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**Standard test method for needle penetration of petroleum
waxes**

石油蜡针入度测定法

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Standard test method for needle penetration of petroleum waxes

WARNING -- Personnel using this document shall have practical experience in formal laboratory work. The use of this document may involve certain hazardous materials, equipment and operations, but it does not provide recommendations for all safety issues related to this. Before using this document, the user is responsible for formulating appropriate safety and protection measures and determining the applicability of relevant regulatory restrictions.

1 Scope

This document describes a method for evaluating wax hardness by measuring the depth of penetration of a standard needle into a wax specimen.

This document is applicable to petroleum wax with a needle penetration value not greater than 250 1/10 mm. It can also be used to determine Fischer-Tropsch wax, synthetic wax and bio-wax, etc. However, this document does not give the precision of determining synthetic wax and bio-wax, etc.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

GB/T 2539, *Determination of melting point of petroleum wax - Cooling curve method*

NB/SH/T 0843, *Evaluation of test systems for the petrochemical industry Statistical techniques*

SH/T 0132, *Determination method for petroleum wax congealing point*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1 penetration of petroleum wax

The depth to which a standard needle vertically penetrates into a wax specimen under specified conditions, expressed in 1/10 mm.

NOTE: For example, a reading of 85 on the penetrometer dial indicates a penetration value of 85 1/10 mm and a penetration depth of 8.5 mm.

3.2 penetrometer

An instrument (or device) for determining the consistency or hardness of a solid or semisolid sample by measuring the depth to which a needle or cone of a specified weight penetrates into the sample under a specified gravity.

4 Principle

Heat the specimen to at least 17°C above the expected freezing point or melting point, melt it, and pour it into the molding machine. Cool it in the air under specified conditions. Then control the temperature of the molded specimen at the test temperature in a water bath. Use the standard needle of the penetrometer to penetrate the sample for 5 s under a load of 100 g. Measure its penetration. The test temperature can be selected from any temperature in the range of 25°C~55°C. Commonly used are 25°C, 35°C, 40°C, 45°C or 55°C.

5 Instruments and reagents

5.1 Penetrometer

5.1.1 Penetrometer: The schematic diagram is shown in Figure 1. In this document, a standard needle is used to measure the hardness of wax specimens. The total load includes the standard needle, the slide bar and the 50 g weight, a total of 100 g.

5.1.2 Manual penetrometer: It is used to make the standard needle contact the specimen surface and measure the penetration depth of the standard needle into the specimen when the test is completed. When the indicator of the manual penetrometer reads "zero", the needle tip shall be accurately placed on the surface of the wax specimen through the adjustment device. One type of adjustment device is an adjustable test table. The other type is an adjustable needle assembly. Either one is selected. The instrument is equipped with a horizontal adjustment screw and a level to ensure that the slide shaft is in a vertical position. When releasing the loaded standard needle, the standard needle shall fall off without obvious resistance. The scale of the indicator shall be calibrated to 1/10 mm. The scale range shall be not less than 250 1/10 mm.

5.1.3 Automatic penetrometer: used to make the standard needle contact the surface of the specimen. The penetration time is controlled by an automatic timing device when

5.7 Thermometer

It is a precision thermometer for measuring water bath temperature. It is a full immersion type. The scale range is 25°C~55°C. It meets the requirements of GB-71 thermometer specified in GB/T 514-2005.

5.8 Brass plate

The specifications are 63.5 mm $\pm$ 1.6 mm in length, 38.0 mm $\pm$ 1.6 mm in width, and 6.4 mm $\pm$ 1.6 mm in thickness. It is used to support the sample when it is prepared for molding. The plate is placed on an insulating material such as a cork or rubber stopper during the cooling of the sample.

5.9 Reagent

Glycerol aqueous solution (50% by volume).

6 Specimen preparation

Heat the wax specimen to melt at least 17°C above its expected freezing point (determined by the method in SH/T0132) or melting point (determined by the method in GB/T2539). Prevent local overheating. Ensure that the wax specimen is uniform and free of bubbles. In a test room or test box (5.5) at 24.0°C $\pm$ 2.0°C, place the brass plate (5.8) on a stable support such as a rubber stopper or cork stopper. Then wet the upper surface of the brass plate with the reagent (5.9). Place the specimen former (5.4) on the brass plate. Then pour the molten wax specimen into the specimen former to form a convex meniscus. Cool it at 24.0°C $\pm$ 2.0°C for 1 h. Scrape off the excess wax specimen on the top of the specimen former. Remove the brass plate. Place the wax specimen surface in contact with the brass plate facing up. Place it on a porous specimen holder. Place in a water bath (5.6) at the test temperature $\pm$ 0.1 °C for 60 min. Very hard wax may sometimes separate from the specimen former due to shrinkage. In this case, it is permissible to use a wedge to plug the wax specimen in the former tightly.

7 Test steps

7.1 Manual penetrometer

7.1.1 Place the head of the manual penetrometer on the edge of the water bath (5.6) and over the porous specimen holder that supports the specimen. A weight may be placed on the base of the penetrometer to balance the head. Adjust the penetrometer and the porous specimen holder in the water bath to a level position.

7.1.2 Ensure that the specimen former and porous specimen holder do not shake during the test. Adjust the water bath surface to at least 25 mm above the upper surface of the specimen. Maintain the test temperature (within $\pm$ 0.1°C). Before measuring each

sample, measure the temperature of the water bath using the thermometer specified in 5.7.

7.1.3 Add a 50 g weight to the standard needle so that the total mass of the standard needle and its accessories is $100\text{ g} \pm 0.15\text{ g}$. Check whether the release device is stuck. The indicator shall be at the "zero" position. Adjust the needle assembly or the test bench according to the different types of adjustment devices. Adjust until the tip of the standard needle almost touches the surface of the specimen. The needle tip is located on a circle at least 3.2 mm from the edge of the specimen former and the four test positions are approximately equally spaced (separated by not less than 12.7 mm). Securely lock the movable components in this position.

7.1.4 Fine-tune the standard needle tip to make it just touch the surface of the wax specimen. Observing the projection of the standard needle tip will help to make accurate adjustments. After confirming that the water bath temperature is within the specified range, release the slide bar and keep it for $5\text{ s} \pm 0.1\text{ s}$. Use an automatic timing device or a stopwatch with an accuracy of 0.1 s to time. Gently press down the indicator rod until it is supported by the slide bar. Read the needle penetration value from the indicator dial.

7.1.5 Carry out the measurement of the remaining three test positions according to the requirements of 7.1.3 and 7.1.4. Before each measurement, wipe carefully along the needle tip with a clean dry cloth to remove all attached wax samples.

7.2 Automatic penetrometer

7.2.1 Place the adjustable needle assembly of the automatic penetrometer above the water bath (5.6) and above the porous specimen holder supporting the specimen. Proceed as in 7.1.2.

7.2.2 Set up the penetrometer according to the instrument manual and test the penetration value according to the requirements in 7.1.3 ~ 7.1.5.

8 Results report

Record the measured values of the four test positions of the specimen. Calculate the average value as the test result. Round to an integer. Also report the test temperature, e.g., $85\frac{1}{10}\text{ mm}$ (35°C).

9 Quality control

9.1 Use uniform, stable, sufficient and representative relevant samples as quality control samples. The reliability of the analytical test process is determined by analyzing the quality control samples.

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