IS : 9316 (Part 9) - 1987

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Indian Standard

METHODS OF TEST FOR RUBBER LATEX

PART 9 DETERMINATION OF TOTAL MANGANESE

[RL : 9]

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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METHODS OF TEST FOR RUBBER LATEX

PART 9 DETERMINATION OF TOTAL MANGANESE

[RL : 9]

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Indian Standard

METHODS OF TEST FOR
RUBBER LATEX

PART 9 DETERMINATION OF TOTAL MANGANESE

[RL : 9]

0. FOREWORD

0.1 This Indian Standard (Part 9) 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:

For natural rubber latex

IS : 3708 (Part 1)-1966[*]

IS : 3708 (Part 2)-1968[†]

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 test methods had been covered under IS : 9316:

IS : 9316 Methods of test for rubber latex:

Part 1-1979 Determination of surface tension

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[*] 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 1655-1975 'Raw rubber and raw latex — Determination of manganese content — Potassium periodate photometric method', issued by the International Organization for Standardization (ISO).

0.6 The estimation of manganese in natural rubber latex is of importance in avoiding danger of active contamination leading to degradation in the final rubber. Manganese 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 recognised also that other forms of manganese 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 manganese in the rubber.

0.6.1 Little is known concerning the influence of manganese 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 manganese in synthetic rubbers is less frequently carried out; nevertheless, the method specified in this 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 9) prescribes a method for determination of total manganese 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.

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*Rules for rounding off numerical values (*revised*).

†Specification for water for general laboratory use (*second revision*).

3. OUTLINE OF THE METHOD

3.1 Dried latex solids are ashed in a crucible and the ash is treated with potassium hydrogen sulphate and sulphuric acid to convert the manganese to a soluble form. After dissolution of the ash in dilute sulphuric acid, iron present is complexed with orthophosphoric acid and the manganese oxidised to permanganate by boiling with potassium periodate. The absorbance of this solution is measured photometrically which is proportional to the concentration of manganese.

4. APPARATUS

4.1 Electrophotometer or Spectrophotometer — capable of measuring absorbance at approximately 525 nm.

4.2 Silica Crucibles — nominal capacity 80 ml.

4.3 Asbestos Board — approximately 100 mm square and 6 mm thick, with a hole in the centre to support the crucible so that approximately two-thirds project below the board.

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

5. REAGENTS

5.1 Potassium Hydrogen Sulphate

5.2 Potassium Periodate

5.3 Sulphuric Acid Concentrated — (*see* IS : 266-1977*).

5.4 Sulphuric Acid Dilute — Mix one volume of concentrated sulphuric acid with 19 volumes of water.

5.5 Orthophosphoric Acid — 85 to 90 percent H_3PO_4.

5.6 Stabilized Water — Dissolve about 0·1 g of potassium permanganate in one litre of water to which a few drops of sulphuric acid have been added. Distil the water through an effective spray trap, discarding the first and last 50 ml of distillate. Collect the rest of the distillate and store in a glass stoppered bottle.

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*Specification for sulphuric acid (*second revision*).

5.7 Potassium Permanganate — approximately 0·001 N solution.

5.8 Standard Manganese Solution — *Either of the following* solutions may be used:

a) Prepare an approximately 0·1 N solution of potassium permanganate and standardize against sodium oxalate. *Transfer the calculated amount of this standardized solution* to contain 0·720 g of $KMnO_4$ to a small beaker and acidify with 2 *ml* of sulphuric acid. Add sulphur dioxide-saturated water until the solution is colourless. Boil the solution for 15 minutes, cool, transfer to a 500-ml volumetric flask and dilute to the mark with stabilized water. Pipette 20 ml of this stock solution into a second 500-ml volumetric flask and again dilute to the mark with stabilized water. This dilute solution contains the equivalent of 0·02 mg of manganese per ml and shall be *freshly prepared from the stock solution* when required.

b) Weigh 0·770 g of manganese sulphate ($MnSO_4.H_2O$) into ~~a~~ a small beaker and dissolve in water containing 2 ml of sulphuric acid. *Transfer the solution to a 500-ml volumetric* flask and dilute to the mark. This solution is stable for at least a month. Pipette 20 ml of this stock solution into a second 500-ml volumetric flask and again dilute to the mark. This dilute solution contains the equivalent of 0·02 mg of manganese per ml and shall be freshly prepared from the stock solution when required.

6. DETERMINATION OF MANGANESE

6.1 Preparation of Test Portion — For the determination of manganese in latex, take a portion of thoroughly mixed latex containing about 5 g of total solids and dry to constant mass as specified in 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.

NOTE — At all stages of sample preparation, take care to avoid contamination of the rubber.

