VISCOSITY TESTING

EN 12596: Bitumen and bituminous binders – Determination of dynamic viscosity by vacuum capillary

Overview

This test method is used to determine the dynamic viscosity of bitumen by means of a vacuum capillary viscometer at 60 °C.

In the test, a fixed volume of bituminous binder is forced by constant vacuum to flow through a capillary tube at a fixed temperature.

The method is not applicable to bituminous emulsions, but can be used for binders recovered and/or stabilized from emulsions.

Experience suggests that the method is not applicable for polymer modified bitumens (PMB); other viscosity test methods, e.g. EN 13302 or EN 13702, are more suitable.

Definition and Terminology

Dynamic viscosity: Measure of the resistance to flow of a liquid, expressed in Pa · s. It is the constant ratio of applied shear stress to the velocity gradient of the liquid.

Kinematic viscosity: Measure of a liquid's resistance to flow under gravity, expressed in mm^2/s.

Newtonian liquid: Liquid with a viscosity that is independent of the rate of shear. If the ratio of applied shear stress to the velocity gradient (rate of shear) of the liquid is not constant, the liquid is non-Newtonian.

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At the time of publication of this document, EN 12596:2023 `Bitumen and bituminous binders - Determination of dynamic viscosity by vacuum capillary` was the reference for testing. This document does not overrule the test standard EN 12596, but is intended to help users of the standard to be aware of the important factors. However, the reference for testing remains EN 12596. Temperatures, times, and dimensions and their tolerances must be strictly observed, that is checked for accuracy and for maintaining the tolerance during application.

Practical Information:

All parts of the capillary viscometer system must be checked and/or calibrated annually.

- A detailed description how to calibrate viscometers is given in Annex B of EN 12596.
- Capillary viscometers shall preferably be calibrated at the intended operational temperature.
- The timing device must be calibrated and accurate to 0,5 s.
- The controller of the vacuum system must be capable of maintaining a vacuum of 40.000 Pa with a maximum permissible error of ± 100 Pa and must be checked regularly.
- The testing bath temperature must be (60 ± 0,3) °C, and should be checked regularly.

The test temperature must be selected carefully.

- Usually, the test is carried out at 60 °C.
- Other temperatures are possible, if calibration constants are known.
- The precision for this test method is based on determinations made at 60°C.

The sample needs to be prepared for testing.

- The sample preparation is described in EN 12594 and section 6 of EN 12596.
- The viscometer tube must be clean and dry before filling.
- Carefully pour the sample into the viscometer tube, avoiding overfilling. Overfilling may lead to covering of tube markings, so measurements become unprecise.
- Air bubbles in the sample lead to incorrect viscosity results. Bubbles can be removed by e.g. placing the charged viscometer in an oven or bath maintained at (135,0 ± 5,0) °C for a period of 10 min, see section 7.4 of EN 12596.
- The viscometer has to be suspended in a vertical position in the bath.
- The sample needs to be conditioned in the tube at the test temperature for at least 1 hour.

Testing and expression of results.

- EN 12596 does not specify the bath liquid to be used. Mineral or silicone oils are widely used, but also water can be used.
- Ensure that the temperature within the bath does not vary by more than 0,3 °C over the length of the viscometer tube. This can be done by making temperature checks at different levels of the bath liquid.
- Select the correct viscometer tube, as described in section 7.2 of EN 12596. The flow time must be greater than 60 s and below 1.000 s.
- The vacuum must not be interrupted during testing. If the vacuum is interrupted, the results must be discarded.
- Measure the time taken for the sample to flow between the timing marks.
- The dynamic viscosity is calculated by multiplying the flow time in seconds by the viscometer calibration factor (VCF), and is expressed in Pa · s.
- From experience, tubes can easiest be cleaned by hanging them upside down in an oven to let the bitumen flow out. After the bitumen has flowed out, the tubes should be cooled and rinsed several times with an appropriate solvent, followed by a completely volatile solvent. Dry the tube by passing filtered dry air through the capillary until all traces of solvent are removed. For details, see section 7.9 of EN 12596.









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