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GB/T 6986-2014

Replacing GB/T 6986-1986

Standard test method for cloud point of petroleum products

石油产品浊点测定法

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Foreword

This Standard is drafted in accordance with the rules specified in GB/T 1.1-2009.

This Standard replaces GB/T 6986-1986 *Standard Test Method for Cloud Point of Petroleum*. Compared with GB/T 6986-1986, the main technical changes are as follows:

- The title is changed into *Standard Test Method for Cloud Point of Petroleum Products*;
- Applicable scope is widened; add biodiesel and biodiesel fuel blend (see Chapter 1);
- Add requirements for sample chroma, that is, the chroma shall be no more than 3.5; samples with chroma exceeding 3.5 can also be tested according to this Standard, however, the precision is not applicable (see Chapter 1);
- Add automatic method (see Chapter 1, 8, 9.2 and 10);
- Add the explanation that the manual method means the arbitration method (see Chapter 1);
- Add “normative references” (see Chapter 2), “terms and definitions” (see Chapter 3), “method application” (see Chapter 5), and “report” (see Chapter 11);
- Add the explanation that the humidity shall not be more than 75% during the testing process (see 6.3);
- Add GB-36, GB-37 and GB-38 temperature gauges that conform to the requirements of GB/T 514 (see 7.1.1);
- Requirements for test tube’s inner diameter and the volume at the scale mark are different;
- Description of casing tube is different;
- Add the treatment process of sodium sulfate dehydrating agent (see 7.2.1), and requirements and processing procedures of filter paper (see 7.2.2);
- Cooling bath temperature control is slightly changed;
- There are some changes in transition temperature of samples in cooling bath;
- There are some changes in precision.

This Standard is redrafted by referring to ASTM D2500-11 *Standard Test Method for Cloud Point of Petroleum Products*. Main technical differences between this Standard and ASTM D2500-11 are as follows:

- Add automatic method which is in accordance with ASTM D5771-12 *Standard Test Method for Cloud Point of Petroleum Products (Optical detection classification cooling method)* (see Chapter 1, 8, 9.2 and 10);
- Change the reference standards into current national standards and industry standards in China in the “normative references” (see Chapter 2);
- Add corresponding terms and definitions (see Chapter 3.3, 3.5 and 3.6);
- Add the explanation that the humidity shall not be more than 75% during the testing process (see Chapter 6.3);
- Add GB-36, GB-37 and GB-38 temperature gauges that conform to the requirements of China’s GB/T 514 (see 7.1.1);
- Add requirements for the volume at the test tube’s scale mark (see 7.1.2);
- Add the specifications and requirements for reagent, material and cleaning solvent (see 7.2 and 8.2);
- Add the treatment process of sodium sulfate dehydrating agent (see 7.2.1), and requirements and processing procedures of filter paper (see 7.2.2).

This Standard was proposed by National Technical Committee of Petroleum Products and Lubricant Standardization (SAC/TC 280).

This Standard shall be under the jurisdiction of Petroleum Fuel and Lubricant Branch of National Technical Committee of Petroleum Products and Lubricant Standardization (SAC/TC 280/SC 1).

Drafting organizations of this Standard: SINOPEC Maoming Company.

The participating drafting organizations of this Standard: SINOPEC Lubricant Maoming Company, Lanzhou Lubricant Research & Development Center of PetroChina Co Ltd., SINOPEC Zhenhai Refinery & Chemical Company, SINOPEC Lubricant Shanghai Company, MaoMing Entry-Exit Inspection and Quarantine of the PRC, and SINOPEC Lubricant Beijing Research & Development Center.

Main drafters of this Standard: Dang Chenxia, Wang Xuemei, Pan Xiaoxia, Huang Hongxia, Gao Yun, Li Ling, Liang Yuquan, and Cui Hai’ou.

Previous version replaced by this Standard is as follows:

- GB/T 6986-1986.

Standard Test Method for Cloud Point of Petroleum Products

Warning: This Standard involves certain dangerous materials, operations and equipment, however, it does not put forward suggestions on all safety problems involved. Therefore, proper safety and protection measures shall be established before the users apply this Standard in practice, and the applicability of relevant rules and regulations shall be determined.

1. Scope

This Standard specifies two test methods, namely, manual and automatic methods, for cloud point of petroleum products, biodiesel and biodiesel fuel blend. The manual method is deemed as the arbitration method.

This Standard only applies to testing cloud point of petroleum products, biodiesel and biodiesel fuel blend of which the cloud point is less than 49°C when it is transparent at the thickness of 40 mm.

