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GB/T 3535-2006

Replacing GB/T 3535-1983

Petroleum products - Determination of pour point

(ISO 3016:1994, MOD)

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Issued by: **General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China;**

Standardization Administration of the People's Republic of China.

Table of Contents

Foreword

This Standard modifies and adopts ISO 3016:1994 "*Petroleum Products - Determination of Pour Point*".

This Standard is redrafted based on ISO 3016:1994.

In order to be suitable for Chinese situation, this Standard makes modifications when adopting ISO 3016:1994. The technical differences between this Standard and ISO 3016:1994 are as follows:

- Add the regulations of "*taking average value of two results of repeated measurement as the test results"*.

In order to be ease of use, this Standards makes editorial changes as follows:

- Wording expression of repeatability and reproducibility are modified based on the habit of our country.

This Standard replaces GB/T 3535-1983 "*Petroleum - Determination of Pour Point".*

Main technical differences between this Standard and GB/T 3535-1983 are as follows:

- The name of this Standard is changed to "*Petroleum Products - Determination of Pour Point"* from "*Petroleum - Determination of Pour Point"*.

- In Clause "Scope", provision "*general steps can be used for measurement of oil pour point, but precision is not applicable"* is added as NOTE;

- Clause "*reagents and materials"* is added; and common refrigerant preparation of -18°C, -33°C, -51°C and -69°C cold bath is supplemented;

- For sleeve in pour point tester, it defines that only metal sleeve can be used; and glass sleeve in the GB/T 3535-1983 is deleted;

- Test procedures are separately described in accordance with two conditions: above -33°C for pour point, and blow or equal to -33°C for pour point. Test procedures of GB/T 3535-1983 were described in accordance with three conditions: 33°C~-33°C, above 33°C, and below -33°C;

- For oil products of which the pour point specification value is not multiple-times of 3°C, it may conduct the test based on 6.9; result shall report if samples can pass or not pass the specification value;

- Add automatic instrument, however, the precision is not applicable;

- Repeatability and reproducibility of this Standard are both applicable to determination of oil products (including fuel oil, residual fuel oil) pour point; while reproducibility of GB/T 3535-1983 is not applicable to determination of oil products

(including fuel oil, residual fuel oil) pour point;

- Add thermometer used for melting point and relevant technical conditions.

Annex A of this Standard is normative.

This Standard was proposed by Sinopec Group.

This Standard shall be under the jurisdiction of SINOPEC Research Institute of Petroleum Processing (RIPP).

Drafting organization of this Standard: SINOPEC Research Institute of Petroleum Processing (RIPP).

Main drafters of this Standard: Guo Tao, and Chen Jie.

Previous version replaced by this Standard is as follows:

— GB/T 3535-1983.

Petroleum products - Determination of pour point

WARNING — The use of this Standard may involve hazardous materials, operations and equipment. This Standard does not purport to address all safety problems associated with its use. It is the responsibility of the user of this Standard to establish appropriate safety and health practices, and determine the applicability of regulatory limitations prior to use.

1 Scope

This Standard specifies a method for determination of the pour point of petroleum products. A separate procedure suitable for determination of lower pour point (see 6.10) of fuel oils, heavy lubricant base stock, and products containing residual fuel components is also described.

NOTE: A method for the pour point of crude oils is under development. The pour point of crude oils may be determined by the general procedure described in this Standard, but some crude oils may need a modified pretreatment to avoid the loss of volatile material. The precision in this Standard was derived on a sample matrix that did not include crude oils (see NOTE in Clause 8).

2 Terms and definitions

For the purposes of this Standard, the following term and definition applies.

pour point

The lowest temperature at which a sample of petroleum product will continue to flow when it is cooled under specified standard conditions.

3 Principle

After preliminary heating, the sample is cooled at a specified rate and examined at intervals of 3°C for flow characteristics. The lowest temperature at which movement of the sample is observed is recorded as the pour point.

4 Reagents and materials

4.1 Sodium chloride (NaCl), crystals.

4.2 Calcium chloride ($CaCl_2$), crystals.

4.3 Carbon dioxide ($C0_2$), solid.

4.4 Coolant liquid: acetone, methanol or petroleum naphtha.

the sample to a temperature greater than 45°C to effect the transfer to the test jar, or when it is known that a sample has been heated to a temperature higher than 45°C during the preceding 24 h, or when the thermal history of the sample is not known, keep the sample at room temperature for 24 h before testing it.

