भारतीय मानक Indian Standard

> पेट्रोलियम और उसके उत्पाद — परीक्षण पद्धतियाँ

भाग 110 आसुत ईंधन का कोल्ड फिल्टर प्लगिंग बिंदु

(पहला पुनरीक्षण)

Petroleum and its Products — Methods of Test

Part 110 Cold Filter Plugging Point of Distillate Fuels

(First Revision)

ICS 75.080; 75.160.20

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Price Group 7

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 110) (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.

Cold filter plugging point (CFPP) is used to estimate lowest temperature that a fuel will flow easily incertain fuel systems. This is important as in cold climatic conditions, a high cold filter plugging point will clog up vehicle engines more easily.

This standard was first published in 1981 and was based on IP 309/80. 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 made:

- a) The scope has been modified to make it applicable to bio diesel as well;
- b) Automated apparatus has been included and details of temperature measuring device has been updated;
- c) Sampling, Apparatus and Reporting clauses have been updated; and
- d) Reference clauses have been incorporated.

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

In reporting the results 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 110 COLD FILTER PLUGGING POINT OF DISTILLATE FUELS

(*First Revision*)

1 SCOPE

This procedure describes for manual and automated determination of low temperature operability of distillates fuels. This test procedure is applicable for CFPP measurement of middle distillate fuels, automotive diesel fuels, gas oil, bio diesel and fuel containing flow improving additives.

2 REFERENCES

The standard given below contains provisions which, through reference in this text, constitutes provisions of this standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standard:

IS No. Title

- IS 1447 (Part 1): Methods of sampling of petroleum 2021 and its products: Part 1 Manual sampling (second revision)
- IS 2480 (Part 1): Specification for general 1983 purpose glass thermometers: Part 1 Solid-stem thermometers (second revision)

3 PRINCIPLE

The test specimen is cooled continuously under prescribed condition at the interval of 1 °C, and specified vacuum is applied to pass the fuel through standardized filter mesh to fill the standard pipette. When test fuel complete cycle of passing through standardized filter mesh, fill the pipette and return to base test jar within 60 s, then test fuel temperature is further automatically reduced to next 1 °C lower. Test cycle is stopped when till test fuel fail to complete the cycle of filling pipette or further fuel returns to base vessel within 60 s. Highest recorded temperature at which fuel was passed through pipette up to or within 60 s is recorded as CFPP.

4 TERMINOLOGY

4.1 Cold Filter Pugging Point (CFPP)

The highest temperature, expressed in multiples of 1 °C, at which a given volume of fuel fails to pass through a standardized filtration device, undergoing prescribed cooling conditions within specified time.

5 APPARATUS

The typical manual apparatus required is illustrated in Fig. 1 and consist of the following components:



All dimensions in millilmetres.

FIG. 1 GENERAL SCHEMATIC OF TYPICAL MANUAL APPARATUS

5.1 Test Jar or Cylinder

A cylinder test jar of clear glass, flat bottom with internal diameter 31.0 mm to 32.0 mm, a wall thickness of 1.0 mm to 1.5 mm, and a height of 115 mm to 125 mm. The jar shall have etched mark at the 45 ml nominal volume level with tolerance of less than 1 ml volume.

5.2 Lowest Jacket

A water-tight cylindrical jacket of brass, flat

bottom, with the dimensions given in Fig. 2.

5.3 Supporting Ring

Supporting ring is an adaptor made of oil resistant polymer, elastomer, rubber, plastic or other suitable material is required to the position of jar and the jacket respectively in relation to the jacket and the cooling bath. A suitable form is shown in Fig. 3. This design may be modified to suit existing cooling baths.



All dimensions in millimetres. FIG. 2 BRASS JACKET (WATER-TIGHT)



All dimensions in millimetres. FIG. 3 SUPPORTING RING

5.4 Insulating Ring

Insulating ring is made of oil thermal resistant polymer, elastomer, rubber, plastic or other suitable material, placed in the bottom of the jacket which provides thermal insulation for the bottom of the test jar. A suitable form is shown in Fig. 4. The design of insulating ring may be altered to suit the cooling baths used.

5.4 Spacers

Spacer is made of oil resistant polymer, elastomer, rubber, plastic or other suitable material which is to be placed as shown in Fig. 1 around the test jar and provide insulation for the test jar from the sides of the jacket. The spacer shall fit closely to the test jar and loosely inside the jacket (*see* Fig. 4).

5.5 Stopper

Stopper made of oil resistant polymer, elastomer, rubber, plastic or other suitable material,

non-metallic, non-absorbent, to hold the test jar and the support ring as shown in Fig. 5. Stopper should have three holes to accommodate the pipette, the temperature measuring device, and another hole to allow venting of the jacket system. The upper part of the stopper may have a scoop to allow the thermometer to read lower temperature. A marker is useful on the upper surface of the stopper to locate the temperature measuring device in relation to the bottom of the test jar. A fastening device is useful to retain the temperature measuring device in the proper position.

