Study on Calibration Technology and Uncertainty Evaluation of Asphalt Vacuum Decompression Capillary Viscometer

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Abstract. 60 °C dynamic viscosity is one of the main indexes to evaluate the quality of asphalt products, and its influence on the quality of asphalt pavement is very important. 60 °C dynamic viscosity is usually measured by the asphalt vacuum decompression capillary viscometer (hereinafter referred to as the viscometer). The quality of the viscometer directly affects the test results of the asphalt dynamic viscosity. Therefore, it is urgent to study the detection method of viscometer to ensure the quality of the viscometer and the accuracy of the test results. In this paper, the calibration test method of viscometer is established, and the uncertainty evaluation is carried out, which provides a technical basis for ensuring the accuracy of viscometer.

1. Introduction
Asphalt pavement plays an extremely important role in our country's highway, both in use and quantity. 60 °C dynamic viscosity [1] is one of the main indexes of asphalt properties. The United States and Australia have used this index as the classification standard of road petroleum asphalt. According to JTG F40-2004, on the basis of properly improving the softening point index of A-grade asphalt, 60 °C dynamic viscosity has been added as the evaluation index of asphalt high temperature performance and has clear technical requirements and qualification standards [2]. After more than 10 years of use, it is shown that 60 °C dynamic viscosity is very important for the quality control of asphalt products [3], and has become one of the most widely used technical indexes in the evaluation of asphalt.

60 °C dynamic viscosity is measured by viscometer [4]. The quality of viscometer directly affects the test results of asphalt dynamic viscosity. At present, there are many problems with viscometer, such as many manufacturers, uneven quality, and the accuracy of viscometer calibration coefficient cannot be verified. Therefore, it is urgent to study the detection method of viscometer to ensure the quality of the viscometer and the accuracy of the test results, and provide technical support for the quality control of asphalt and asphalt pavement. In this paper, the calibration test method of viscometer is established, and the uncertainty evaluation [5-7] is carried out, which provides a technical basis for ensuring the accuracy of viscometer.

2. Calibration test method of viscometer

2.1. Introduction of viscometer
Viscometer [8] is used to determine dynamic viscosity of viscous petroleum asphalt, which is composed of vacuum decompression capillary viscometer and vacuum decompression system. Vacuum
decompression system is composed of constant temperature and temperature measuring device, vacuum decompression device, timing device and controller. The structure of viscometer is shown in Figure 1.

![Figure 1. The structure of viscometer](image)

1--Constant temperature and temperature measuring device, 2--Vacuum decompression capillary viscometer, 3--Vacuum decompression device, 4--Controller, 5--Timing device.

2.2. Design of calibration test scheme for viscometer

The calibration test scheme designed in this paper is as follows: first, calibrate [9] the viscometer coefficient. Then control the viscometer at a certain temperature and vacuum, and measure the time of the standard viscosity liquid flows through a certain height between the timing marks of the vacuum decompression capillary viscometer, the measured dynamic viscosity value can be converted by multiplying this time by the calibrated viscometer coefficient. By comparing the measured dynamic viscosity value with the standard one, the dynamic viscosity indication error of the viscometer can be calculated, so as to judge whether the measured value of the viscometer is accurate.

The spacing of timing marks is an important parameter to ensure the accuracy of the equipment. In this test, it is specified that the spacing of timing marks shall be 20 mm±0.6 mm. During the test, the temperature of the loading area of each vacuum decompression capillary viscometer shall be 60 ℃±0.1 ℃ and the vacuum degree shall be 40 kPa±0.07 kPa. The indication error of timing device shall not exceed ±0.1 s. When the dynamic viscosity range is not more than 1000 Pa·s, the indication error of dynamic viscosity shall not exceed ±15%. When the dynamic viscosity range is 1000 Pa·s or above, the indication error of dynamic viscosity shall not exceed ±20%. The American canon standard viscosity liquid is used in the test.

