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भारतीय मानक मसौदा

पेट्रोलियम कोक — विशिष्टि

(IS 8502 का तीसरा पुनरीक्षण)

Draft Indian Standard

PETROLEUM COKE — SPECIFICATION

(*Third Revision of* IS 8502)

(ICS No. 75.160.20)

Petroleum and their related products of synthesis or Biological origin Sectional Committee, PCD 03	Last date for receipt of comment is **15 December 2024**

FOREWORD

(*Formal clauses will be added later*)

Petroleum coke is manufactured by delayed coking [thermal cracking] of residues obtained from crude oil distillation units, primarily from vacuum distillation and sometimes from atmospheric distillation units, of petroleum refineries. The petroleum coke thus obtained from different crude oil residues is categorised as Raw Petroleum Coke (RPC). The petroleum coke obtained by calcining the RPC at a temperature of 1200 °C to 1400 °C is known as Calcined Petroleum Coke (CPC).

The RPC is either sold as such in the market or after calcination. RPC has limited application in industry, while the CPC is extensively used for the manufacture of carbon and graphite products and carbon anodes for aluminium smelters.

India is one of the major producers and consumer of coal and coke on an extensive scale. National standards have been published, covering methods used for sampling and testing of coal and coke. But petroleum coke is comparatively a newcomer in the field of industrial applications and various industries, utilizing petroleum coke, have so far been guided by either ASTM or individual specifications and methods of test to suit their requirements. As a consequence of such practice, the major producers of this commodity are put to inconvenience. To overcome this difficulty, after collecting as much data as possible from various consumers and after giving due weightage to the various requirements, specifications and test methods, this specification for petroleum coke was first published in 1977.

This standard was subsequently revised in 1994. Requirements for volatile matter, sulphur and trace metals, that is, silicon, iron, vanadium and nickel for RPC were modified.

The second revision of this standard was published in 2018 after a thorough review of the standard in consultation with the major producers and consumers of these products. In this revision, requirements for volatile matter, sulphur and fixed carbon were modified and specification requirements for premium grade of RPC were incorporated. Test method for determination of moisture content by moisture balance was incorporated.

This third revision has been brought out to keep pace with the latest technological developments and international practices. In this revision, Amendment 1 to IS 8502 : 2018 has been incorporated and test methods for moisture content and metal content have been updated.

There is a separate standard IS 17049 for specification for petroleum coke for anode making in aluminium industry, keeping in view the sulphur content required in CPC for the equipment used by them.

Various documents relating to methods of test for coal and coke which are relevant and adaptable have been taken into consideration in this standard to avoid duplication of effort. The following Indian Standards for sampling and testing of coal and coke fall under the above category:

IS 436 (Part l/Sec 1) : 2024	Methods for Sampling of Coal and Coke Part 1 Sampling of Coal Section 1 Manual Sampling (*second revision*)
IS 16143 (Part 5) : 2021 / ISO 13909-5 : 2016	Hard coal and coke - Mechanical sampling Part 5 Coke - Sampling from moving streams (*first revision*)
IS 16143 (Part 6) : 2021 / ISO 13909-6 : 2016	Hard coal and coke - Mechanical sampling Part 6 Coke - Preparation of test samples (*first revision*)
IS 1350 (Part 1) :1984	Methods for testing of coal and coke: Part 1 Proximate analysis (*second revision*)
IS 1350 (Part 3) : 2022 / ISO 334 : 2020	Methods of test for coal and coke : Part 3 Determination of total sulphur - Eschka method (*second revision*)
IS 1354 : 2024	Coke - Methods of test - Special tests (*third revision*)
IS 1355 : 1984	Methods of determination of the chemical composition of ash of coal and coke (*first revision*)
IS 7929 : 1975	Methods for determination of electrical resistivity of chemical coke

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.

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for raw petroleum coke and calcined petroleum coke, used for the manufacture of electrodes, carbon and graphite products, carbon anodes and for other uses like manufacture of cement.

