

Testing of Paraffin

Determination of Needle Penetration

DIN
51 579

Prüfung von Paraffin; Bestimmung der Nadelpenetration

This Standard has been prepared in conformity with Method D 1321-61 T of the American Society for Testing and Materials (ASTM), Philadelphia, Pa., USA, obtainable from the DIN Deutsches Institut für Normung e.V. (German Institute for Standardization).

Dimensions in mm

1 Range of application

Paraffins (hydrocarbon waxes from mineral oil, coal, shale) with a needle penetration of up to 250 tenths of a millimetre

Note: Unlike ASTM D 1321-61 T, it is usual in Germany to examine specimens with needle penetrations of more than 150 tenths of a millimetre according to DIN 51 580 "Testing of paraffin, determination of cone penetration", and not by this Standard.

2 Purpose of determination

Assessment and characterization of the serviceability of paraffins for specific purposes

3 Definitions

Penetration is understood to be the entry of a test piece into the material to be tested.

Needle penetration, as an empirical measure of the hardness of paraffins, is understood to be the depth in tenths of a millimetre through which a test needle passes into the specimen in the conditions of this Standard.

4 Unit of measurement

Tenths of a millimetre (0.1 mm)

5 Brief description of the method

The specimen is heated to about 15 deg. above its congealing point on a rotating thermometer, poured into a test cylinder, cooled under prescribed conditions to 25 °C and conditioned in a water bath to the test temperature — usually 25 °C. The depth of penetration of a loaded and temperature-controlled test needle (total weight 100 g) is then determined with a penetrometer under water at the test temperature, which is maintained within ± 0.1 deg., for a test period of 5 seconds.

6 Sampling

According to DIN 51 594

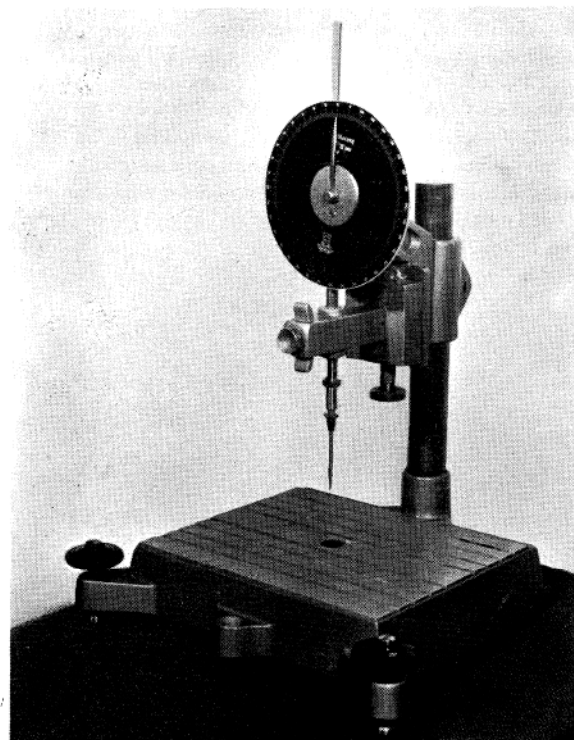


Figure 1. Penetrometer with test needle

7 Apparatus**7.1 Penetrometer**

The penetrometer (see Fig. 1) consists of a table to take the test cylinder filled with the specimen, a vertical column with a vertical mounting, adjustable for height, for the needle guide rod and the indicating device. Any penetrometer is suitable which allows the needle guide rod to slide in the guide without perceptible friction and the depth of penetration reached to be read off in tenths of a millimetre.

The penetrometer should be fitted with levelling screws and a spirit level; it should have a fine adjustment allow-

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ing the test needle point to be placed exactly on the surface of the specimen, the indicating device being set to the zero mark. The indicating device covers a range of 0 to at least 250 tenths of a millimetre.

It is also permissible to use an automatically operating device fitted to the penetrometer which, when the catch is released, simultaneously sets the needle guide rod and a stopwatch in motion and stops both again after the prescribed period of 5 seconds $\pm$ 0.1 second.

7.2 Test needle

The test needle according to Fig. 2 should be made of round stainless steel. After the taper has been made, the point is ground off at right angles to the axis to form a truncated cone. The finally shaped needle is then hardened and very highly polished. The Rockwell hardness HRC, according to DIN 50 103, should be 57 to 60 and the concentricity deviation of the cone when the shaft is secured in a prism, should not exceed a maximum of 0.013 mm, measured at the base and at the tip of the cone.