Note: Chroma of samples tested in accordance with this Standard shall be ≤ 3.5 (according to GB/T 6540); samples with chroma exceeding 3.5 can also be tested according to this Standard, however, the precision is not applicable.

2. Normative references

The following documents are indispensable for application of this document. For the dated documents so quoted, only dated versions apply to this document. For the undated documents so quoted, the latest versions (including all modification sheets) apply to this document.

GB/T 514-2005 Specification for liquid-in-glass thermometers for testing of petroleum products

GB 1922-2006 Petroleum solvents for paints and cleaning

GB/T 4756 Petroleum liquids - Manual sampling (GB/T 4756-1998, eqv ISO 3170:1988)

GB/T 6540 Petroleum products - Determination of colour

GB/T 15000.3 Directives for the work of reference materials (3) - Reference materials

- General and statistical principles for certification (GB/T 15000.3-2008, ISO Guide 35:2006, IDT)

GB/T 15000.7 Directives for the work of reference materials (7) - General requirements for the competence of reference material producers (GB/T 15000.7-2012, ISO Guide 34:2009, IDT)

GB 17602 Commercial hexanes

JB/T 8622 Technical specification and reference table for industrial platinum thermal resistance (JB/T 8622-1997, neq IEC 751:1983)

3. Terms and definitions

Following terms and definitions apply to this Standard.

3.1 Biodiesel

It is a kind of fuel derived from vegetable oil or animal fat, and it is composed of long-chain fatty acid mono-alkyl ester, expressed by BD100.

Note: Typical biodiesel is a kind of monoester produced in the reaction BETWEEN vegetable oil or animal fat AND alcohol (e.g. methyl alcohol or ethyl alcohol) by using catalyst. The fuel may include up to 14 different fatty acids, and the fatty acid methyl ester (FAME) that is transformed through chemical reactions.

3.2 Biodiesel fuel blend

It is a blend of biodiesel and petroleum distillate fuel, expressed by Bxx. In which, xx means the volume percentage of biodiesel.

3.3 Distillate fuels

It refers to the atmospheric or vacuum distillate oil and their product fuels, obtained through primary processing or second processing.

3.4 Cloud point

It means the highest temperature at which the clear-transparent liquid petroleum products, biodiesel and biodiesel fuel blend APPEAR in foggy or turbid state at the first time under the specified conditions because of the appearance of wax crystals. It is expressed by °C.

Note 1: For many observers, wax crystal looks like a cloud in white or milky turbid spots, which wins the name for it. When the sample's temperature decreases to a certain degree, wax crystal will form, and turbid state will appear. For many samples, the crystal first appears at the lower part around the wall of the test tube with the lowest temperature. The difference of sample nature will result in different sizes and positions of crystal and turbidity at the cloud point. Some samples will form large and easy-to-observe crystals, while others form crystals

method or through continuous monitoring of optical system. The highest temperature, at which wax crystal first appears at the bottom of the test tube and presents fog or turbid state, is the cloud point of the sample, expressed by °C.

5. Method application

For petroleum products, biodiesel and biodiesel fuel blend, cloud point is the minimum temperature index which can be effectively used for special purposes. Too many wax crystals will block the filter of certain fuel system.

6. Sampling and preparation of samples

6.1 Unless otherwise specified, sampling shall be conducted according to GB/T 4756.

6.2 In normal temperature, samples shall be fluid, transparent and free from impurities.

6.3 If there is water in the samples, dehydration treatment can be conducted through any of the following methods. During the whole treatment process, relative humidity in the environment shall not be more than 75%, during the filtering, the sample's temperature shall be at least 14°C higher than the expected cloud point, but shall not exceed 49°C.

6.3.1 If the sample is not transparent in normal temperature, its temperature shall be adjusted to at least 14°C higher than the expected cloud point, and then filter the sample by using dry filter paper (7.2.2) until the sample is totally clear.

Note: The turbid or fog state of the wax crystals first appears at the bottom of the test tube with the lowest temperature. However, a slight fog state due to the existence of trace water in the oil will appear in the whole sample, and the state will gradually become more obvious with the temperature going down. Generally, this water fog will not interfere with the test of wax cloud point, and if interference exists, just use dry filter paper to filter the sample.

6.3.2 For diesel, if the fog state is heavy, pour 100mL of new sample in the test tube and add in 5g of anhydrous sodium sulfate (7.2.1), shake for at least 5 min, then let the test tube stay till the sample is clear, and filter the sample with dry filter paper (7.2.2). As long as the time is sufficient, this method can dehydrate the sample or minimize the water fog; it is convenient for the observer to properly observe the turbid phenomenon presented by the wax crystals.