6.2 Close the test jar with the cork carrying the high-cloud-and-pour thermometer or, if the expected pour point is above 36°C, with the melting point thermometer (annex A). Adjust the position of the cork and thermometer so that the cork fits tightly, the thermometer and the test jar are coaxial, and the thermometer bulb is immersed to a depth which places the beginning of the capillary 3 mm below the surface of the sample.

6.3 Subject the sample in the test jar to a preliminary treatment, appropriate to its pour point, in accordance with 6.4 or 6.5.

6.4 Samples having pour points above - 33°C shall be treated as follows.

6.4.1 Heat the sample without stirring to 9°C above the expected pour point, or to 45°C, whichever is greater, in a bath maintained at 12°C above the expected pour point, but at least 48°C.

6.4.2 Transfer the test jar to a bath maintained at 24°C ± 1.5°C.

6.4.3 When the sample temperature reaches 9°C above the expected pour point (estimated as a multiple of 3°C), commence observations for flow in accordance with 6.7.

6.4.4 If the sample has not ceased to flow when the temperature has reached 27°C, carefully remove the test jar from the bath, wipe the outer surface with a clean cloth moistened with wiping fluid (4.5), and place it in the 0°C bath (5.7) in accordance with 6.6. Make observations for flow in accordance with 6.7 and cool as specified in the schedule given in 6.8.

6.5 Samples having pour points of - 33°C and below shall be treated as follows.

6.5.1 Heat the sample without stirring to 45°C in a bath maintained at 48°C, and cool to 15°C in a bath maintained at 6°C ± 1.5°C.

6.5.2 When the temperature has reached 15°C, carefully remove the test jar from the water bath, wipe the outer surface with a clean cloth moistened with wiping fluid, remove the high-cloud-and-pour thermometer and replace it with the low-cloud-and-pour thermometer (annex A). Place the test jar in the 0°C bath in accordance with 6.6. Successively place the test jar into lower-temperature baths in accordance with the schedule specified in 6.8.

6.5.3 When the temperature reaches 9°C above the expected pour point, commence observations for flow in accordance with 6.7.

6.6 Ensure that the disc, gasket and the inside of the jacket are clean and dry, and place the disc in the bottom of the jacket. The disc and jacket shall have been placed in the cooling medium a minimum of 10 min before the test jar is inserted. Place the gasket around the test jar approximately 25 mm from the bottom, and insert the test jar into the

specification temperature is reached. Report the sample as passing or failing the specification limit.

6.10 For fuel oils, heavy lubricant base stock and products containing residual fuel components, the result obtained by the procedure given in 6.1 to 6.8 is the upper (maximum) pour point. If required, determine the lower (minimum) pour point by heating the sample while stirring to 105°C, pouring it into the jar, and determining the pour point as given in 6.2 to 6.8.

6.11 If automatic testing instruments are used, the user shall ensure that all of the manufacturer's instructions for calibration, adjustment and operation of the instrument are followed. As the precision of automatic pour-point testers has not been determined, in any case of dispute, the pour point shall be determined by the manual method described herein and shall be considered as the reference test.

7 Expression of results

Add 3°C to the temperature recorded in 6.7.4 or 6.10 and report this as the pour point, or lower pour point, as applicable.

8 Precision

To judge the reliability of test results according to following stipulations (95% confidence level).

8.1 Repeatability r

The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material, in the long run, in the normal and correct operation of the test method shall not exceed 3°C.

8.2 Reproducibility R

The difference between two single and independent results, obtained by different operators working in 'different laboratories on nominally identical test material, in the long run, in the normal and correct operation of the test method shall not exceed 6°C.

NOTE: The precision statements were prepared with data on ten new (unused) mineral oil-based lubricants and 16 assorted fuel oils tested by 12 co-operators. The mineral oil-based lubricants had pour points ranging from -48°C to -6°C, while the fuel oils had pour points ranging from -33°C to 51°C. The following precision data was obtained:

Name of sample	Repeatability	Reproducibility
Mineral oils lubricants	2.87°C	6.43°C
Fuel oils	2.52 °C	6.59°C

9 Test report

The test report shall contain at least the following information:

1) sufficient details for complete identification of the product tested;

2) a reference to this Standard;

3) the result of the test (see clause 7);

4) any deviation, by agreement or otherwise, from the procedures specified;

5) the date of the test;

6) whether the test was determined by an automatic instrument.

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