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भारतीय मानक

कोल तार पिच — विशिष्टि

( दूसरा पुनरीक्षण )

*Indian Standard*

COAL TAR PITCH — SPECIFICATION

*( Second Revision )*

ICS 75.140

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

## FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Bitumen, Tar and Their Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Coal tar pitch serves as a valuable ingredient in the production of a number of waterproofing, protective and binding compounds employed in masonry, steel and timber structures. It is also used for waterproofing concrete structures, caulking of decks, as a saturant for roofing felts, as a binder for a variety of carbon products and coal briquettes, for damp-proof courses, flooring mastics and as a base for coal tar paints. Coal tar pitch to this specification is not suitable for formulation of quick drying black enamels nor for road construction, for which purposes IS 215 : 1995 'Road tar (*third revision*)' should be referred to.

Considering the importance of the subject, this standard was first published in 1951 and subsequently revised in 1961. Further revision was necessitated to incorporate special coal tar pitch grades and to modify the requirements of coal tar pitch of various grades prescribed in the existing version.

In this revision, the following modifications have been made:

- a) Scope has been modified to incorporate the requirements of special coal tar pitch grades.
- b) Requirements for various grades of coal tar pitch have been modified.
- c) Requirements for special coal tar pitch of three grades have been incorporated.

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 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

(Continued from second cover)

# Indian Standard

## COAL TAR PITCH — SPECIFICATION

### ( Second Revision )

#### 1 SCOPE

This standard covers the requirements, methods of sampling and test for the range of four grades of coal tar pitch from soft to hard consistencies with softening points varying from 45° to 95°C and for the range of three grades of special coal tar pitch from binder to extra hard consistencies with softening points varying from 86° to 130°C intended for the production of water proofing protective and binding compounds employed in masonry, steel, timber and concrete structures and also for the preparation of roofing felts, besides their use as binder in the production of a variety of carbon products such as graphite electrodes, Soderberg or pre-baked anodes and carbon or graphite blocks.

#### 2 REFERENCES

The following standards contain provision 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 indicated below:

IS No.	Title
334 : 2002	Glossary of terms relating to bitumen and tar ( <i>third revision</i> )
1201 : 1978 <sup>1)</sup>	Methods for testing tar and bituminous materials: Sampling ( <i>first revision</i> )
1202 : 1978 <sup>1)</sup>	Determination of specific gravity ( <i>first revision</i> )
1205 : 1978 <sup>1)</sup>	Determination of softening point ( <i>first revision</i> )
1215 : 1978 <sup>1)</sup>	Determination of matter insoluble in toluene ( <i>first revision</i> )
1217 : 1978 <sup>1)</sup>	Determination of mineral matter (Ash) ( <i>first revision</i> )
1220 : 1978 <sup>1)</sup>	Determination of volatile matter content ( <i>first revision</i> )
1350 (Part 3) : 1969	Methods of test for coal and coke: Part 3 Determination of sulphur ( <i>first revision</i> )
4284 : 1967	Method for volumetric determination of iron

<sup>1)</sup> IS 1201 : 1978 to IS 1220 : 1978 printed in one volume.

#### IS No.

#### Title

4542 : 1968	Colorimetric methods for determination of iron
9497 : 1980	Method for determination of sodium and potassium (flame photometric)
13320 : 1992	Determination of iron by atomic absorption spectrophotometry — Test method
13758	Coal tar pitch; Methods of test
(Part 1) : 1993	Determination of matter insoluble in quinoline
(Part 2) : 1993	Determination of coking value

#### 3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 334 shall apply.

#### 4 GRADES

##### 4.1 Coal Tar Pitch

The coal tar pitch shall be classified into the following four grades:

- a) Soft pitch,
- b) Soft medium pitch,
- c) Hard medium pitch, and
- d) Hard pitch.

##### 4.2 Special Coal Tar Pitch

The special coal tar pitch shall be classified into the following three grades:

- a) Binder pitch,
- b) Hard pitch, and
- c) Super hard pitch.

#### 5 REQUIREMENTS

##### 5.1 Composition

The material shall be:

- a) either the residue of the direct distillation of crude tar produced by the high temperature carbonization of coal in coke ovens or retorts, or
- b) obtained by fluxing back such pitch residues with high boiling coal tar distillates to give products of the desired softening point.

5.2 The material shall also comply with the requirements, given in Table 1 and Table 2 when tested according to test methods as indicated in respective columns.