2.3. Calibration test process of viscometer

2.3.1. Spacing of timing marks. Use a standard caliper to measure the spacing of each timing mark three times. Compare the measured value of the standard caliper with the spacing of timing marks of the viscometer, so as to judge whether the spacing of the timing marks of the viscometer meets the test requirements.

2.3.2. Temperature of constant temperature and temperature measuring device. Start the viscometer and set the water temperature to 60 ℃, after the temperature is stable, insert the thermometer into the constant temperature and temperature measuring device, so that the thermometer is at the loading area
of the vacuum decompression capillary viscometer. Read the temperature every 2 minutes after the data is stable, read a total of 9 data. Compare the value of the thermometer with the set value, it can judge whether the temperature of the loading area of each vacuum decompression capillary viscometer meets the test requirements.

2.3.3. Vacuum degree of vacuum system. Connect the precision pressure gauge to the vacuum device, read the pressure data every 3 minutes after the data is stable, read 3 data in total. Compare the value of the precision pressure gauge with the vacuum degree setting value, so as to judge whether the vacuum degree of the vacuum system meets the test requirements.

2.3.4. Timing device. Fix the fixture of the time calibration device on the time trigger button of the viscometer, and adjust the distance between the impact head of the fixture and the time trigger button, so that the equipment can work normally. Set a random time greater than 60 s on the time calibration device, start the viscometer and the time calibration device at the same time, read the time after the time calibration device is triggered. Compared the measured value of the time calibration device with the indicating value of the timing device of the viscometer, it can judge whether the timing device of the viscometer meets the test requirements.

2.3.5. Indication error of dynamic viscosity. According to the method specified in JTG E20, the dynamic viscosity value of standard viscosity liquid is measured by viscometer. By comparing the measured dynamic viscosity value with the standard dynamic viscosity value, the dynamic viscosity indication error of the viscometer can be calculated, so as to judge whether the measured value of the viscometer is accurate.

3. Uncertainty evaluation

3.1. Uncertainty of distance measurement results

3.1.1. Establishment of measurement model

\[ \Delta = l - l_0 \]  

In style:
\( \Delta \) — Indication error of the spacing of timing marks, mm.
\( l \) — Measured value of the spacing of timing marks, mm.
\( l_0 \) — Standard value of the spacing of timing marks, mm.

3.1.2. Evaluation of uncertainty components.

(1) Uncertainty introduced by repeatability [10] \( u_1 \)

Repeat the measurement for 3 times and the obtained data are: 20.20 mm, 20.10 mm and 20.12 mm. The difference between the maximum value and the minimum value is 0.1 mm. The uncertainty is calculated by the range method (3 times of measurement, the range coefficient is 1.69):

\[ u_1 = \frac{0.1 \text{ mm}}{1.69 \times \sqrt{3}} = 0.03 \text{ mm} \]  

(2) Uncertainty introduced by the standard digital calipers \( u_2 \)

According to the certificate of the standard digital calipers, \( U=0.02 \text{ mm} \), \( k=2 \), the uncertainty introduced by the standard digital caliper is:

\[ u_2 = \frac{U}{k} = \frac{0.02 \text{ mm}}{2} = 0.01 \text{ mm} \]
3.1.3. Composite standard uncertainty \( u_c \). The calculation results of the composite standard uncertainty are as follows:

\[
u_c = \sqrt{u_1^2 + u_2^2} = \sqrt{0.03^2 + 0.01^2} = 0.03 \text{ mm} \quad (4)
\]

3.1.4. Relative standard uncertainty \( u_{r1} \)

\[
u_{r1} = 0.03 \text{ mm} / 20.14 \text{ mm} \times 100\% = 0.15\% \quad (5)
\]

3.2. Uncertainty of temperature measurement results

3.2.1. Establishment of measurement model

\[
\Delta = T - T_0 \quad (6)
\]

In style:

\( \Delta \) — Indication error of temperature, °C.
\( T \) — Measured value of temperature, °C.
\( T_0 \) — Standard value of temperature, °C.