6.2 Preparation of Calibration Curve

6.2.1 Prepare a series of standard matching solutions, each containing 20 ml of dilute sulphuric acid, 3 ml of orthophosphoric acid and potassium hydrogen sulphate equal in amount to that used in ashing the test portion. *To these solutions, add* portions of the

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

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

standard manganese solution ranging from 0 to 15 ml, followed in each case by 0·3 g of potassium periodate. Bring the solutions to the boil, maintain at boiling point for 10 minutes, and hold at 90°C or above for a further 10 minutes to ensure full development of the permanganate colour. Cool each solution, transfer to a 50-ml volumetric flask and dilute to the mark with stabilized water.

6.2.2 Rinse the cell of the electrophotometer or spectrophotometer first with approximately 0·001 N potassium permanganate solution, then with stabilized water and finally with the appropriate standard matching solution. Fill the cell with the standard matching solution and measure the absorbance at the absorption maximum (about 525 nm). Correct the reading by subtracting the absorbance of the solution containing no added manganese. If the absorbance is measured on a double-beam or null-point instrument, place the cell containing the blank solution in the reference beam and measure the absorbance of each standard solution against that of the solution containing no added manganese.

6.2.3 Plot the reading thus obtained for each solution against the appropriate concentration of manganese to give the calibration curve, which shall be checked periodically according to local conditions and the type of instrument used.

6.3 Procedure

6.3.1 Weigh to the nearest 10 mg, about 5 g of the dried latex solids, cut into small pieces, and place in a crucible which is supported in the hole cut in the asbestos board. Heat the crucible and contents with a small gas flame until a dry carbonaceous residue remains and then transfer the crucible to the muffle furnace maintained at a temperature of 550 ± 25°C and heat until all carbon has been oxidized. Remove the crucible and allow to cool. Add concentrated sulphuric acid delivered drop by drop from a pipette with a fine jet, round the sides of the crucible in amount just sufficient to moisten the ash. Fume off the excess acid by gentle heating and replace the crucible in the muffle furnace at 550 ± 25°C to remove the last traces of carbon. After cooling, give the ash a further treatment with sulphuric acid, adding the acid as before from a pipette, but heating only until fuming ceases in order to retain the ash as far as possible in the form of sulphate. Cool the crucible. Add 2 to 3 g of potassium hydrogen sulphate and, supporting the crucible on the asbestos board, apply strong heat from a burner until a clear melt is obtained. Cool the crucible and contents.

6.3.2 As an alternative to the above method of ashing, wrap the 10 g portion, weighed to the nearest 10 mg, in a piece of ashless filter paper about 150 mm in diameter and place in a crucible. Place about 5 g of

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 : 4511-1986	Part 2 (SBRL : 6)
Determination of volatile unsaturates	IS : 4511-1967	Part 1 (SBRL : 8)	IS : 4511	Part 3 (SBRL : 8)
Determination of bound styrene	IS : 4511-1967	Part 1 (SBRL : 9)	IS : 4511	Part 4 (SBRL : 9)
Determination of soap content	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)

potassium hydrogen sulphate on top of the wrapped test portion, place the crucible in the furnace at 550 ± 25°C and close the door. Owing to the risk of ignition of flammable gases, the furnace door shall not be opened during the first hour. When all the carbon has been oxidized, allow the crucible and contents to cool.

6.3.3 In parallel with the determination and following the same procedure, carry out a blank test, using a similar crucible and filter paper and the same quantities of all the reagents as used for the determination. To the ash produced by either of the above procedures add 20 ml of dilute sulphuric acid, and heat the crucible on a steam-bath until the solid material is dissolved or loosened from the walls of the crucible. Wash the contents into a small beaker using a glass rod to dislodge undissolved solid and boil the solution gently until no more will dissolve. Filter the solution through a sintered glass filter pad into a small conical flask, and wash the filter and insoluble material with two or three portions of water. Add 3 ml of orthophosphoric acid to the solution in the flask, making further additions of 1 ml as may be necessary to remove any yellow colour due to iron. Add to the solution 0·3 g of potassium periodate, bring the solution to the boil and maintain at boiling point for 10 minutes. Hold the temperature above 90°C for a further 10 minutes to ensure full development of the permanganate colour. After cooling, transfer the solution to a 50-ml volumetric flask and dilute to the mark with stabilized water. After mixing, the colour should be stable for several hours; any tendency to fading indicates the incomplete removal of organic matter or chloride.

6.3.4 Rinse the cell of the electrophotometer, absorptiometer or spectrophotometer first with approximately 0·001 N potassium permanganate solution, then with stabilized water, and finally with the test solution. Fill the cell with the test solution and measure the absorbance at the wavelength used in preparing the calibration curve. Correct the reading by subtracting the absorbance of the blank solution. If the absorbance is measured on a double-beam or null-point instrument, place the cell containing the blank solution in the reference beam and measure the absorbance of the test solution against that of the blank.

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

7. EXPRESSION OF RESULTS

7.1 By means of the calibration curve, determine the concentration of manganese corresponding to the corrected reading and from this calculate the manganese content of the test portion. Express the result as parts per million (ppm) or percentage of manganese (Mn) 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 Revision		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)	
Determination of *p*H	IS : 9316-1982	Part 6	IS : 9316	Part 6 (RL : 6)	
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)	

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