6 SAMPLING AND CRITERIA FOR CONFORMITY

6.1 Lot

In any consignment, all the containers of coal tar pitch of the same grade and from the same batch of manufacture shall be grouped together to constitute a lot.

6.2 The number of containers to be selected at random from the lot shall depend upon the size of the lot and shall be in accordance with Table 3.

6.3 From each of the containers selected as in 6.2 an average sample representative of the material in the container shall be drawn in accordance with the methods prescribed in IS 1201, taking all the precautions mentioned therein. All these samples from the individual containers shall be stored separately.

6.4 Number of Tests

6.4.1 All the individual samples shall be tested for softening point.

6.4.2 For the remaining characteristics, a composite sample prepared by mixing together equal quantities of coal tar pitch from all the individual samples shall be tested.

6.5 Criteria for Conformity

6.5.1 The lot shall be considered as conforming to the requirements of this standard, if the conditions mentioned in 6.5.2 and 6.5.3 are satisfied.

6.5.2 From the test results of softening point the mean ( $\bar{X}$ ) and the ( $R$ ) shall be calculated. The following conditions shall be satisfied:

- a) ( $\bar{X} - 0.6 R$ ) shall be greater than or equal to the minimum specification limit, and
- b) ( $\bar{X} + 0.6 R$ ) shall be less than or equal to the maximum specification limit.

6.5.3 The composite sample when tested for the characteristics mentioned in 6.4.2 shall satisfy the corresponding requirements of the characteristics.

7 PACKING AND MARKING

7.1 Packing

The material shall be suitably packed in a container as agreed to between the supplier and the purchaser.

7.2 Marking

7.2.1 Each container shall be legibly and indelibly marked with the following:

- a) Manufacturer's name and trade-mark, if any;
- b) Grade;
- c) Batch number, code number, etc; and
- d) Date of manufacture.

Table 1 Requirements for Coal Tar Pitch  
(Clause 5.2)

Sl No.	Characteristic	Requirement				Method of Test, Ref to
		Soft Pitch	Soft Medium Pitch	Hard Medium Pitch	Hard Pitch	
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Softening point (Ring and Ball method), °C	45-55	56-75	76-85	86-95	IS 1205
ii)	Specific gravity at 25°C	1.20-1.30	1.22-1.32	1.24-1.32	1.26-1.32	IS 1202
iii)	Volatile matter content, percent by mass, <i>Max</i>	75	65	60	50	IS 1220
iv)	Matter insoluble in toluene (free carbon), percent by mass, <i>Max</i>	25	25	30	35	IS 1215
v)	Ash, percent by mass, <i>Max</i>	0.5	0.5	0.75	0.8	IS 1217

**Table 2 Requirements for Special Coal Tar Pitch**  
(Clause 5.2)

Sl No.	Characteristic	Requirements for Grades			Method of Test, Ref to
		Binder Pitch	Extra Hard Pitch	Super Hard Pitch	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Softening point (Ring and Ball method), °C	86-105	106-115	116-130	IS 1205
ii)	Specific gravity at 25°C	> 1.28	< 1.30	> 1.30	IS 1202
iii)	Coking value [by Conardson Carbon Method], percent by mass, <i>Min</i>	54	56	58	IS 13758 (Part 2)
iv)	Matter insoluble in toluene by G4, percent by mass, <i>Min</i>	30	—	—	IS 1215
v)	Matter insoluble in quinoline, percent by mass, <i>Max</i>	12	15	—	IS 13758 (Part 1)
vi)	Beta resin content, percent, <i>Min</i>	20	25	20	<sup>b)</sup>
vii)	Ash, percent by mass, <i>Max</i>	0.5	—	—	IS 1217
viii)	Iron (as Fe), percent, <i>Max</i>	0.05	—	—	IS 4284, IS 4542, and IS 13320
ix)	Silicon (as Si), percent, <i>Max</i>	0.02	—	—	Annex A
x)	Sodium (Na), ppm, <i>Max</i>	200	—	—	IS 9497
xi)	Sulphur (S), percent, <i>Max</i>	0.50	—	—	IS 1350 (Part 3)

<sup>b)</sup> The difference between the determined values of matter insoluble in toluene [see SI No. (iv)] and matter insoluble in quinoline [see SI No. (v)] shall be reported as beta resin content.

### 7.2.2 BIS Certification Marking

The containers may also be marked with the Standard Mark.