2 REFERENCES

The standards listed below contain provisions 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 the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
IS 7929 : 1975	Methods for determination of electrical resistivity of chemical coke
IS 1447 (Part 4): 1989	Petroleum and its products methods of sampling: Part 4 sampling of petroleum coke for laboratory analysis (*first revision*)
IS 1448	Methods of test for petroleum and its products
(Part 33) : 2021	Sulphur by high pressure decomposition device method
(Part 79) : 2024	Determination of vanadium and nickel in furnace oil, crude oil and their residues - Spectrophotometric method (*second revision*)
(Part 126) : 2023	Determination of ash content in raw and calcined petroleum coke (*first revision*)
(Part 127) : 2024	Determination of iron in petroleum coke (*first revision*)
(Part 128) : 2018	Determination of nickel in calcined petroleum coke (*first revision*)
(Part 130) : 2019	Determination of vibrated bulk density of calcined petroleum coke (*first revision*)
(Part 131) : 2024	Determination of silicon in petroleum coke (*first revision*)
(Part 133) : 2018	Determination of real density of calcined petroleum coke (*first revision*)
(Part 134) : 2018	Determination of volatile matter in raw and calcined petroleum coke (*first revision*)
(Part 139) : 1992	Determination of real density of calcined petroleum coke using butanol or toluene (*first revision*)
ASTM D7582-24	Standard Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis
ASTMD 2622-24	Standard Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry
ASTM D 4239-18	Standard Test Method for Sulfur in the Analysis Sample of Coal and Coke Using High-Temperature Tube Furnace Combustion
ASTM D 4294-21	Standard Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry
ASTM D 6376-10	Standard Test Method for Determination of Trace Metals in Petroleum Coke by Wavelength Dispersive X-ray Fluorescence Spectroscopy
ASTM D 6357-21	Standard Test Methods for Determination of Trace Elements in Coal, Coke, and Combustion Residues from Coal Utilization Processes by Inductively Coupled Plasma Atomic Emission Spectrometry, Inductively Coupled Plasma Mass Spectrometry, and Graphite Fur

3 TYPES

The material shall be of the following types and grades:

a) Raw Petroleum Coke (RPC)

i. *Premium* — Very low sulphur content
ii. *Grade A* — Low sulphur content
iii. *Grade B* — High sulphur content

b) Calcined Petroleum Coke (CPC)

i. *Grade A* — Low sulphur content
ii. *Grade B* — High sulphur content

4 REQUIREMENTS

4.1 The material shall be a petroleum product, free from all foreign matter and visible impurities.

4.2 The material shall also comply with the requirements given in Table 1, when tested according to the appropriate methods as given in col 8 of the table.

4.3 Optional Requirements

In addition to the requirements prescribed in Table 1, the material shall also comply with the requirements in **4.3.1** to **4.3.3** as agreed upon between the purchaser and the supplier.

4.3.1 *Size Analysis*

The product shall meet the requirements of size analysis, the limit and the method of test shall be as agreed upon between the purchaser and the supplier.

4.3.2 *Electrical Resistivity*

The product shall also meet the requirements of electrical resistivity; the values shall be reported as per two-electrode method of IS 7929, or by any other suitable method as agreed upon between the purchaser and the supplier.

4.3.3 *Trace Metals*

In addition to the requirements of trace metals, as given under sl no. (ix) of Table 1, the material shall also meet the requirements of the following trace impurities. The limits and the methods of test for the determination of these trace impurities shall be as agreed upon between the purchaser and the supplier.

a) Titanium (Ti); and
b) Calcium (Ca).

5 SAMPLING

Representative samples of the material shall be drawn and prepared in accordance with IS 1447 (Part 4).

6 PACKING AND MARKING

6.1 Packing

The material shall be supplied in suitable containers as agreed upon between the purchaser and the supplier.

6.2 Marking

6.2.1 Each container shall be marked with the following information and any other information as agreed upon between the purchaser and the supplier:

a) Name, type, grade and mass of the material;
b) Indication of the source of manufacture, initials or trade-mark, if any; and
c) Batch or code number.

6.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

NOTE — In case of selling the material in bulk by road / rail / sea route, clause 6 shall not be applicable and instead packing and marking shall be as agreed upon between the purchaser and the supplier.

