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IS 19008 : 2023

भारतीय मानक

Indian Standard

बिटुमिनस फ़र्श मिश्रण में बिटुमेन सामग्री का निर्धारण

Determination of Bitumen Content in Bituminous Paving Mixtures

ICS 91.100.50; 93.080.20

BIS 2023

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

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Bitumen, Tar and Related Products Sectional Committee, PCD 06

FOREWORD

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

Quantitative determination of bitumen in bituminous paving mixtures and pavement samples is useful for specification compliance; quality control; service evaluation; investigation; and research. Aggregate obtained by this method may be used for sieve analysis.

The composition of the committee, responsible for formulation of this standard is listed in Annex A.

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 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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Indian Standard

DETRMINATION OF BITUMEN CONTENT IN BITUMINOUS PAVING MIXTURES

1 SCOPE

1.1 This standard prescribes two methods of test for determination of bitumen content in bituminous paving mixtures, namely, Solvent Extraction Method (Method A) and Ignition Method (Method B).

1.2 Method A covers the extraction of bituminous component from paving mixture using trichloroethylene solvent. Then the bitumen content is calculated by difference from the mass of the extracted aggregate, moisture content, and mineral matter in the extract and expressed as masspercent of moisture-free mixture.

1.3 In Method B, bituminous paving mixture is ignited in a furnace at 540 °C to burn off all the bitumen. The bitumen content is then calculated by difference between the mass of the total specimen and the mass of the residual aggregate and moisture content and expressed as mass percent of moisture-free mixtures.

2 REFERENCES

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

IS No.	*Title*
IS 245 : 2020	Specification for trichloroethylene, technical (*fourth revision*)
IS 334 : 2002	Glossary of terms relating to bitumen and tar (*third revision*)

3 TERMINOLOGY

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

4 METHOD A — SOLVENT EXTRACTION METHOD

4.1 Apparatus

4.1.1 *Oven*, capable of maintaining the temperature at (110 ± 5) °C.

4.1.2 *Flat Pan* — 305 mm (12 inch) long, 203 mm (8 inch) wide, and 25 mm (1 inch) deep.

4.1.3 *Balance*

4.1.4 *Hot Plate* — Electric, 700-W continuous or low, medium, and high settings.

4.1.5 *Small-Mouth Graduate* — 1 000 ml or 2 000 ml capacity. Optional small-mouth graduate, 100 ml capacity.

4.1.6 *Ignition Dish* — 125 ml capacity.

4.1.7 *Desiccator*

4.1.8 *Extraction Apparatus*

It consists of a bowl approximating (*see* Fig. 1) and an apparatus in which the bowl may be revolved at controlled variable speeds up to 3 600 rpm. The speed may be controlled manually or with a preset speed control. The apparatus should be provided with a container for catching the solvent thrown from the bowl and a drain for removing the solvent. The apparatus preferably shall be provided with explosion-proof features and installed in a hood or an effective surface exhaust system to provide ventilation.