3.2.2. Evaluation of uncertainty components. (1) Uncertainty introduced by repeatability \( u_1 \)

Repeat the measurement for 3 times and the obtained data are: 59.92 °C, 60.08 °C and 60.10 °C. The difference between the maximum value and the minimum value is 0.18 °C. The uncertainty is calculated by the range method (3 times of measurement, the range coefficient is 1.69):

\[
u_1 = \frac{0.18^\circ C}{1.69 \times \sqrt{3}} = 0.06^\circ C \quad (7)
\]

(2) Uncertainty introduced by the standard thermometer \( u_2 \)

According to the certificate of the standard thermometer, \( U=0.08 \ ^\circ C, k=2 \), the uncertainty introduced by the standard thermometer is:

\[
u_2 = \frac{U}{k} = \frac{0.08^\circ C}{2} = 0.04^\circ C \quad (8)
\]

3.2.3. Composite standard uncertainty \( u_c \). The calculation results of the composite standard uncertainty are as follows:

\[
u_c = \sqrt{u_1^2 + u_2^2} = \sqrt{0.06^2 + 0.04^2} = 0.07^\circ C \quad (9)
\]

3.2.4. Relative standard uncertainty \( u_{r2} \). At 60 °C, the test results show that, the relative standard uncertainty of the influence of temperature on the dynamic viscosity of standard viscosity fluid is as follows:

\[
u_{r2} = 0.33\% \quad (10)
\]

3.3. Uncertainty of pressure measurement results

3.3.1. Establishment of measurement model

\[
\Delta = P - P_0 \quad (11)
\]

In style:

\( \Delta \) — Indication error of pressure, kPa.
\( P \) — Measured value of pressure, kPa.
\( P_0 \) — Standard value of pressure, kPa.
3.3.2. Evaluation of uncertainty components. (1) Uncertainty introduced by repeatability $u_1$

Repeat the measurement for 3 times and the obtained data are: 40.18 kPa, 40.07 kPa and 40.12 kPa. The difference between the maximum value and the minimum value is 0.11 kPa. The uncertainty is calculated by the range method (3 times of measurement, the range coefficient is 1.69):

$$u_1 = \frac{0.11 \text{kPa}}{1.69 \times \sqrt{3}} = 0.04 \text{kPa}$$  \hspace{1cm} (12)

(2) Uncertainty introduced by the standard digital pressure gauge $u_2$

According to the certificate of the standard digital manometer, MPE: ±0.03 kPa, assuming uniform distribution, $k=\sqrt{3}$, the uncertainty introduced by the standard digital manometer is:

$$u_2 = \frac{0.03 \text{kPa}}{\sqrt{3}} = 0.02 \text{kPa}$$  \hspace{1cm} (13)

3.3.3. Composite standard uncertainty $u_c$. The calculation results of the composite standard uncertainty are as follows:

$$u_c = \sqrt{u_1^2 + u_2^2} = \sqrt{0.04^2 + 0.02^2} = 0.04 \text{kPa}$$  \hspace{1cm} (14)

3.3.4. Relative standard uncertainty $u_r$

$$u_r = \frac{0.04 \text{kPa}}{40 \text{kPa}} \times 100\% = 0.1\%$$  \hspace{1cm} (15)

3.4. Uncertainty of time measurement results

3.4.1. Establishment of measurement model

$$\Delta = t - t_0$$  \hspace{1cm} (16)

In style:

$\Delta$—Indication error of the time calibration device, s.
$t$—Measured value of the time calibration device, s.
$t_0$—Standard value of the time calibration device, s.

