

# इंटरनेट

# मानक

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IS 13758-2 (1993): Coal tar pitch: Methods of test Part 2; Determination of coking value [PCD 6: Bitumen Tar and their Products]



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“Knowledge is such a treasure which cannot be stolen”



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भारतीय मानक

कोलतार पिच — परीक्षण पद्धतियाँ

भाग 2 कोकिंग मान ज्ञात करना

*Indian Standard*

COAL TAR PITCH — METHODS OF TEST

PART 2 DETERMINATION OF COKING VALUE

UDC 662·749·38 : 543·824·42

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NEW DELHI 110002

## FOREWORD

This Indian Standard 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.

The quality of coal tar pitch depends on various factors such as parent tar, process of distillation adopted and temperature of distillation. The quality of the pitch may also be modified by blending, thermal treatment, air blowing at high temperature, etc. Coal tar pitch is assessed by many properties, namely, content of different types of resins, coking value, softening point, specific gravity and lower boiling fractions present in the pitch.

Bitumen, Tar and Their Products Sectional Committee had prepared Methods of testing for tar and bituminous materials, namely, IS 1201 : 1978 to IS 1220 : 1978 'Methods of testing tar and bituminous materials (*first revision*) ( IS 1201 to 1220 in one volume )', IS 9381 : 1979 'Methods of testing tar and bituminous materials : Determination of FRAASS breaking point of bitumen' and IS 9382 : 1979 'Methods of testing tar and bituminous materials : Determination of effect of heat and air by thin film oven tests'. However, the Specification for coal tar pitch requires test methods for additional characteristics which are intended to be covered in this series of Standards. This standard IS 13758 ( Part 2 ) : 1993 is the second in the series. Eventually it may be possible to combine the test methods for tar, bituminous material and coal tar pitch in one series.

In this standard two methods namely Method A and Method B have been given. However, committee envisaged to eventually retain only Method B ( Rapid Method ) because it is expected that in future Method A may not be in vogue.

In the preparation of this standard due consideration has been given to the views of producers, consumers and technologists and manufacturing and trade practices followed in different countries. Due weightage has also been given to the need for international co-ordination among standards prevailing in different countries of the world. These considerations have led the Sectional Committee to derive assistance from the publications of Institute of Petroleum, United Kingdom, ASTM and Standardization of Tar Products Committee ( STPTC ), U. K.

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

# Indian Standard

## COAL TAR PITCH — METHODS OF TEST

### PART 2 DETERMINATION OF COKING VALUE

#### 1 SCOPE

**1.1** This standard ( Part 2 ) covers the method of test for the determination of coking value of coal tar pitch. Two methods namely Method A and Method B have been given.

**1.2** This standard does not cover the coal tar pitch having ash content more than 0.8 percent by mass.

#### 2 NORMATIVE REFERENCES

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

IS No.	Title
216 : 1961	Coal tar pitch ( revised )
460 : 1978	Test sieves

#### 3 APPARATUS

**3.1** Glazed porcelain or silica crucibles ( for Method A ) 29 to 31 ml capacity, 46 to 49 mm in rim diameter.

#### 3.2 Skidmore Crucible

Iron crucible flanged and ringed, 65 to 82 ml capacity, 53 to 57 mm inside and 60 to 67 mm outside diameter of flange, 37 to 39 mm in height supplied with a cover without delivery tubes and having the vertical opening closed. The horizontal opening of about 6.5 mm shall be kept clean. The outside diameter of the flat bottom shall be 30 to 32 mm.

#### 3.3 Metal Crucible

Spun-sheet-iron or nickel crucible, with cover, 78 to 82 mm in diameter and 50 to 60 mm in height. At the bottom of this crucible and level before each test shall be a layer of 25 ml of sand or calcined petroleum coke enough to bring the skidmore crucible with cover on nearly to the top of the sheet iron crucible. The sand or calcined coke should be ground to pass through 600 micron ( IS 460 : 1978 ) and to be retained on 75 micron IS sieve.

#### 3.4 Muffle Furnace

Fitted with pyrometer controller, capable of

attaining the desired temperature within 3 minutes after inserting the sample. The furnace shall have sufficient space to accommodate the whole assembly.

#### 3.5 Desiccator

#### 4 METHOD A

##### 4.1 Preparation of Samples

**4.1.1** In the case of soft pitch small portions may be taken out by means of spatula or any other suitable tool with or without prior melting.

**4.1.2** In the case of hard pitch sample shall be prepared by grinding in a mortar such that it passes through 600 mesh sieve. The crushed samples shall not be kept for future analysis since changes in composition sometimes occur in pulverized pitch.