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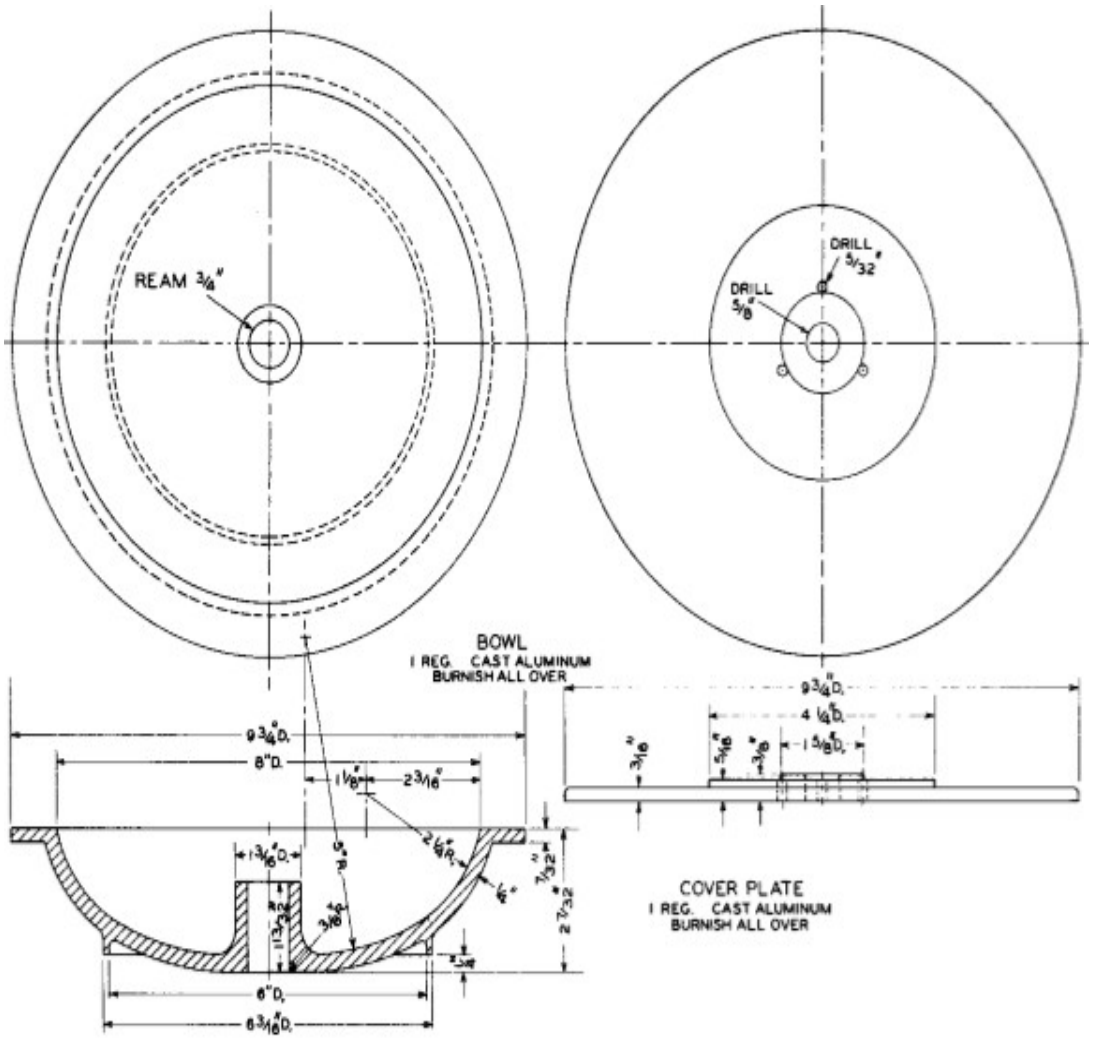

FIG. 1 Extraction Unit Bowl

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4.1.9 *Filter Rings* — Felt or paper, to fit the rim of the bowl. Low-ash paper filter rings may be usedin place of the felt filter ring. Such filter rings shall consist of low ash filter paper stock with (0.05 ± 0.005) inch or (1.27 ± 0.127) mm thickness. The nominal base weight of the paper shall be (330 ± 30) lb or (149.685 ± 13.608) kg for a ream. The ash content of the paper should not exceed 0.2 percent (approximately 0.034 g per ring).

NOTE — There is no international standard designation for large- size sheets of filter paper from which theround filters are cut out. These round filter papers are sold by the test equipment supplier.

4.2 Reagent

4.2.1 *Trichloroethylene*, technical grade conforming to IS 245.

NOTE — Benzene shall not be used because of its high toxicity.

4.3 Preparation of Test Specimen

4.3.1 If the mixture is not sufficiently soft to separate with a spatula or trowel, place it in a large, flat pan and warm to (110 ± 5) °C only until it can be handled or mixed. Split or quarter the material until the mass of material required for test is obtained.

