analysis shall be observed.

7. EXPRESSION OF RESULT

7.1 The result should be expressed as parts per million (ppm) of copper (Cu) calculated by mass.

APPENDIX A

(*Clause* 0.4.1)

TABLE SHOWING CORRESPONDENCE OF THE VARIOUS METHODS OF TEST COVERED IN THE EXISTING IS : 9316 (PARTS 1 TO 5)-1979, IS : 9316 (PART 6)-1982, IS : 3708 (PART 1)-1966, IS : 3708 (PART 2)-1968, IS : 4511 (PART 1)-1967 *WITH* THE REVISION PROPOSED REVISION OF IS : 9316, IS : 3708 AND IS : 4511

Existing Test Methods			Proposed Revisions		Remarks
Test Method	IS No.	Part (Series)	IS No.	Part (Series)	
(1)	(2)	(3)	(4)	(5)	(6)
RL Series					
Determination of surface tension	IS : 9316-1979	Part 1	IS : 9316	Part 1 (RL : 1)	
Determination of viscosity	IS : 9316-1979	Part 2	IS : 9316	Part 2 (RL : 2)	
Determination of coagulum content	IS : 9316-1979	Part 3	IS : 9316	Part 3 (RL : 3)	
Determination of total solids content	IS : 9316-1979	Part 4	IS : 9316	Part 4 (RL : 4)	Under revision
Drawing of samples	IS : 9316-1979	Part 5	IS : 9316	Part 5 (RL : 5)	Under revision
Determination of *p*H	IS : 9316-1982	Part 6	IS : 9316	Part 6 (RL : 6)	Under revision
Determination of total copper	IS : 3708-1966	Part 1 (NRL : 13)	IS : 9316	Part 7 (RL : 7)	
Determination of total iron	IS : 3708-1966	Part 1 (NRL : 14)	IS : 9316	Part 8 (RL : 8)	
Determination of total manganese	IS : 3708-1966	Part 1 (NRL : 15)	IS : 9316	Part 9 (RL : 9)	
NRL Series					
Determination of dry rubber content	IS : 3708-1966	Part 1 (NRL : 1)	IS : 3708-1985	Part 1 (NRL : 1)	
Determination of sludge content	IS : 3708-1966	Part 1 (NRL : 5)	IS : 3708-1985	Part 2 (NRL : 5)	
Determination of density	IS : 3708-1966	Part 1 (NRL : 6)	IS : 3708-1985	Part 3 (NRL : 6)	
Determination of total alkalinity	IS : 3708-1966	Part 1 (NRL : 7)	IS : 3708-1985	Part 4 (NRL : 7)	

Determination of KOH-number	IS : 3708-1966	Part 1 (NRL : 8)	IS : 3708-1985	Part 5 (NRL : 8)
Determination of mechanical stability	IS : 3708-1966	Part 1 (NRL : 9)	IS : 3708-1985	Part 6 (NRL : 9)
Determination of volatile fatty acid number	IS : 3708-1966	Part 1 (NRL : 10)	IS : 3708-1986	Part 7 (NRL : 10)
Determination of total nitrogen	IS : 3708-1966	Part 1 (NRL : 12)	IS : 3708	Part 8 (NRL : 12)
Determination of total ash	IS : 3708-1966	Part 1 (NRL : 16)	IS : 3708-1986	Part 9 (NRL : 16)
Determination of boric acid	IS : 3708-1968	Part 2 (NRL : 17)	IS : 3708	Part 10 (NRL : 17)
Determination of magnesium	IS : 3708-1968	Part 2 (NRL : 18)	IS : 3708	Part 11 (NRL : 18)
SBRL Series				
Determination of dry polymer	IS : 4511-1967	Part 1 (SBRL : 1)	IS : 4511-1986	Part 1 (SBRL : 1)
Determination of density	IS : 4511-1967	Part 1 (SBRL : 6)	IS : 4511	Part 2 (SBRL : 6)
Determination of volatile unsaturates	IS : 4511-1967	Part 1 (SBRL : 8)	IS : 4511	Part 3 (SBRL : 8)
Determination of soap content	IS : 4511-1967	Part 1 (SBRL : 9)	IS : 4511	Part 4 (SBRL : 9)
Determination of bound styrene	IS : 4511-1967	Part 1 (SBRL : 10)	IS : 4511	Part 5 (SBRL : 10)
Determination of high-speed mechanical stability	—	—	IS : 4511	Part 6 (SBRL : 11)

(*Continued from page* 2)

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