##### 4.2 Procedure

**4.2.1** Heat a clean porcelain or silica crucible in a muffle furnace at 900°C for one hour. Cool in a desiccator and weigh to nearest 1 mg (  $M_1$  ). Transfer about 3 g of dry pitch sample into the tared crucible and weigh to the nearest 1 mg (  $M_2$  ). Place this crucible in the centre of the skidmore crucible. Level the sand or calcined coke in the metal crucible and place the skidmore crucible in the centre. Apply covers to both the skidmore and metal crucibles, the latter fitting loosely to allow free exit of the vapours formed. Insert the assembly in the muffle furnace maintained at a temperature of  $900 \pm 10^\circ\text{C}$  as quickly as possible. The assembly shall be placed in a manner that there is a minimum of space of 6 mm between it and the floor, walls and roof of the furnace. The distance between the crucible and the front door of the furnace shall be 50 mm, *Min.*

Keep the crucible and the contents inside the muffle furnace for 30 minutes. Remove the crucible with contents from the furnace after 30 minutes and allow to cool for 15 minutes. Take out the porcelain crucible with the help of heated tongs and place in a desiccator, cool and weigh (  $M_3$  ).

##### 4.2.2 Calculation

Calculate the coking value of coal tar pitch as follows:

$$\text{Coking value, percent by mass} = \frac{M_3 - M_1}{M_2 - M_1} \times 100$$

where

$M_1$  = mass in g of tared crucible;

$M_2$  = mass in g of tared crucible with sample; and

$M_3$  = mass in g of tared crucible with the residue left after heating in muffle furnace at  $900 \pm 10^\circ\text{C}$ .

#### 4.2.3 Report

Report the coking value nearest to 0.1 percent.

#### 4.2.4 Precision

The duplicate test results shall not differ by more than the following:

Repeatability	Reproducibility
5.0 percent	10.0 percent

### 5 METHOD B ( RAPID METHOD )

#### 5.1 General

This method covers the determination of the amount of carbon residue left after evaporation and pyrolysis of coal tar pitch under specified condition.

This method is intended to provide some indication of relative coke forming properties.

NOTE — The term 'carbon residue' is used throughout this test method to designate the carbonaceous residue formed after evaporation and pyrolysis of coal tar pitch. This residue is not composed entirely of carbon, but is a coke which can further be changed by pyrolysis.

#### 5.2 Apparatus

##### 5.2.1 Silica Crucible

Cylindrical, translucent with a lid of capsule type and having the following dimensions:

Crucible	Lid
Height, mm — 38.0	Internal diameter, mm — 27.0
Diameter, mm — 25.0 ( external )	Diameter of the well, mm — 21.0
22.0 ( internal )	Depth of the well, mm — 4.0
Combined weight of the crucible and the lid, g	12-14
Clearance between crucible and the lid at the top edge, mm, Max	0.5

#### 5.2.2 Muffle Furnace

Electrically heated, in addition to having heat gaining capacity and having an arrangement to exclude air-circulation inside the furnace during the experiment. It shall be provided with pyrometer controller, capable of attaining the temperature within 3 minutes after the drop in temperature that occurs while inserting the sample.

#### 5.3 Preparation of the Sample

The sample taken for the test shall be free from water and representation of the whole lot.

#### 5.4 Procedure

5.4.1 The quantity of the sample to be taken for the determination of carbon residue shall be as per the following:

Coking Value, %	Quantity of Sample Required, g
10	1.5-2.0
10-30	1.0-1.5
30	0.5-1.0

5.4.2 Accurately weighed sample ( 0.5-2.0 g ) is taken in the silica crucible. To this accurately weighed inert material ( 2-3 g ) of sand of specific grade ( see Note ) is mixed.

NOTE — The essential characteristics of the sand suitable for use in the determination of coking value shall be as follows:

- It shall be completely inert and be of pure silica, free from impurities like clay, chalk and iron carbonates.
- Shall pass through a 30-mesh IS-sieve and be retained on a 20-mesh IS-sieve.
- Shall not contain 5 percent oversize and or 10 percent undersize.
- On heating for 3 h at  $920^\circ\text{C}$ , the undersize produced by disintegration shall not exceed 2.5 percent.
- Solubility in hot dilute hydrochloric acid shall not exceed 0.5 percent.

5.4.3 The crucible containing the sample and sand is introduced in the muffle furnace maintained at a temperature of  $570 \pm 5^\circ\text{C}$  and is kept for 7 minutes. The crucible is then taken-out and allowed to cool to  $50-60^\circ\text{C}$  and finally kept in a desiccator, cooled and weighed.

#### 5.4.4 Calculation

Calculate the carbon residue of coal tar pitch as follows:

$$\text{Carbon residue, percent by mass} = \frac{M_3 - M_1}{M_2 - M_1} \times 100$$

where

$M_1$  = mass in g of tared crucible and sand;

$M_2$  = mass in g of tared crucible, sand and sample; and

$M_3$  = mass in g of tared crucible, sand and the residue.

#### 5.4.5 Report

Report the carbon residue value nearest to 0.1 percent.

#### 5.4.6 Precision

The duplicate result shall not differ by more than 2.4 percent of the average of two results.



### **Standard Mark**

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