4.3.2 The size of the test sample shall be governed by the nominal maximum aggregate size of the mixture and shall conform to the mass requirement shown in Table 1.

Table 1 Size of Sample Required for Method A
(*Clause* 4.3.2)

Sl No.	Nominal Maximum Aggregate Size, mm	Sieve Size	Minimum Mass of Sample, kg
(1)	(2)	(3)	(4)
i)	4.75	No. 4	0.5
ii)	9.5	3/8 in	1.0
iii)	12.5	½ in	1.5
iv)	19.0	¾ in	2.0
v)	25.0	1 in	3.0
vi)	37.5	1 ½ in	4.0

NOTES

1 When the mass of the test specimen exceeds the capacity of the extraction equipment, the test specimen may be divided in suitable increments, tested, and results appropriately combined for calculation of bitumen content.

2 In addition, a test specimen is required for the determination of moisture in the mixtures. Take this test specimen from the remaining sample of the mixture immediately after obtaining the extraction test specimen.

3 If recovery of bitumen from the solution obtained from the extraction test is not required, the entire test specimen may be dried to constant mass in an oven at a temperature of (110 ± 5) °C prior to extraction instead of determining the moisture content.

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4.4 Procedure

4.4.1 Determine the moisture content of the sample from the test specimen taken separately. A weighed amount of sample is dried in an oven at (110 ± 5) °C to constant mass and its moisture content is determined.

4.4.2 Place a 650 g to 2 500 g test portion ($W1$) into a bowl. Cover it with trichloroethylene and allow sufficient time (not more than 1 h) for the solvent to disintegrate the test portion. Place the bowl containing the test portion and the solvent in the extraction apparatus. Dry and determine the mass of the filter ring and fit it around the edge of the bowl. Clamp the cover on the bowl tightly and place a beaker under the drain to collect the extract.

4.4.3 Start the centrifuge revolving slowly and gradually increase the speed to a maximum of 3 600 rpm or until solvent ceases to flow from the drain. Allow the machine to stop, add 200 ml of trichloroethylene and repeat the procedure. Use sufficient 200 ml solvent additions so that the extract is not darker than a light straw color. Collect the extract and the washings in a suitable graduate.

4.4.4 Remove the filter ring from the bowl and dry in air. If felt filter rings are used, brush off mineral matter adhering to the surface of the ring and add to the extracted aggregate. Dry the ring to constant mass in an oven at (110 ± 5) °C. Carefully remove all the contents of the bowl into a metal pan and dry to constant mass in an oven or on a hot plate at (110 ± 5) °C.

4.4.5 The mass of the extracted aggregate, $W3$, is equal to the mass of the aggregate in the pan plus the increase in mass of the filter rings.

NOTE — Since dry aggregate absorbs moisture when exposed to air containing moisture, determine the mass of the extracted aggregate immediately after cooling to a suitable temperature.

4.4.6 Record the volume of the total extract liquid in the graduate.

4.4.7 Determine the mass of an ignition dish. Agitate the extract thoroughly and immediately measure approximately 100 mL into the ignition dish. Evaporate to dryness on a hot plate. Ash residue at a dull red heat (500 °C to 600 °C), cool, and add 5 ml of saturated ammonium carbonate solution per gram of ash. Digest at room temperature for 1h. Dry in an oven at 100 °C to constant mass, cool in a desiccator, and determine the mass (G).

4.4.8 Calculate the mass of mineral matter in the total volume of extract liquid, W4, as follows

$$\boldsymbol{W4 = G \times [V1/(V1 - V2)]}$$

where

W_4 = mass, in g, of mineral matter in the total volume of extract;

G = ash, in g, in aliquot;

V_1 = total volume, in ml, of liquid extract; and

V_2 = volume, in ml, after removing aliquot.

