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मानक

IS 13758-1 (1993): Coal Tar Pitch; Methods of test, Part 1: Determination of matter insoluble in quinoline [PCD 6: Bitumen Tar and their Products]



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Indian Standard

COAL TAR PITCH — METHODS OF TEST

PART 1 DETERMINATION OF MATTER INSOLUBLE IN QUINOLINE

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Price Group 1

Bitumen Tar and Their Products Sectional Committee, PCD 6

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 distillationadopted 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, IS 9381: 1979 and IS 9282: 1979. However, the specification for coal-tar pitch requires test methods for additional characteristics which are intended to be covered in this series. This standard IS 13758 (Part 1): 1993 is the first in the series. Eventually it may be possible to combine the test methods for tar, bituminous material and coal pitch in one series.

In the preparation of this standard due consideration has been given to the views of producers, consumers and technologists and the 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 Tests Committee (STPTC, UK).

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 1 DETERMINATION OF MATTER INSOLUBLE IN QUINOLINE

1 SCOPE

1.1 This standard (Part 1) covers the method of test for the determination of matter insoluble in quinoline for coal tar pitch.

2 REFERENCES

2.1 The Indian Standards listed below are necessary adjuncts to this standard:

IS No. Title

216:1961 Coal tar pitch (first revision)

334:1982 Glossary of terms relating to bitumen and tar (second revision)

460 Wire cloth test sieves (third (Part 1): 1985 revision)

1839 : 1961 Toluene, reagent grade

3 TERMINOLOGY

3.1 For the purpose of this standard, the definitions given in IS 334 : 1982 shall apply.

4 APPARATUS

4.1 Beaker — 100-ml.

4.2 Gooch Crucible – G 4.

4.3 Water Bath — Maintained at constant temperature of $75 \pm 5^{\circ}$ C.

4.4 Filtering Flask - 500-ml.

4.5 Sieves — 300 micron and 600 micron [see 1S 460 (Part 1) : 1985].

5 REAGENTS

5.1 Toluene — See IS 1839 : 1961.

5.2 Quinoline — refined of light colour and conforming to the following requirements:

- a) Specific gravity $(27/27^{\circ}C)$: 1.085 to 1.091
- b) Water content, percent by : 0.5 mass, Max
- c) Boiling range : 5 percent to 95 percent distilling at 238°C

6 PREPARATION OF SAMPLE

6.1 Heat the sample of coal-tar pitch to a temperature not exceeding 130°C until the free water has been removed. Heating shall be done, while stirring constantly, when possible, during melting, as expeditiously as possible.

6.2 In the case of hard pitches having softening point higher than 70° C, a representative sample shall be prepared by crushing, mixing and quartering of the total sample such that the material passes through 250 micron IS sieve.

7 PROCEDURE

7.1 Place 1 g of sample in case of HT or 3 g of sample in the case LT pitch or lignite pitch in the tare beaker and weigh again nearest to 0.001 g, M_2 . Add 25 ml of quinoline (5.2) to the beaker and place the beaker in a water bath maintained at 75 ± 5°C. Allow it to stand for 15 minutes while stirring the contents of the beaker to ensure complete digestion.

7.2 Insert the filter tube in the stopper of the filtering flask, set the Gooch crucible in the filter tube, and connect the flask to the suction pump. Fill the crucible with some of the suspension of asbestos in the water, allow it to settle partly in the crucible and apply a light suction to draw off the water, leaving a firm mat of asbestos in the crucible. Add more suspended asbestos and repeat the process until a mat weighing 0.5 ± 0.1 g is built up after drying. Wash the asbestos mat thoroughly with water and dry in the oven at a temperature of 150° C. Cool the crucible in the desiccator, weigh (M_3) and replace it in the dry filter tube supported in the clean, dry filtering flask.

7.3 Place the previously prepared and weighed Gooch crucible (7.2) in the crucible holder and set up in the filtering flask for filtering under suction carefully. Add sufficient quinoline to the crucible carefully to moisten the asbestos mat thoroughly. When ready to filter the test solution apply suction to the Gooch crucible and form a mat of evenly distributed asbestos wetted with quinoline. While the asbestos mat in the crucible is still wet, stir the contents of the beaker and filter through the Gooch crucible. Stir the mixture in the beaker immediately before pouring successive portions into the crucible. Maintain suction on the flask and finally allow the crucible to drain but do not allow the quinoline solution to drain to such an extent that solid material on the filter becomes dry. Rinse the beaker with quinoline, using 25 ml of quinoline at 70° to 80°C, and transferring this portion of quinoline and all insoluble matter to the filter, allowing the crucible to drain.

7.4 Wash the beaker with a total of 130 ml of toluene using four portions of 30 to 35 ml each to ensure that all insoluble matter in the beaker is transferred to the filter and is washed. Allow each portion of toluene to filter slowly and drain from the crucible before introducing a subsequent portion but do not allow solid matter on the filter to dry or form a broken cake until washing is completed. Drain all benzene from the filter under suction.

7.5 Remove the Gooch crucible from the holder, clean the exterior surfaces if necessary and allow residual toluene to evaporate from the crucible and its contents at room temperature. Place the Gocch crucible in the drying oven at 105° C to 110° C for 30 to 60 minutes and bring to constant mass. Transfer the crucible to the desiccator and cool it for 20 to 30 minutes and weigh (M_4).

8 CALCULATION

8.1 Calculate and express the result as percentage of mass of the material taken for the test as follows:

> Matter insoluble in quinoline, percent by mass $= \frac{M_4 - M_3}{M_2 - M_1} \times 100$

where

 $M_1 = \text{mass in g of the tare beaker;}$

 $M_2 = \text{mass in g of tare beaker with sample;}$

 $M_3 = \text{mass in g of Gooch crucible}$ with asbestos mat; and

 $M_4 = \text{mass in g of Gooch crucible}$ with asbestos mat and residue.

8.2 Report — Report the result nearest to 0.1 percent.

9 PRECISION

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

Repeatability	Reproducibility
0.5 percent	0 ¹ percent

10 PRECAUTION

10.1 Dry asbestos mat gains mass when exposed to air and therefore rapid weighing is necessary for accurate determination of dry mass.

10.2 Insoluble matter on the filter, after washing with toluene, should have no odour of quinoline, if it does, the odour is evidence of insufficient washing. In this case the determination shall be discarded and the test repeated.

10.3 After use in not more than six determination, remove any residual matter from pores in the filtering area of the crucible by passing hot dilute hydrochloric acid through the crucible, washing with distilled water, drying igniting the crucible.

10.4 Fumes of the solvents should be removed by means of proper hoods from all working areas. The working area should be kept free of sparks and flames. Quinoline fumes should not be inhaled, and prolonged contact with the skin should be avoided.

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Review of Indian Standards

PCD 6 (833)

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