भारतीय मानक IS 1448 (Part 127) : 2023

Indian Standard

पेट्रोलियम और उसके उत्पाद — परीक्षण पद्धतियाँ

भाग 127 पेट्रोलियम कोक में लोहे का निर्धारण

(पहला पुनरीक्षण)

Petroleum and its Products — Methods of Test

Part 127 Determination of Iron in Petroleum Coke

(First Revision)

ICS 75.080; 75.160.10

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December 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

FOREWORD

This Indian Standard (Part 127) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods for Sampling and Test for Petroleum and Related Products of Natural or Synthetic Origin (Excluding Bitumen) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

The presence and concentration of iron elements in a petroleum coke are major factors in the suitability of the coke for various uses. This test method provides procedure for measuring commercially important metallic impurity iron in coke samples.

This standard was first published in 1988. The first revision has been brought out to keep pace with the latest technological developments and international practices. In this revision following major changes have been incorporated:

a) Applicable range of the method has been incorporated in the scope;
b) Heating temperature and time for ash have been modified; and
c) Wavelength has been changed from 490 nm to a range of 505 nm to 515 nm.

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

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*)’.

Indian Standard

PETROLEUM AND ITS PRODUCTS — METHODS OF TEST

PART 127 DETERMINATION OF IRON IN PETROLEUM COKE

(First Revision)

1 SCOPE

This standard (Part 127) prescribes the method of test for determination of iron in petroleum coke. This test method is suitable for measuring 0.1 mg/l to 5 mg/l with matrix matched standards.

2 APPARATUS

2.1 Spectrophotometer

UV-visible spectrometer shall be capable of measuring wavelength ranging from 200 nm to 850 nm and records or reads the molecular absorption at each wavelength versus a blank measurementusing 1 cm absorption cell.

2.2 Platinum Crucible with Cover — 30 ml to 50 ml capacity

2.3 Volumetric Flasks — 100 ml, 500 ml and 1 000 ml capacity

2.4 Pipettes — 2 ml, 5 ml, 10 ml, 20 ml and 25 ml capacity

2.5 Muffle Furnace

Electric muffle furnace with a working temperature range of 1 100 °C to 1 200 °C capable of regulation of temperature at 900 °C ± 50 °C.

2.6 Hot Plate

3 REAGENTS

3.1 Reagent-grade chemicals shall be used in all tests and reagents shall be of sufficiently high purity to permit their use without lessening the accuracy of the determination.

3.2 Hydroxylamine Hydrochloride ($NH_2OH.HCl$ 10 Percent Solution)

Into a 250 ml beaker, weigh 25 g $NH_2OH.HCl$, add about 100 ml water and stir until dissolved. Transfer to a 250 ml flask and dilute with water to mark. Mix well. Store in a refrigerator and discard if any colour develops. This solution is stable for one week.

NOTE — One millilitre of this reagent is sufficient upto 100 µg of Fe.

3.3 2,4-Dinitrophenol Indicator (0.1 Percent Solution)

Dissolve 0.5 g of 2,4-dinitrophenol in water and dilute to 500 ml with water.

3.4 Ortho Phenanthroline (1,10-Phenanthroline) (1.5 Percent Alcoholic Solution)

Take 1.5 g weight of o-phenanthroline monohydrate in to 200 ml beaker, add 95 percent ethanol and stir until dissolved. Transfer to 100 ml graduated flask and dilute to the mark with ethanol. Store in a refrigerator and discard if any colour develops.

3.5 Ultra-Pure Hydrochloric Acid (6 N Solution)

3.5.1 12 N concentrated hydrochloric acid containing less than 0.000 05 percent iron.

3.5.2 Dilute 12 N concentrated hydrochloric acid with an equal volume of water.

3.6 Ammonium Hydroxide, NH_4OH (6 N Solution)

Take 120 ml of distilled water, add 80 ml of concentrated ammonium hydroxide (15 N), dilute to 200 ml in a volumetric flask. Mix well and store in a polyethylene bottle.

3.7 Bromine Water

Add 10 ml bromine to 1 litre of water. Mix well and allow stand for 24 h.

3.8 Standard iron powder or wire having minimum 99.99 percent purity.

3.9 Lithium tetraborate ($Li_2B_4O_7$) powder (high purity) or lithium metaborate ($LiBO_3$) powder (high purity).

4 PROCEDURE

4.1 Standard Stock Solution

Prepare standard stock solution from high purity iron metal or oxide or from iron salt. Stock solution of 1 000 mg/l is required for the preparation of dilute standard in the range of 0 mg/l

to 50 mg/l. Commercially available standard solution with traceable certificate may also be used.

4.2 Preparation of Standard Iron Solution, (100 mg/l, 1 ml = 0.1 mg Fe)

Dissolve 0.103 2 g of pure iron powder or wire (*see* 3.8) in 6 N HCl. Add about 5 ml of bromine water solution and boil off to remove excess bromine. Cool, transfer to a 1 litre volumetric flask, dilute to mark and mix well. This solution may be used as a 'primary standard' or it may be standardized in the usual manner volumetrically against a standard potassium dichromate solution.