4.5 Calculation

$$\text{Bitumen Content, percent} = \left(\frac{(W1-W2)-(W3-W4)}{(W1-W2)}\right) \times 100$$

where

$W1$ = mass, in g, of test portion taken;

$W2$ = mass, in g, of water in the test portion;

$W3$ = mass, in g, of the extracted aggregate; and

$W4$ = mass, in g, of the mineral matter in the total volume of extract.

4.6 Report

Report the bitumen content up to two decimal places (nearest 0.01 percent). Also report mass of bituminous mixture taken for testing (nearest 0.1 g).

5 METHOD B — IGNITION METHOD

5.1 Apparatus

5.1.1 *Ignition Furnace*

A forced-air ignition furnace that heats the specimens by either the convection or direct irradiation method. The convection-type furnace must have a minimum temperature capability of 580 °C. The furnace shall have an internal weighing system capable of weighing sample size of at least 2 500 g. A data collection system shall also be included so that the sample mass loss can be determined to an accuracy of 0.1 g and displayed during the test. The test is deemed complete when the difference between consecutive mass losses does not exceed 0.01 percent of the sample mass for three consecutive 1 minute intervals. The equipment shall provide a print out of the test results.

The furnace door shall be equipped so that it cannot be opened during the ignition test. A method for reducing furnace emissions shall be provided and the furnace shall be vented so that no emissions escape into the laboratory. The furnace shall have a fan to pull air through the furnace to expedite the test and to eliminate the escape of smoke into the laboratory.

NOTE — The temperature of furnace, sample, sample trays(s), and catch pan is extremely high. Therefore, caution should be always exercised while handling these items otherwise serious injuries may result. These very hot items should not be placed for cooling near other things which may ignite. The manufacturer's instruction manual should be followed to take all safety precautions.

5.1.2 *Sample Basket Assembly*

It consists of sample basket(s), catch pan, and basket guards. Sample basket(s) will be of appropriate size allowing samples to be thinly spread and allowing air to

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flow through and around the sample particles. Sets of two or more baskets shall be nested. A catch pan of sufficient size to hold the sample basket(s) so that aggregate particles and melting binder falling through the screen mesh are caught. Basket guards will completely enclose the basket and be made of screen mesh, perforated stainless steel plate, or other suitable material.

5.1.3 *Thermometer,* or other temperature measuring device, with a temperature range of 10 °C to 260 °C and readable to 0.1 °C .

5.1.4 *Oven*, capable of maintaining (110 ± 5) °C.

5.1.5 *Balance***,** with accuracy of 0.1 g and capacity sufficient for measuring the mass of the specimen, sample trays, and catch pan.

5.2 Calibration

5.2.1 Since different types of aggregates lose mass to varying degrees on ignition, the results of this test may be affected. Also, presence of modifiers and additives can affect the test results. Therefore, a calibration factor shall be established by three calibration mixture samples prepared with known bitumen content and aggregate gradation to be used on the project.

5.2.2 The calibration sample shall be approximately of the same mass and gradation as of test sample. In case of convection-type furnace, set the furnace temperature to (540 ± 5) °C for calibration mixtures. In case of direct-irradiation type furnace, set the burn profile to the DEFAULT mode. Prepare the three calibration mix samples at the design bitumen content (*P*). Include any modifiersor additives to be used on the project. Determine the mass of the sample tray(s) and catch pan to the nearest 0.1 g. Distribute the loose mix evenly in the sample tray(s). Determine the mass of thesample, sample tray(s), and catch pan to nearest 0.1 g. Calculate and record the initial mass of the sample (W_I). Heat the calibration sample in the furnace at (540 ± 5) °C until the change in the mass of sample during three consecutive intervals of 1 min does not exceed 0.01 percent of the sample mass (W_I). Measure and record the mass loss (W_L) of the sample after ignition to the nearest 0.1 g.This mass shall be shown in the print out and display also Calculate the calibration factor (*CF*) as follows:

$$CF = \left(\frac{W_I - W_L}{W_I} \times 100\right) - P$$

where

W_I = total mass, in g, of the mixture calibration sample prior to ignition;

W_L = total mass, in g, of the mixture calibration sample after ignition; and

P = percentage of actual bitumen content in the mix by mass of the total mix expressed as a percentage.