5.6 Pipette

A 20 ml pipette made of clear glass with a calibration mark corresponding to a controlled volume of 20 ml \pm 0.2 ml located at a point 149 mm \pm 0.5 mm from the bottom point of the pipette (*see* Fig. 6). Pipette is connected to the filter unit with screw cap, O-rings and brass spacers.



All dimensions in millimetres. FIG. 4 INSULATING RING AND SPACERS



All dimensions in millimetres.

FIG. 5 STOPPER WITH HOLES FOR THERMOMETER, PIPETTE AND VENT



All dimensions in millimetres

FIG. 6 PIPETTE

5.7 Filter Unit

Pictorial representation of filter unit is illustrated in Fig. 7. It consists of following elements which are given below.

5.7.1 Brass Body

Brass body is consisting with threaded cavity that houses the wire mesh holder. The cavity is fitted with an O-ring of oil-resistant plastics. The internal diameter of the central tube is $4 \text{ mm} \pm 0.1 \text{ mm}$.

5.7.2 Brass Screw Cap

Brass screw cap is the upper part of filter unit body to the lower part of the pipette. Brass screw cap provides leak-free joint. An example of suitable connection is shown in Fig. 7.

5.7.3 Disc

Disc dimension is 15 mm \pm 0.1 mm diameter, plain weave stainless steel wire mesh gauze with a nominal aperture size of 45 μ m. The nominal diameter of the wire is 32 μ m with size tolerance

individual aperture is as follows:

- a) No aperture size shall exceed the nominal size by more than 22 μm;
- b) The average aperture size shall be within \pm 3.1 μ m of the nominal size; and
- c) Not more than 6 percent of the apertures shall be above the nominal size by more than 13μm.

5.7.4 Filter Holder of Brass Assembly

Filter holder brass assembly is where a disc of wire mesh gauze is held securely. The diameter of the exposed portion of the gauze shall be 12 mm + 0.1 mm - 0.0 mm (see Fig. 8).

5.7.5 Brass Cylinder

Brass cylinder is threaded on the outside which is screwed into the cavity of the filter unit body to clamp the filter holder. The lower end should have four openings for the specimen to flow into the filter unit.



All dimensions in millimetres.

FIG. 7 FILTER UNIT



All dimensions in millimetres. FIG. 8 FILTER HOLDER (BRASS)

5.8 Temperature Measuring Device

A suitable liquid-in-glass thermometer having ranges conforming to the requirements prescribed in IS 2480 (Part 1), or an electronic temperature measurement device, digital contact thermometer (DCT) may be used.

5.9 Cooling Bath

Any suitable cooling bath may be used which shall have suitable shape and size to contain the jacket in a stable and up-right position at the desired depth. The bath should be capable to with stand the desired temperature value by a refrigeration unit with a homogenous temperature in the bath by stirring or other means.

5.10 Vacuum System

Partly water filled large glass bottle connected to a suitable vacuum pump may act as constant vacuum reservoir. The constant 200 mm water gauge vacuum is maintained in to the reservoir by ensuring unremitting air bleed of 3 l/h to 4 l/h, or by a suitable regulating device. Manual test apparatus layout is illustrated in Fig. 1.

5.11 Timing Device

Any suitable electronic time measuring device with minimum read ability of 0.2 s or lower with an accuracy of 0.1 percent over a span of 15 minutes may be found suitable.

5.12 Automated Apparatus

An automated apparatus shall be like manual CFPP apparatus with automation of CFPP measurements.

Automated CFPP apparatus shall comply with **5.1** to **5.12** for platinum resistance thermometers, cooling system, vacuum system, and suitable electronic control and measurement devices. The cooling system may be a refrigeration unit capable of maintaining the required temperature and also of automatically changing the bath temperature at the appropriate stage. The vacuum pump should have enough power to ensure a regulated air flow rate at a minimum of $15 \text{ l/h} \pm 1 \text{ l/h}$ and maintain a constant vacuum of 200 mm ± 1 mm during complete test.

6 SAMPLING

6.1 Draw samples in accordance to IS 1447 (Part 1) or other suitable methods.

6.2 Keep samples away from direct sunlight or sources of direct heat.

6.3 Filter the sample through dry lint-less filter paper at a temperature of 15 °C or higher.

7 APPARATUS PREPARATION

7.1 Prepare instrument ready for CFPP testing complying with manufacturer's instruction.

7.2 Dismantle filter unit after each test and clean with suitable light solvents and dry for next use.

7.3 Reassemble the filter unit and pipette, tighten thread by hand and ensure no leakage at the screw cap.

7.4 Many automated apparatuses are making provision to perform cleaning and drying without dismantle.

Sl No.	Thermometer	Temperature Range	Schedule Mark as per IS 2480 (Part 1)
(1)	(2)	(3)	(4)
i)	High range for CFPP down to -30 °C	- 38 °C to + 50 °C	2
ii)	Low range from CFPP below -30 °C	-80 °C to $+20$ °C	1
iii)	Cooling bath	- 80 °C to + 20 °C	1

8 PROCEDURE

8.1 Place dry insulating ring and spacers into the jacket. Bring the temperature of the sample and container to a temperature at least 10 °C below the expected initial boiling point of the material.

