भारतीय मानक

Indian Standard

IS 1448 (Part 195) : 2023
ISO 10143 : 2019

पेट्रोलियम और उसके उत्पाद — परीक्षण पद्धतियाँ

भाग 195 पेट्रोलियम कोक की प्रतिरोधकता का भाग निर्धारण

Petroleum and its Products — Methods of Test

Part 195 Determination of Resistivity of Petroleum Coke

ICS 75.100.10

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October 2023 **Price Group 5**

Methods for Sampling and Test for Petroleum and Related Products of Natural or Synthetic Origin (Excluding Bitumen) Sectional Committee, PCD 01

NATIONAL FOREWORD

This Indian Standard which is identical to ISO 10143 : 2019 'Carbonaceous materials for the production of aluminium — Calcined coke for electrodes — Determination of the electrical resistivity of granules' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Methods for Sampling and Test for Petroleum and Related Products of Natural or Synthetic Origin (Excluding Bitumen) Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

Certain modifications due to particular needs of Indian petroleum industry have been made, which have been mentioned in National Annex A.

Except the deviations identified in National Annex A, the text of ISO Standard has been approved as suitable for publication as an Indian Standard. Certain conventions are, however,not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

This standard is one of the series of Indian Standards on 'Methods of test for petroleum and itsproducts' IS 1448.

The technical committee has reviewed the provisions of the following International Standardsreferred in this adopted standard and has decided that these are acceptable for use in conjunction with this standard:

InternationalStandard	*Title*
ISO 6375	Carbonaceous materials for the production of aluminium — Coke for electrodes — Sampling
ISO 6997	Carbonaceous materials for the production of aluminium — Calcined coke — Determination of apparent oil content — Heating method
ISO 8723	Carbonaceous materials for the production of aluminium — Calcined coke — Determination of oil content — Method by solvent extraction
ISO 11412	Carbonaceous materials for the production of aluminium — Calcined coke — Determination of water content

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 : 2022 'Rules for rounding off numerical values (*second revision*)'.

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Indian Standard

PETROLEUM AND ITS PRODUCTS — METHODS OF TEST

PART 195 DETERMINTION OF RESISTIVITY OF PETROLEUM COKE

1 Scope

This document specifies a method for the determination of the electrical resistivity of granular carbon (calcined or graphitized) used in the manufacture of carbon electrodes for the production of aluminium.

The measurement of electrical resistivity assists in assessing the extent of coke calcination. The electrical resistivity of the coke aggregate will influence the electrical resistivity of the coke electrodes made from it.

In general, a more highly calcined coke will have a lower electrical resistivity if other factors, such as grain size, are similar.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 6375, *Carbonaceous materials for the production of aluminium — Coke for electrodes — Sampling*

ISO 6997, *Carbonaceous materials for the production of aluminium — Calcined coke — Determination of apparent oil content — Heating method*

ISO 8723, *Carbonaceous materials for the production of aluminium — Calcined coke — Determination of oil content — Method by solvent extraction*

ISO 11412, *Carbonaceous materials for the production of aluminium — Calcined coke — Determination of water content*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

A test portion of the granular carbon is placed in a cylindrical holder which has electrical contacts at the top and bottom. A fixed pressure is applied to the test portion to ensure good electrical contact and a fixed, constant direct current is applied. The voltage drop and the height of the column of granules are measured and the electrical resistivity is calculated.