Table 1 Requirements for Petroleum Coke
(*Clauses* 4.2, 4.3 *and* 4.3.3)

Sl. No.	Characteristics	Requirement for					Method of Test
		Raw Petroleum Coke			Calcined Petroleum Coke		
		Premium	Grade A	Grade B	Grade A	Grade B	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i	Moisture content (as received), percent by mass, *Max*	10	10	10	–	–	Annex A[a] / ASTM D7582
ii	Moisture content (after initial drying), percent by mass, *Max*	2	2	2	0.1	0.1	
iii	Total Moisture content, percent by mass, *Max*	12	12	12	0.1	0.1	

iv	Ash content, percent by mass, *Max*	0.45	0.45	0.45	0.50	0.50	IS 1448 (Part 126)[a] / ASTM D7582
v	Volatile matter, a) percent by mass, *Min*. b) percent by mass, *Max*.	 6 16	 6 16	 6 16	 – 0.4	 – 0.4	 IS 1448 (Part 134)[a] / ASTM D7582
vi	Density:						
	a) Vibrated bulk, g/cm^3	-	-	-	To Report		IS 1448 (Part 130)
	b) Real[b], g/cm^3 *Min*	-	-	-	2.03	2.03	IS 1448 (Part 133) / IS 1448 (Part 139)
vii	Fixed carbon, percent by mass, *Min*	85	85	85	97	97	Annex B[a] / ASTM D7582
viii	Total sulphur, percent by mass, *Max*	1.0	1.50	2.5	1.25	2.5	IS 1448 (Part 33)[a] / ASTMD 2622 / ASTM D 4239 / ASTM D 4294
ix	Trace Metals:						
	a) Silicon (Si), percent by mass, *Max*	To Report			0.05	0.05	IS 1448 (Part 131)[a] / ASTM D 6376 / ASTM D 6357[c]
	b) Iron (Fe), percent by mass, *Max*	-do-			0.04	0.04	IS 1448 (Part 127)[a] / ASTM D 6357[c]
	c) Vanadium[b] (V), percent by mass, *Max*	-do-			0.03	0.03	IS 1448 (Part 79)[a] / ASTM D 6357[c]
	d) Nickel (Ni), percent by mass, *Max*	-do-			To Report		IS 1448 (Part 128)[a] / ASTM D 6357[c]

[a] In case of disputes, this method shall be the referee method.
[b] For graphite industry a higher real density and low vanadium content product is required; the limits for this may be settled between the purchaser and the supplier.
[c] ASTM D6357 test method is not having testing scope of silicon and iron, however latest equipment are having provision for testing silicon and iron. Lab shall establish precision in equipment before reporting.

ANNEX A
[*Table* 1, *Sl No.* (i)]
DETERMINATION OF MOISTURE CONTENT IN PETROLEUM COKE BY MOISTURE BALANCE

A-1 GENERAL This test method covers determination of moisture (as received basis) in RPC and CPC using moisture balance.

A-2 SUMMARY OF THE TEST METHOD

A known sample is placed in the moisture balance. The moisture balance records the weight of the sample. Determination of moisture content is initiated based on manufacturer's operation manual. The sample is gradually heated to a predefined set temperature. At the set temperature, the sample is heated till weight loss in two consecutive readings is nearly constant. Loss in weight is recorded as moisture content in the sample.

A-3 APPARATUS

Any standard automated moisture balance for determination of moisture in solids.

A-4 PROCEDURE

The sample is crushed to ensure that does not have large lumps. The sample is homogenized by manual mixing. About 20 g of sample is placed on a clean and dry pan of the moisture balance. The moisture balance will record the initial weight of the sample. Proceed with the determination of moisture content as per manufacturer's guidelines to attain a set temperature of 107 ± 3º C. During moisture content determination periodically instrument will measure the sample weight to determine the weight loss. Heating the sample and weighing the sample will be continued till instrument records constant weight loss in two consecutive determinations. When the weight loss is constant the instrument will display the moisture content on the screen.

A-5 REPORT

Report the moisture content up to single decimal point as percent, mass.

A-6 PRECISION

Precision may be calculated, as mentioned in the manufacturer's manual.

NOTE: Halogen Moisture Meter can also be used for this test.

ANNEX B
[*Table* 1, *Sl No.* (vii)]
DETERMINATION OF FIXED CARBON IN PETROLEUM COKE

B-1 GENERAL

B-1.1 Fixed carbon in petroleum coke is the solid residue, other than ash, moisture and volatile matter, obtained by a process of calculation.

B-1.2 Total moisture, ash and volatile matter shall be determined by the methods given col 8 of Table 1.

NOTE — Fixed carbon is made up of carbon mainly, but may contain sulphur, hydrogen, nitrogen and oxygen as contaminants.

B-2 CALCULATION

Fixed carbon, percent by mass = 100 – (Moisture + Ash + Volatile matter)