Note: The mass of the anhydrous sodium sulfate can be increased or decreased according to the water content of the sample, and the optimum mass is for better observing the cloud point.

7. Manual method

7.1 Instrument

Qualitative or quantitative filter paper.

Note: It shall be dried in the drying oven of 100°C~105°C till constant weight, then be put in the dryer for later use. It is better to dry it when required.

7.2.3 Cooling agent

7.2.3.1 Ice and water.

7.2.3.2 Sodium chloride crystal, chemically pure.

7.2.3.3 Solid carbon dioxide (dry ice).

7.2.3.4 Acetone: chemically pure.

Warning: highly combustible.

7.2.3.5 Solvent oil: conforming to the requirements for No. 3 in GB 1922-2006.

Warning: combustible, steam is poisonous.

7.2.3.6 Absolute ethyl alcohol: chemically pure.

Warning: combustible.

7.2.3.7 Absolute methyl alcohol: chemically pure.

Warning: combustible, steam is poisonous.

7.3 Test procedures

7.3.1 Pour the sample (6.1) into the test tube till the scale mark.

7.3.2 Tightly block the test tube by using a cork stopper with a temperature gauge. If the expected cloud point is higher than -36°C, choose the GB-37 temperature gauge (7.1.1.2); if the expected cloud point is lower than -36°C, choose the GB-36 temperature gauge (7.1.1.2). Adjust the position of the cork stopper and the temperature gauge to make the cork stopper tightly block the test tube; make the temperature gauge and the test tube on the same axis; and make the mercury bulb just reach the bottom of the test tube.

Note: The mercury line of the temperature gauge may sometimes break, which can not be discovered instantly. It is recommended to regularly inspect the temperature gauge. When not being put in the bath, it shall indicate atmospheric temperature, and when put in the bath, it shall indicate 0°C ± 1°C. Or put it in a cooling bath with known temperature to check its usability.

7.3.3 Spacer, gasket and casing tube shall be clean and dry. Put the spacer at the bottom of the casing tube. Before putting in the test tube, the spacer and casing tube shall be firstly maintained in the cooling medium for at least 10 min. Cap the empty casing tube during the cooling. Place the gasket in the test tube, about 25 mm distance from the

8.4.3 Adjust the set of recirculation cooler to the proper temperature; cool the casing to the needed temperature (refer to Table 3).

8.5 Calibration and standardization

8.5.1 Ensure to calibrate, check and operate the instrument based on the instrument specification.

8.5.2 The tool which is used to calibrate the instrument is a kind of stimulator with known resistance. This can be conducted as per the calibration specification in the operation manual.

Note: The test probe stimulator can be obtained from the instrument supplier; the stimulator can help to verify the accuracy of the temperature measurement.

8.5.3 The samples with mutual identified cloud point, such as the samples verified by several labs, can be used in the inspection of the instrument performance.

Note 1: The instrument can be inspected through the certified reference material (CRM), the reference material (RM), comparison samples between laboratories and the proficiency testing samples.

Note 2: In terms of the new instrument or the instrument used once a year, it can be inspected with the samples mentioned in "Note 1", the inspected result of the cloud point shall be within the range $\pm 1^{\circ}\text{C}$ of the nominal value or median value.

8.6 Test procedures

8.6.1 Set the parameter of the instrument, such as the sensitivity, threshold value and the accuracy of temperature, as per the requirement of instrument specification.

Note 1: The general regulated accuracy of temperature in the laboratory is 1°C . If having higher requirement on the results, 0.1°C can also be selected.

Note 2: In terms of the samples of which the chroma is 0 or not less than 3.5, such as the bright stock produced under the refining lubricating oil technology (BS), naphthenic oil and the hydrogenated oil produced by secondary processing technology etc., if the testing result is not ideal during the cloud point test, it can be realized by adjusting the sensitivity and threshold value.

8.6.2 Inject the sample (6.1) into the test tube (8.1.3), until it reaches the scale.

8.6.3 Put the cork pad at the bottom of the casing; sleeve the cork washer in the test tube. If needed, finally adjust it as per the instrument specification. The cork washer shall be $25\text{ mm} \pm 3.0\text{ mm}$ away from the bottom of the test tube.