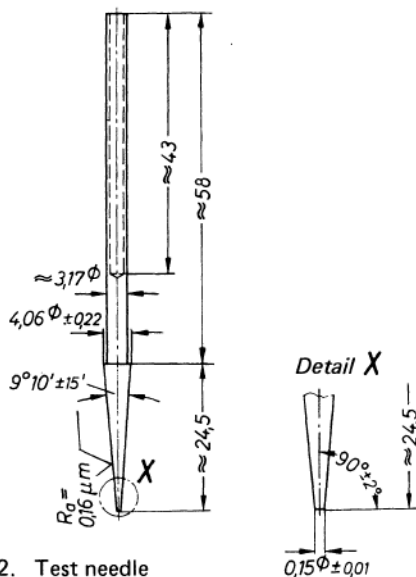


Figure 2. Test needle

The weight of the test needle should be $2.5 \text{ g} \pm 0.05 \text{ g}$ and that of the guide rod $97.5 \text{ g} \pm 0.1 \text{ g}$.

Note: Guide rods which are also intended for determining the cone penetration according to DIN 51 580 and which have a weight of $47.5 \text{ g} \pm 0.05 \text{ g}$ receive an additional attachable weight of $50 \text{ g} \pm 0.05 \text{ g}$.

It is recommended that only test needles be used which have been tested by the Physikalisch-Technische Bundesanstalt (Federal Physical-Technical Institute), Berlin or Braunschweig. Only needles so tested may be used for arbitration analyses.

7.3 Test cylinder

The test cylinder of brass according to Fig. 3 is open at both ends. Some helical grooves are cut in the inner surface of the cylinder wall to prevent the specimen from slipping out. The radial base surface of the test cylinder must be ground flat.

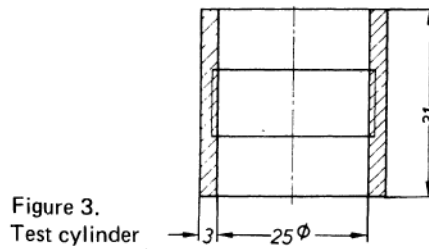


Figure 3.
Test cylinder

7.4 Base plate for test cylinder

As the base plate a flat glass plate $65 \text{ mm} \times 40 \text{ mm} \times$ about 6 mm or a brass plate $65 \text{ mm} \times 40 \text{ mm} \times 6 \text{ mm}$ is used. Some flat pieces of cork are used with the base plate as explained in Section 8.1.

7.5 Thermostat

Water bath of at least 10 l capacity, for temperatures between 20 and 55 °C, adjustable to within ± 0.1 deg. The container for the water bath either consists entirely of glass or, if made of sheet metal, has a glass window through which the specimen can be viewed horizontally from the side. Belonging to the water bath is a rigid perforated stand on which the test cylinder rests some 50 mm below the surface of the water while the determination is carried out. The depth of the water bath is to be such that, during conditioning (see Section 8.2), the test cylinder is immersed to a depth of at least 100 mm into the water when standing 50 mm above the bottom of the water bath on a perforated base.

Should the thermostat be placed on the penetrometer table, smaller water baths may be used, provided that they permit adequate circulation of the water above and below the specimen and maintenance of the prescribed test temperature.

7.6 Stopwatch

Graduated in 1/10 s

7.7 Thermometer

Graduated in 0.1 deg., adjusted for full immersion. If the thermometer is used without total immersion, a stem correction must be applied.

8 Preparation for the determination

8.1 Filling the test cylinder

To prevent the solidifying specimen from sticking to the base plate, the latter is smeared with a mixture of equal parts by volume of glycerine and water or with a mixture of silicone oil (of a dynamic viscosity of about 300 cP at 20 °C) and light benzene (2 g to 100 ml). The test cylinder can be placed at once, if necessary after the benzene has evaporated, on to the base plate treated in this way. Base plate and test cylinder are then taken into a draught-free room having a constant temperature of $25 \text{ °C} \pm 2$ deg. Some flat pieces of cork are placed under the base plate.

The specimen is heated on the rotating thermometer (see DIN 51 556) to about 15 deg. above its solidification point, care being taken to avoid local overheating. If it has still not melted to a clear condition, the heating must be continued until the melt is clear. After any

stirring required to eliminate bubbles, the melt is poured into the test cylinder in the constant-temperature room until a convex (raised) meniscus is formed.

After one hour, any convexity (dome) of the specimen projecting beyond the upper rim of the test cylinder is cut off with a warmed knife. On the other hand, any crater which has formed in the surface of the specimen is left as it is and is not filled up.

8.2 Conditioning of the filled test cylinder

The test cylinder is detached from the base plate and placed in the thermostat, 50 mm above the bottom of the water bath, for one hour, with the smooth reflecting bottom surface uppermost.