**7.2.2.1** The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

**Table 3 Number of Containers to be Selected**  
(Clause 6.2)

Sl No.	Lot Size	No. of Containers
(1)	(2)	(3)
i)	Up to 50	2
ii)	51 to 100	3
iii)	101 to 200	4
iv)	201 to 300	5
v)	301 to 500	7
vi)	501 and above	10

## ANNEX A

[Table 2, Sl No. (ix)]

DETERMINATION OF SILICON (Si) BY MOLYBDENUM BLUE METHOD  
(COLORIMETRIC METHOD)

## A-1 PRINCIPLE

This method is suitable for the determination of silicon in the range 0.2 to 10 ppm and is also free from interference from other ions usually present in these materials.

The coal-tar pitch will be ashed as per the standard procedure.

## A-2 REAGENTS

**A-2.1 Ammonium Molybdate (50 g/litre)** — Dissolve 50 g of ammonium molybdate ( $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ ) in water and dilute to one litre.

**A-2.2 Hydrochloric Acid (HCl) [1:1]** — Equal volume of HCl (sp. gr. 1.19) and water.

**A-2.3 Silicon Standard Solution (1 ml = 1 mg of Si)** — Dissolve 10.1 g of sodium silicate ( $\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$ ) in water and make to one litre in a volumetric flask and store in polyethylene bottle.

**A-2.4 Silicon Working Solution (1 ml = 0.01 mg of Si)** — Dilute 10 ml of the standard silicon solution to one litre in a volumetric flask.

**A-2.5 Stannous Chloride Solution** — Dissolve 2.5 g of stannous chloride ( $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ ) in 5 ml of concentrated HCl and dilute to 250 ml. Stable up to two weeks.

**A-2.6 Sodium Carbonate Solution (100 g/litre)** — Dissolve 100 g of  $\text{Na}_2\text{CO}_3$  in water and dilute to one litre.

**A-2.7 Sulphuric Acid (1:3)** — Carefully mix one volume of concentrated sulphuric acid with three volumes of water.

## A-3 APPARATUS

**A-3.1 Spectrophotometer**, visible.

## A-4 PROCEDURE

The ashed sample is fused with sodium carbonate and subsequently dissolved in 6 N hydrochloric acid. An aliquot of the solution is diluted to suitable concentration and the pH is adjusted to the values between 6-8. Silicomolibdydic acid is formed by the addition of ammonium molybdate solution, which is

then reduced with stannous chloride to form a deep blue suspension. The optical absorption of the solution is measured at 765 nm using a spectrophotometer.

## A-4.1 Calibration Curve

Take 1, 3, 5, 7 and 10 ml of silicon working solution, in 100-ml volumetric flasks and add 5 drops of  $\text{H}_2\text{SO}_4$  (1 : 3) to each flask and dilute to 10 ml. Add 2.5 ml of ammonium molybdate solution to each flask and allow to stand for 5 min. Then add 5 ml of  $\text{H}_2\text{SO}_4$  (1 : 3) mix well followed by the addition of 5 drops of  $\text{SnCl}_2$  solution and make up to 100 ml. Measure the absorbance at 765 nm after 5 min and plot the absorbance volumes against the micrograms of silicon. The blue colour is stable only up to 12 h.

## A-4.2 Method for Pitch

The pitch to be analyzed for silicon content is ashed using the standard procedure.

Fuse gently weighed quantity of the ash with sodium carbonate (around 6 g) in a platinum crucible over a Bunsen burner. When the fusion is complete remove the crucible from the burner, swirl to distribute the melt on the sides of the crucible and cool the crucible. The crucible is kept in a 250-ml beaker and 25 ml of water added. Cover the beaker with a watch glass and add cautiously HCl (1:1) to decompose the melt. Boil the solution for several minutes and cool. Transfer to a 100-ml volumetric flask and dilute to volume. Transfer a suitable aliquot of this solution to 100-ml volumetric flask, adjust the pH to 6 - 8 with  $\text{Na}_2\text{CO}_3$  solution and determine the silicon colorimetrically as explained in the procedure for the calibration graph in A-4.1 above.

Find the microgram of silicon in the sample solution from the calibration curve.

## A-5 CALCULATION

$$\text{Silicon, ppm} = (A \times B)/W$$

where

$A$  = microgram of silicon per 100 ml of the solution found in the aliquot used,

$B$  = aliquot factor (original volume divided by aliquot taken for analysis), and

$W$  = original weight of the sample.

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This Indian Standard has been developed from Doc: No. PCD 6 (1948).

### Amendments Issued Since Publication

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