3.4.2. Evaluation of uncertainty components. (1) Uncertainty introduced by repeatability $u_1$

Repeat the measurement for 3 times and the obtained data are: 90.01 s, 89.92 s and 90.03 s. The difference between the maximum value and the minimum value is 0.02 s. The uncertainty is calculated by the range method (3 times of measurement, the range coefficient is 1.69):

$$u_1 = \frac{0.11 \text{s}}{1.69 \times \sqrt{3}} = 0.04 \text{s}$$  \hspace{1cm} (17)

(2) Uncertainty introduced by the standard time calibration device $u_2$

According to the certificate of the standard time calibration device, $U=0.01 \text{s}$, $k=2$, the uncertainty introduced by the standard time calibration device is:

$$u_2 = \frac{U}{k} = \frac{0.01 \text{s}}{2} = 0.01 \text{s}$$  \hspace{1cm} (18)

3.4.3. Composite standard uncertainty $u_c$. The calculation results of the composite standard uncertainty are as follows:

$$u_c = \sqrt{u_1^2 + u_2^2} = \sqrt{0.04^2 + 0.01^2} = 0.04 \text{s}$$  \hspace{1cm} (19)

3.4.4. Relative standard uncertainty $u_r$
\[ u_{r4} = 0.04 \text{ s} / 89.99 \text{ s} \times 100\% = 0.04\% \]  

(20)

3.5. Uncertainty of dynamic viscosity measurement results

3.5.1. Establishment of measurement model

\[ \Delta = x - x_0 \]  

(21)

In style:

\( \Delta \) — Indication error of dynamic viscosity, Pa·s.

\( x \) — Measured value of dynamic viscosity, Pa·s.

\( x_0 \) — Standard value of dynamic viscosity, Pa·s.

3.5.2. Evaluation of uncertainty components

(1) Uncertainty introduced by repeatability \( u_1 \)

Repeat the measurement for 10 times and the obtained data are: 100.8904, 101.3155, 101.7406, 100.7487, 102.3074, 101.8823, 102.3074, 99.4734, 100.7487, 101.4572. The uncertainty is calculated:

\[ u_1 = s(\bar{X}) = \frac{s(X)}{\sqrt{n}} = 0.9\% \]  

(22)

(2) Uncertainty introduced by the standard viscosity liquid \( u_2 \)

According to the certificate of the standard viscosity liquid, the maximum uncertainty of the standard viscosity liquid is 2%, then \( u_2 = 2\% \).

3.5.3. Composite standard uncertainty \( u_c \). The calculation results of the composite standard uncertainty are as follows:

\[ u_c = \sqrt{u_{r1}^2 + u_{r2}^2 + u_{r3}^2 + u_{r4}^2 + u_1^2 + u_2^2} = 2.23\% \]  

(23)

3.5.4. Composite extended uncertainty \( U \). Take \( k = 2 \),

\[ U = ku_c = 2 \times 2.23\% = 4.46\% \approx 4.5\% \]  

(24)

The expanded uncertainty of dynamic viscosity measurement results is: \( U = 4.5\% \), \( k = 2 \).

4. Conclusion

In this paper, the parameters of viscometer are studied and determined. The spacing of timing marks shall be 20 mm±0.6 mm. The temperature of the loading area of each vacuum decompression capillary viscometer shall be 60 °C±0.1 °C. The vacuum degree shall be 40 kPa±0.07 kPa. The indication error of timing device shall not exceed ±0.1 s. When the dynamic viscosity range is not more than 1000 Pa·s, the indication error of dynamic viscosity shall not exceed ±15% and when the dynamic viscosity range is 1000 Pa·s or above, the indication error of dynamic viscosity shall not exceed ±20%.

According to the requirements of the above parameters, the calibration test method of viscometer is established and the uncertainty evaluation is carried out, which ensures the quality of the viscometer and the accuracy of the test results, and provides technical support for the quality control of asphalt and asphalt pavement. The research results of this paper lay a theoretical and experimental foundation for ensuring the accuracy of viscometer.

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