5 Apparatus

Ordinary laboratory apparatus, plus the following:

5.1 Sample holder and plunger, with removable base for cleaning as shown in Figure 1.

5.2 Length-measuring device, capable of measuring the movement of the compression plunger to ± 0,1 mm.

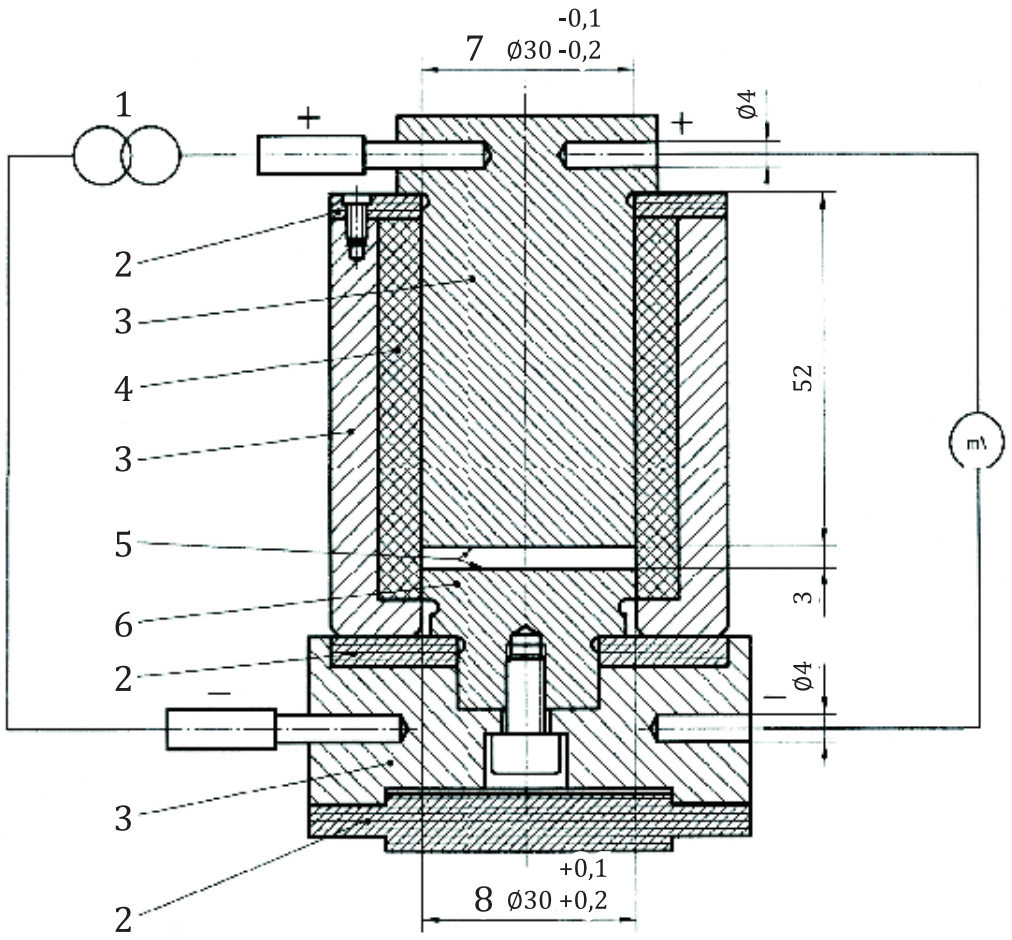
5.3 Plastic reference cylinder, with a height of 22 mm ± 0,01 mm and a diameter of 25 mm, used for calibrating the length-measuring device (5.2).

5.4 Test machine, capable of applying a constant pressure of 3 MPa ± 1 % to the plunger (5.1), giving a velocity to the plunger of not more than 10 mm/s, and with a centering device for the sample holder (5.1).

5.5 Power supply, capable of providing a constant direct current of 1 A ± 0,002 A.

5.6 Voltmeter, capable of measuring to the nearest ± 0,01 mV.

5.7 Oven, capable of being maintained at 110 °C for 12 h.



Key

1 power supply
2 Polytetrafluoroethylene (PTFE)
3 brass
4 agate
5 hard-chromed surfaces
6 stainless steel
7 plunger
8 mould

Figure 1 — Sample holder and plunger, showing electrical connections

6 Sampling and preparation

6.1 Sampling

Sample the coke in accordance with ISO 6375.

