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Replacing GB 510-77

Petroleum products – Determination of solidification point

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Petroleum Products –

Determination of solidification point

This Method applies to determination of solidification point of petroleum products.

The maximum temperature, at which lubricating oils and dark petroleum products cool down under test conditions so that the liquid surface stops moving, is called solidification point.

1 Method summary

In accordance with the determination method, a specimen is loaded into a specified test tube and cooled to the expected temperature, and then the test tube is tilted by 45 degrees for 1 min before it is observed whether the liquid surface moves.

2 Apparatus and materials

2.1 Apparatus

2.1.1 Round-bottom test tube: height 160 ± 10 mm; inner diameter 20 ± 1 mm; there is a annular mark on the outer wall, 30 mm from the tube bottom.

2.1.2 Round-bottom glass bushing: height 130 ± 10 mm; inner diameter 40 ± 2 mm.

2.1.3 Wide-mouth vacuum flask or cylindrical container holding coolant: height not less than 160 mm; inner diameter not less than 120 mm; it can be made of ceramics, glass, wood or sheet iron with insulation layer.

2.1.4 Mercurial thermometer: it meets the specifications of GB 514-75; it is used for petroleum products whose solidification point is lower than -35°C .

2.1.5 Liquid thermometer: it shall be as specified in GB 514-75, *Specification for liquid thermometers for testing of petroleum products*, and shall be used to determine the petroleum products having a solidification point higher than -35°C .

2.1.6 Thermometer of any type: it is used to measure the temperature of coolant.

2.1.7 Stand: it has a device to hold bushing, coolant container and thermometer.

2.1.8 Water bath.

mesh or a little cotton in a funnel and then spread newly calcined coarse salt crystal on the funnel. When specimens contain lots of water, they shall be filtered through the salt layers of 2 to 3 funnels.

4.3 Inject specimen into a dry, clean test tube, to make the liquid level to reach the annular mark. Use a cork to fix a thermometer in the middle of the test tube, making the mercury bulb 8 to 10 mm from the tube bottom.

4.4 The test tube containing specimen and thermometer is immersed vertically in a water bath $50 \pm 1^{\circ}\text{C}$, until the temperature of the specimen reaches $50 \pm 1^{\circ}\text{C}$.

5 Test procedure

5.1 Take out the test tube containing specimen and thermometer from the water bath, wipe the outer wall, use a cork to fix the test tube in a bushing and make sure the outer wall of the test tube and the inner wall of the bushing are equally distant at any spot.

The installed device shall be fixed vertically on a clamp of the stand, and allowed to stand at room temperature until the specimen in the test tube cools down to $35 \pm 5^{\circ}\text{C}$. And then immerse the set of devices into a container containing coolant. The temperature of coolant is 7 to 8°C lower than the expected solidification point of the specimen. The depth of the test tube (outer bushing) immersed in the coolant shall not be less than 70 mm.

When cooling the specimen, the temperature of coolant shall be accurate to $\pm 1^{\circ}\text{C}$. When the temperature of the specimen reaches the expected solidification point, tilt the device immersed in coolant to 45 degrees, and maintain this tilting state for 1 min, but the specimen part of the device shall still be immersed in coolant.

Then, take the device out carefully from coolant, use industrial alcohol to wipe the outer wall of the bushing rapidly, place the device vertically and observe through the bushing whether the liquid level in the test tube is movable.

NOTE When determining a solidification point lower than 0°C , 1 to 2 mL of absolute ethyl alcohol shall be injected to the bottom of the test tube before test.

5.2 When the liquid level is movable, take out the test tube from the bushing, preheat the specimen in the test tube to $50 \pm 1^{\circ}\text{C}$ once again, then carry out the determination at a temperature 4°C lower than the temperature of last test or other temperature even lower, until some test temperature makes the liquid level stop moving.

NOTE When the test temperature is lower than -20°C , place the test tube containing specimen and thermometer in room temperature before redetermination, and wait until the temperature of the specimen rises to -20°C before immersing the test tube in water bath for heating.

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