5.2.3 Repeat the preceding steps for the remaining two additional calibration samples. Then calculate the average calibration factor (CF_{avg}) for this specific mix by taking average of three *CF* values.

5.2.4 The temperature for testing bituminous mixtures later for this project shall be the same as used in this calibration process.

5.3 Procedure

5.3.1 Obtain samples of bituminous mixture.

5.3.2 If the mixture is not sufficiently soft to separate with a spatula or trowel, place it in a large flat pan in an oven at (110 ± 5) °C until soft enough.

5.3.3 Test sample size shall conform to the mass requirement shown in Table 2.

Table 2 Size of Sample Required for Method B
(*Clause* 5.3.3)

Sl No.	Nominal Maximum Aggregate Size, mm	Minimum Mass of Sample, kg	Maximum Mass of Sample, kg
(1)	(2)	(3)	(4)
i)	37.5	4.0	4.5
ii)	25.0	3.0	3.5
iii)	19.0	2.0	2.5
iv)	12.5	1.5	2.0
v)	9.5	1.2	1.7
vi)	4.75	1.2	1.7

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5.3.4 Obtain the sample of the bituminous mixture for testing, its mass should be about the same as that used in the calibration process.

5.3.5 Dry the sample in an oven at (110 ± 5) °C to constant mass so that its moisture content can be determined.

5.3.6 Set the furnace temperature or burn profile in accordance with the instructions of the furnace manufacturer. Mix sample to be placed in the furnace can be at any temperature because the furnace will heat it up quickly.

5.3.7 Determine the mass of the sample tray(s) and catch pan to the nearest 0.1 g. Note that all these and subsequent measurements will be made by the built-in weighing system.

5.3.8 Place the loose sample mix evenly in the sample tray(s).

5.3.9 Determine the mass of the mix sample, sample tray(s) and catch pan to the nearest 0.1 g. Calculate and record the initial mass of the sample (*W*B).

5.3.10 Heat the sample in the ignition oven at the specified temperature until the difference between consecutive measured mass loss does not exceed 0.01 percent of the sample mass (W_B) for three consecutive 1-minute time intervals. This point shall be determined by the automated furnace data collection system.

5.3.11 The furnace's data collection system shall measure and record the mass of the sample after ignition (W_A) to the nearest 0.1 g. This will be determined by the furnace data collection system.

5.4 Calculation

The corrected bitumen content shall be calculated automatically by the furnace's data collectionsystem as follows:

Bitumen Content, percent

$$= \left(\frac{W_B - W_A}{W_B} \times 100 \right) - CF_{avg}$$

where

W_A = total mass, in g, of the aggregate remaining after ignition;

W_B = total mass, in g, of the bituminous mixture prior to ignition; and

CF_{avg} = calibration factor obtained (**5.2.3**) and entered into the furnace's data collection system for this specific mix

5.5 Report

Report the bitumen content up to two decimal places (nearest 0.01 percent). Also report calibrationfactor and mass of bituminous mixture before and after ignition (nearest 0.1 g).

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ANNEX A
(*Foreword*)

COMMITTEE COMPOSITION
Bitumen, Tar and Related Products Sectional Committee, PCD 06

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices: *Telephones*

Central : 601/A, Konnectus Tower -1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002 { 2323 7617

Eastern : 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091 { 2367 0012 2320 9474

Northern : Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019 { 265 9930

Southern : C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113 { 2254 1442 2254 1216

Western : Plot No. E-9, Road No.-8, MIDC, Andheri (East), Mumbai 400093 { 2821 8093

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