6.2 Sample preparation calcined petroleum coke

Heat the sample to 110 °C ± 5 °C for 12 h, using the oven (5.7) in accordance with ISO 11412.

De-oil the sample by washing with dichloromethane in accordance with ISO 8723 or by heating in accordance with ISO 6997.

Crush all the sample material > 1,4 mm in a jaw crusher with a minimum aperture of 1,5 mm so that all the sample material passes through the 1,4 mm sieve.

Sieve and collect the fraction 1,4 mm to 1,0 mm for the analysis.

6.3 Sample preparation all samples

Divide the sample, by sieving, into three fractions (I, lla, and Ill) as follows:

— fraction l > 1,0 mm;

— fraction lla 0,5 mm to 1,0 mm;

— fraction Ill < 0,5 mm.

Crush fraction l so that it is predominantly 0,5 mm to 1,0 mm and sieve to form fraction llb.

Mix fractions lla and llb thoroughly.

Wash the sample with dichloromethane in accordance with ISO 8723.

Heat the sample to 120 °C ± 2 °C for 30 min using the oven (5.7), allow to cool in a desiccator and weigh to the nearest 1 g or better. Repeat the operations of heating, cooling and weighing until the difference between two successive weighings does not exceed 1 % of the original sample mass.

7 Procedure

7.1 Test portion

Weigh out 15 g ± 0,1 g of the mixture of fraction 1,4 mm to 1,0 mm (6.2) or of fractions lla and llb (6.3).

7.2 Setting up the test machine

Place the plastic reference cylinder (5.3) in the sample holder (5.1).

Place the sample holder with plunger (5.1) in the test machine (5.4) and apply a pressure of 3 MPa. Set the length-measuring device (5.2) to the reference level of 22 mm. Connect up the electrical wires and switch on the power supply (5.5).

7.3 Determination

Pour the test portion into the sample holder, insert the plunger and place the assembly in the test machine.

Apply a pressure of 3 MPa to the test portion and after 30 s record the voltage drop and the height of the column of granules.

7.4 Number of determinations

Carry out two determinations using a fresh test portion for each determination.

Clean the base and the plunger surfaces carefully after each determination.

8 Expression of results

Calculate the electrical resistivity, ρ, expressed in microohm metres, of the calcined coke using Formula (1):

$$\rho = \frac{S \times U}{I \times h} \quad (1)$$

where

S is the surface area, in square millimetres, of the sample holder (707 mm^2 for the sample holder shown in Figure 1);

U is the voltage drop, in millivolts;

I is the current applied, in amps (1 A ± 0,002 A);

h is the height, in millimetres, of the column of granules.

Express the result as the arithmetic mean of two determinations.

9 Precision[1)]

9.1 Repeatability

The difference between the values of duplicate determinations, carried out in rapid succession by the same operator using the same apparatus on granules taken from the same test sample, shall not exceed the repeatability limit r = 3 %.

9.2 Reproducibility

The difference between the values of the average of duplicate determinations obtained by two laboratories using this method for the analysis of the same laboratory sample is not expected to exceed the reproducibility limit R = 5 %.

10 Test report

The test report shall include the following information:

a) an identification of the sample;

b) a reference to this document;

c) the method used;

d) the date of the test;

e) the result(s), including a reference to the clause which explains how the results were calculated;

f) any deviations from the procedure;

g) any unusual features observed.

1) ISO 5725:1986, Precision of test methods — *Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests* (now withdrawn), was used to obtain the precision data.

National Annex A

(*National Foreword*)

Clause/Subclause	Modifications
1 Scope	Replace the existing clause with the following:

1 Scope

This document specifies a method for the determination of the electrical resistivity of granular carbon (calcined or graphitized).

The measurement of electrical resistivity assists in assessing the extent of coke calcination.

In general, a more highly calcined coke will have a lower electrical resistivity if other factors, such as grain size, are similar.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

This Indian Standard has been developed from Doc No.: PCD 01 (20132).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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