4.3 Preparation of Standard Iron Graph

4.3.1 *Low Range* (1 mg/l *to* 10 mg/l)

Transfer 1 ml, 2 ml, 4 ml, 6 ml, 8 ml and 10 ml aliquot of standard iron solution (*see* 4.2) into 100 ml volumetric flask. Bring the volume to approximately 50 ml with distilled water. Place about 20 ml of distilled water in a separate 100 ml volumetric flask for preparing blank solution.

4.3.2 *High Range* (5 mg/l *to* 30 mg/l)

Transfer 5 ml, 10 ml, 15 ml, 20 ml, 25 ml and 30 ml aliquot of standard iron solution (*see* 4.2) into 100 ml volumetric flask. Bring the volume to approximately 50 ml with distilled water. Place about 20 ml of distilled water in a separate 100 ml volumetric flask for preparing blank solution.

4.3.3 Place the volumetric flask containing standards and blank on a shallow water bath at 55 °C to 60 °C or on a hot plate at 55 °C to 60 °C. Add 2 ml of 10 percent hydoxylamine hydrochloride solution and stir. Allow the solutions to remain in the bath for 5 min.

4.3.4 Remove the flasks from the water bath. Add 0.5 ml of 0.1 percent 2,4-dinitrophenol indicator solution. Then, add 6 N ammonium hydroxide solution drop wise with stirring until the solution assumes a slight yellow colour. The *p*H is now about 4.4. Add very carefully drop wise 6 N HCl until the yellow colour of the solution is just discharged. The *p*H of the solution is about 2.6. Now add 1 ml of 1.5 percent o-phenanthroline solution. Stir well and cool to room temperature. The orange coloured iron, which is immediately produced, is due to the formation of the ferrous-o-phenanthroline complex. Make up to the 100 ml mark with distilled water and mix well. Allow the solutions to stand for minimum 15 min.

4.3.5 Transfer a suitable portion of the standard solutions to a 1 cm absorption cell and measurethe absorbance against reagent blank at maximum wavelength (505 nm to 515 nm). Record the absorbance of each standard solution (E_S) and the absorbance of reagent blank (E_b).

4.3.6 Subtract the blank absorbance from absorbance reading obtained for the standard solutions and plot the corrected absorbance ($E_S - E_b$) against milligram iron content or mg/l iron content in each standard solution.

4.4 Procedure for Analysis of Coke (Both Calcined and Raw)

4.4.1 Weigh 25 g of dried and crushed petroleum coke sample and sieve using 250 micron sieve and collect the coke sample passing through 250 micron sieve. Weigh accurately 1 g to 2 g of sieved sample in to a previously cleaned and heated platinum crucible. Place the platinum crucible in a muffle furnace and heat directly to 525 °C with the furnace door opened approximately 7 mm to allow exchange of combustion gases and air until all carbonaceous matter is removed. Close the muffle furnace door and heat 700 °C to 750 °C for 4 h to 5 h or until all the carbonaceous matter was removed. Transfer the platinum dish containing the ash to a desiccator and cool to room temperature.

4.4.2 Weigh around 1 g of lithium tetraborate powder and carefully sprinkle it over the ash in the platinum crucible. Place the platinum crucible in the muffle furnace previously heated to 900 °C to 950 °C for 10 min to 15 min or until the fusion completes.

4.4.3 Allow the platinum crucible containing fusion melt to cool to room temperature. Add 25 ml of ultra-pure hydrochloric acid. Place the crucible on a hot plate and heat the solution just to boil and maintain for 30 min with constant stirring to dissolve completely the melt.

4.4.4 Remove the crucible from the hot plate, rinse down the walls of the crucible with water and quantitatively transfer the solution to a 100 ml flask and make up to the mark with distilled water. This solution is ready for iron analysis.

4.4.5 Pipette out 20 ml aliquot of sample solution and 20 ml of distilled water into 100 ml volumetric flask and develop the colour as described in 4.3.1 to 4.3.4 for the standard. Measure the absorbance (E_S) sample and (E_b) absorbance of the blank solution. Use the appropriate calibration graph and determine the concentration of iron content in 100 ml of the sample solution.

NOTE — Cadmium, zinc, nickel, cobalt and copper form soluble complexes with 1,10-phenanthroline. When these metals are present in the sample, the intensity of the colour

gets retarded and the absorbance will be reduced. In general, each 0.1 mg of iron required 1.7 ml to 2.0 ml of 1,10-phenanthroline. To overcome the interference effect, an additional 1 ml of 1,10-phenanthroline to be added to the solution.

5 CALCULATION

Determine the iron in mg/l or mg present in 100 ml of sample solution (M) as described in **4.4.5** and calculate as given below:

$$\text{Iron content in coke sample} = \frac{\text{M} \times 10^{-3}}{W} \text{ (M, in mg)}$$

$$\text{or} \quad \frac{\text{M} \times V \times 10^{-3}}{W} \text{ (M, in ppm)}$$

where

W = mass, in g, of coke sample taken for analysis; and

V = volume, in ml, of sample solution.

6 REPORT

Report the iron content in mg/l in two significant figures.

7 PRECISION

Method precision shall be established based on the round robin outcome.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

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

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