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Determination of solidification point for petroleum products

石油产品凝点测定法

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Table of Contents

Determination of solidification point for petroleum products

Warning - Personnel using this standard shall have practical experience in formal laboratory work. This standard does not point out all possible safety issues. The user is responsible for taking appropriate safety and health measures AND ensuring compliance with the conditions, which are stipulated by relevant national laws and regulations.

1 Scope

This standard specifies the method for determining the solidification point of petroleum products.

This standard applies to petroleum products, such as liquid fuels (such as diesel and biodiesel blended fuels) and lubricants. The automatic micro-solidification point tester, in this standard, is only applicable to the determination of diesel distillate samples without additives.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) is applicable to this standard.

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

GB/T 4756 Method for manual sampling of petroleum liquids

GB/T 6683 Petroleum products - Determination of precision data in relation to methods of test

3 Terms and definitions

The following terms and definitions apply to this document.

3.1

Solidification point

The highest temperature, at which the liquid level stops moving, when the sample is cooled, under specified conditions, expressed in °C.

4 Method summary

Contain the specimen in the specified test tube. Cool it to the expected temperature. Tilt the test tube to 45°, from the horizontal. Let it stand for 1 min. Observe whether the liquid level moves. The highest temperature, at which the liquid level does not move, is used as the solidification point of the specimen.

5 Reagents and materials

5.1 Reagents

5.1.1 Anhydrous ethanol: Chemically pure.

5.1.2 Anhydrous sodium sulfate: Chemically pure.

5.1.3 Anhydrous calcium chloride: Chemically pure.

5.2 Material

5.2.1 Coolant or material: Industrial ethanol, dry ice or liquid nitrogen, etc., OR any other liquid or material, which can cool the sample to the temperature, which is specified in the test. Compressor refrigeration or semiconductor refrigeration equipment can also be used.

5.2.2 Filter paper: Qualitative filter paper.

5.2.3 Table salt: Coarse table salt.

5.2.4 Absorbent cotton.

6 Instruments

6.1 Manual solidification point tester

6.1.1 Test tube: Transparent glass test tube, with round bottom; the height is 160 mm ± 10 mm; the inner diameter is 20 mm ± 1 mm; there is a circular marking on the outer wall, 30 mm from the bottom of the test tube; there is a plug, which can be inserted into the thermometer.

6.1.2 Casing: Glass casing, with round bottom; the height is 130 mm ± 10 mm; the inner diameter is 40 mm ± 2 mm; it is able to fix the test tube vertically, in the middle of the casing.

9 Test procedure

9.1 Manual instrument

9.1.1 When the temperature of the specimen reaches 50 °C ± 1 °C, take out the test tube, which contains the specimen and thermometer, from the water bath. Wipe dry the outer wall of the test tube. Put the test tube firmly into the casing. It shall ensure that the distance between the outer wall of the test tube and the inner wall of the case is equal.

9.1.2 Fix the casing and test tube assembly, vertically on the bracket. Let it stand, at room temperature, until the specimen is cooled to 35 °C ± 5 °C. Then immerse the assembly in the prepared cooling bath. The temperature of the cooling bath shall be 7 °C ~ 8 °C lower than the expected solidification point of the specimen. The depth of the casing and test tube assembly, which is immersed in the coolant, shall not be less than 70 mm.

9.1.3 When the temperature of the specimen is cooled to the expected solidification point, tilt the sleeve and test tube assembly, which is immersed in the cooling bath, to 45° from the horizontal. Keep it for 1 min. At this time, the specimen in it is still required to be immersed in the coolant.

9.1.4 Carefully remove the casing and test tube assembly, from the cooling bath. Use absolute ethanol to quickly wipe the outer wall of the sleeve. Place it vertically. Observe, through the casing, whether the liquid level in the test tube shows signs of movement.

Note: When measuring the specimen, which has a solidification point lower than 0 °C, in order to facilitate observation, it may inject 1 mL ~ 2 mL of absolute ethanol, into the bottom of the casing, before the test.

9.1.5 When the position of the specimen liquid level moves, take out the test tube from the casing. Reheat the test tube, until the specimen reaches 50 °C ± 1 °C. Then use a temperature, which is 4 °C lower than the previous test temperature OR other lower temperature, to make measurement again, until a certain test temperature can stop the liquid level of the specimen from moving.

Note: When the test temperature is lower than -20 °C, put the test tube, which contains the specimen and thermometer, at room temperature, before re-measurement. Wait for the temperature of the specimen to rise to -20 °C; then immerse the test tube in a water bath for heating.

9.1.6 When the position of the specimen liquid level does not move, take out the test tube from the casing. Reheat the test tube, until the specimen reaches 50 °C ± 1 °C. Then use a temperature, which is 4 °C higher than the previous test temperature OR other higher temperature, to make measurement again,

until a certain test temperature can cause the specimen liquid level to move.

9.1.7 After finding the temperature range of the solidification point (that is, the temperature range, in which the liquid level changes from moving to not moving OR not moving to moving), select a temperature, which is 2 °C lower than the temperature, at which the specimen can move, OR a temperature, which is 2 °C higher than the temperature, at which the specimen cannot move, to carry out test again. Repeat the test in this way, until it is determined that, a certain test temperature can stop the movement of the specimen liquid level, whilst increasing 2 °C can make the specimen liquid level move again. Take the temperature, at which the specimen liquid level does not move, as the solidification point of the specimen.

9.1.8 If it is necessary to check whether the solidification point of the specimen meets the specification value, the test shall be carried out, at 1 °C higher than the specification value. At this time, if the specimen liquid level can move, it is considered that, the solidification point of the specimen meets the specification value.

9.2 Automatic micrometer (only applicable to diesel distillate samples without additives)

9.2.1 Use manual sample injection or automatic sample injection, to take a certain amount of specimen. Select the appropriate test procedure, to start the test, according to the manual of the automatic micro-solidification point meter. A new sample shall be replaced, when repeating the test.

9.2.2 Record or print the final test result, according to the displayed result on the automatic micro solidification point tester, as the solidification point of the specimen.

Note: Before sample injection, use air to eject all the remaining specimen from the previous test. Then use the specimen to be tested, to clean the sample injection tube. Start sample injection. It shall make sure that the sample injection tube is completely filled with the specimen AND there are no bubbles (the presence of bubbles will affect the stability of the test results). It is not recommended to use any organic solvents to clean the sample injection tube.

10 Report of results

10.1 Take the arithmetic average of the two repeated test results, as the solidification point of the specimen.

10.2 The manual solidification point tester shall report the solidification point of the specimen, in accordance with the requirements of 9.1.7; the result shall be accurate to 1 °C.

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