8.6.4 Put the test tube into the casing. Connect the temperature probe as per the instrument specification.

8.6.5 Start testing based on the instrument specification. The instrument will adjust the temperature of the casing automatically as shown in Table 3; start to monitor the cloud point of the sample with light. The instrument will monitor and indicate the temperature of the casing and sample during the testing process.

8.6.6 The instrument will continuously monitor and then adjust the temperature of the casing based on the Table 3. The time used to move from one casing to the next casing shall not exceed 200s; this time applies to the casing temperature declining to -52°C .

8.6.7 As for the casing with lower temperature, the time used to move from one casing to the next casing shall not exceed 300s. The temperature of the cooling system shall be as low as possible to achieve the temperature of these casings in the shortest time. The cooling capability of the cooling system can achieve the lowest temperature that the test needs.

8.6.8 The instrument shall be able to inspect the temperature of the turbidity appeared at the bottom of the test tube. The highest indicated temperature of the turbidity appeared at the bottom of the test tube is the cloud point of the sample; record it with accuracy to 0.1°C or 1°C .

8.6.9 The instrument shall be equipped with alarm which can remind the operator immediately. This function can also help the operator take out the sample from the instrument and observe whether the sample has turbidity, then further verify the cloud point result of the sample.

8.6.10 After the inspection, adjust the temperature of the casing to $23^{\circ}\text{C}\pm 2^{\circ}\text{C}$, then disassemble the test tube.

Note: Disassembling the test tube under the condition of low temperature may result in the damage to the temperature probe.

9. Result expression

9.1 Manually take the temperature recorded in 7.3.7 as the cloud point, accurate to 1°C .

9.2 Automatically take the temperature recorded in 8.6.8 as the cloud point, accurate to 0.1°C or 1°C .

Note: The accuracy requirement on the inspected result is related to the needs of customer or the technical specifications of the product.

10. Accuracy and deviation

10.1 Accuracy

Judge the reliability of the inspection result with the following specification (the confidence

level is 95%).

Note: The accuracy in this Standard is obtained through the statistical analysis of several laboratories' results. Manual method is obtained through the analysis of the participants on the distillate fuel and lubricating oil of 13 kinds of samples. The cloud point of these samples ranges from -1°C to -37°C ; the eight participant laboratories use the manual instruments. Automatically method is obtained through the analysis of the participants on the distillate fuel and lubricating oil of 11 kinds of samples; the cloud point of these samples ranges from $+34^{\circ}\text{C}$ to -56°C . The ten participant laboratories use the automatic instruments; the eight participant laboratories use the manual instruments.

10.1.1 Repeatability (r)

The deviation between the two results of the repeated tests which are conducted on the same sample, by one operator, with the same instrument, shall not exceed the repeatability value as shown in Table 4 and Table 5.

Table 4 Precision of Manual Method

Samples	Repeatability (r) / $^{\circ}\text{C}$	Reproducibility (R) / $^{\circ}\text{C}$
Distillate fuel and lubricating oil	2	4
Biodiesel and biodiesel blend fuels	2	3

Table 5 Precision of Automatic Method

Samples	Repeatability (r) / $^{\circ}\text{C}$	Reproducibility (R) / $^{\circ}\text{C}$
Distillate fuel and lubricating oil	2.2	3.9
Biodiesel and biodiesel blend fuels	1.2	2.7

10.1.2 Reproducibility (R)

The deviation between the two single and independent results of the tests which are conducted on the same sample, by different operators, with different instruments in different laboratories, shall not exceed the reproducibility value as shown in Table 4 and Table 5.

10.2 Deviation

10.2.1 Deviation of manual method

Because the result of cloud point is defined only by one method, therefore, the deviation of the cloud point in the manual method has not been determined.

10.2.2 Deviation of automatic method

Because there is no appropriate reference material that can be used to define the deviation, therefore, the deviation of the cloud point in the automatic method has not been determined, including the deviation of biodiesel.

10.3 Relative deviation

The relative deviation of the automatic method: It is the relative deviation between the test and the manual method of several laboratories. Although it has been recognized that there is effective deviation in statistics, however, the tested value is so small (-0.56°C) that have no actual meaning.

11. Report

The following contents shall be included in the testing report:

- a) Indicate the quotation of this Standard;
- b) The type and mark of the tested samples;
- c) Whether the samples have been filtered and dehydrated;
- d) Testing results;
- e) Whether the inspection on the testing results adopts the automatic method or the annual method;
- f) Any difference of the testing steps BETWEEN the provisions in agreement or other provisions AND this Standard shall be indicated clearly;
- g) Testing date.

_____ **END** _____

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