8.2 Pour the filtered sample into a clean and dry test jar to the mark at 45 ml.

8.3 Close the test jar with the stopper carrying the pipette with filter unit and the appropriate thermometer or temperature measuring device.

8.4 Use a low range thermometer if the expected CFPP is below - 30 °C.

8.5 Apparatus should be adjusted and to ensure that the bottom of the filter unit rests on the bottom of the test jar and position the thermometer so that its lower end is 1.3 mm to 1.7 mm above the bottom of the test jar.

8.6 No part of the thermometer should be in contact with the side of the test jar or the filter unit.

8.7 Insert the test jar assembly in a stable vertical position into the jacket.

8.8 If the jacket is not an integral part of the cooling bath, place the jacket vertically to a depth of 83 mm to 87 mm in the cooling bath. Cooling bath should be maintained at the temperature of - $34 \text{ }^{\circ}\text{C} \pm 0.5 \text{ }^{\circ}\text{C}$.

8.9 Connect the pipette to the vacuum system and close off the vacuum line. Switch on the vacuum source and ensure a regulated air flow rate of 15 l/h in the vacuum regulator. Check that the U-tube manometer (if used) indicates a 200 mm \pm 1 mm depression 2 kPa \pm 0.05 kPa or electronic vacuum regulator maintain pressure of 2 kPa \pm 0.05 kPa.

8.10 Start the test immediately after insertion of the test jar assembly into the jacket in the cooling bath.

8.11 Continue testing at the multiple of 1 °C by applying the regulated vacuum while starting timing, causing the sample to be drawn through the fine wire mesh into the pipette. Once the sample has reached the mark on the pipette, stop timing and release the vacuum on the pipette which allow the sample to return to the test jar.

8.12 Abandon the testing if the time to reach the mark on the pipette exceeds 60 s on the first filtration and repeat the testing with fresh portion of sample starting at a higher temperature.

8.13 Continue testing and repeat the aspiration

operations at each 1 °C decrease of the sample temperature until the temperature is reached at which the pipette is not filled to the 20 ml mark within 60 s. Record the temperature at which this last filtration was commenced as CFPP.

8.14 If the filter has not plugged when the temperature of the sample reaches -20 °C, continue the test by using a second cooling bath maintained at -51 °C ± 1 °C. Quickly transferring the test jar and filtration assembly to a new jacket placed on the alternate lower temperature cooling bath.

8.15 Many automated instruments are having single cooling bath and capable to reach temperature from $34 \text{ }^{\circ}\text{C} \pm 0.5 \text{ }^{\circ}\text{C}$ to $-51 \text{ }^{\circ}\text{C} \pm 1 \text{ }^{\circ}\text{C}$ within two and half min.

8.15 Repeat the operations **8.8** to **8.13** for each 1 °C decrease of the sample temperature.

8.16 In case, if temperature of test specimen reaches – 35 °C and filter has not been plugged when, quickly transfer the filtration assembly along with test jar to next cooling bath maintained at – 67 °C \pm 2 °C. In case of automated apparatus using single cooling bath, temperature should reach within 2 min 30 s. Repeat the operations **8.8** to **8.10** at each 1 °C decrease of the specimen temperature.

8.17 In case, specimen reaches to temperature -51 °C and filter has not plugged than discontinue the test and record as 'not plugged at -51 °C'.

NOTES

1 Some latest CFPP instruments are capable of maintaining test specimen temperature up to -90 °C. User may continue to lower temperature CFPP measurement with such instruments, however cooling bath temperature must be at least 16 °C than the test specimen.

2 In case, if after cooling in accordance with the procedures, the sample fills the pipette to the mark in less than 60 s, however fails to flow back completely into the test jar when the pipette vacuum is released before the start of the next aspiration. Record the temperature at the commencement of the filtration as the CFPPs.

3 Some automated apparatus are using electronic temperature devices, timing, vacuum control, cooling systems, optical sensors to monitor the sample level in the pipette, and recording the information related to the test.

9 REPORTING

Test specimen temperature recorded at the beginning of last filtration will be reported as CFPP of test fuel. In case, if the test specimen temperature reaches -51 °C without filter plugging, it should be reported as 'Not plugged at -51 °C'.

Result 2:

10 PRECISION

Precision of this method either manual or automated are as follows:

Repeatability	Reproducibility
$0.033 \times (30 - x)$	$0.092 \times (30 - x)$

where

x = average of two results. Precision of typical CFPP values of x are given as under:

Recult	1	•
Result	T	٠

<i>CFPP</i> , ⁰C	Repeatability, °C
0 to - 3	1
- 4 to - 33	2
- 33 to - 35	3

<i>CFPP</i> , [◦] C	<i>Reproducibility</i> , °C	
0 to - 3	3	
- 4 to -14	4	
- 15 to - 25	5	
- 26 to - 35	6	

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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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.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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