The temperature of the water bath should be the same as the subsequent test temperature. It is usually $25^{\circ}\text{C} \pm 0.1^{\circ}\text{deg.}$, but may also be between 20°C and 55°C . These temperatures, too, must be kept constant within $\pm 0.1^{\circ}\text{deg.}$, any emergent thermometer stem corrections being taken into account.

9 Procedure of determination

Since the penetration is highly temperature-dependent, it must be determined in the thermostat at constant temperature. For this purpose, either

- the thermostat is placed alongside the penetrometer and, if necessary, the penetrometer table loaded with a counterweight, or
- the thermostat is placed on the penetrometer table, or
- the penetrometer is placed in the thermostat.

The water level in the thermostat is to be adjusted to 25 mm above the upper rim of the test cylinder.

Immediately prior to the determination, the test temperature is to be checked and also the test needle brought to the test temperature. The test cylinder must stand firmly and may not be displaced during measurement. The test needle is first lowered coarsely until its point almost touches the surface of the specimen. By means of the fine adjustment, the test needle is further lowered until it just touches the surface (check by observing the shadow of the illuminated point on the reflecting surface of the specimen!). Finally, the rack of the indicating device is carefully placed on the needle guide rod and the zero mark on the scale disc is aligned with the pointer.

After 5 minutes, the arresting knob is quickly released, whereby the needle guide rod may not be impeded, and the needle guide rod is again secured after $5 \text{ seconds} \pm 0.1 \text{ second}$. The rack of the indicating device is again carefully pressed down until it meets with slight resistance and the depth of penetration of the test needle is read off from the scale.

4 measurements are made on each specimen at points lying at least 3.5 mm from the inside wall of the test cylinder and at least 12 mm apart from each other.

After each measurement, the point of the test needle is carefully wiped with a clean, soft, dry cloth. In doing so, the test needle is raised to its highest position to avoid damage to the guide. The test needle may not be rotated because this would in time cause wear of the release mechanism.

10 Statement of the results

The needle penetration is stated, with reference to this Standard, as the mean value of 4 measurements, rounded off to the nearest tenth of a millimetre according to DIN 1333, stating the test temperature. In this Standard, the mean value is regarded and treated as a "single measured value".

11 Test errors (according to DIN 51 849) ¹⁾

Needle penetration	Scatter ranges ²⁾			
	Repeatable scatter range (one observer, one apparatus)		Reproducible scatter range (different observers, different apparatus)	
	absolute	relative % of numerical value of the lower result	absolute	relative % of numerical value of the lower result
0.1 mm	0.1 mm		0.1 mm	
up to 30	± 2	—	± 4	—
over 30	—	± 8	—	± 25

Note: If finer differences in the needle penetration than are obtainable with the range of test errors of this Standard are required, e.g., for paraffins and waxes with a needle penetration not exceeding 3 tenths of a millimetre, attention is drawn to DGF-Einheitsmethode (DGF Uniform Method) M-III 9a (57) "Bestimmung der Härte von Wachsen, Kugeldruckmethode" (Determination of the hardness of waxes, ball indentation method) (obtainable from Wissenschaftliche Verlags-GmbH, Stuttgart; see also G. v. Rosenberg, Messung der Härte von Wachsen. Fette, Seifen, Anstrichmittel (Measurement of the hardness of waxes. Greases, soaps, paints) 56, (1954), pp. 214 et seq.).

¹⁾ The absolute values (without plus and without minus signs) stated for the test error are almost the same as the numerical values given for the test errors quoted in ASTM D 1321-61 T for "repeatability" and "reproducibility". The above numerical values (without plus and without minus signs) can consequently also be interpreted as the maximum difference between two values measured individually which can be expected with a statistical certainty $P = 95\%$. The difference in the definition, as compared with DIN 51 849, is to be strictly observed in practice.

²⁾ See DIN 51 849, March 1960x issue, section 1.26 examples Ia and Ib

Explanations

When paraffins and waxes are poured into and cooled in the test cylinder, craters occur on the surface of the specimen which, in the case of specimens with a needle penetration of more than 150 tenths of a millimetre, can result in the needle entering the hollow space of the crater during the determination. The Note in Section 1 "Range of application" therefore takes account of the actual circumstances which, for the reasons given, have normally been found to arise.

As with all substances, the structural formation and hardness of poured and subsequently cooled paraffins and waxes are largely dependant on the melting temperature and the rate of cooling, and for this reason alone the values obtained according to DIN 51 579 are only conventional and not absolute values. With natural and synthetic waxes or compounds and preparations, the dependance on melting temperature and rate of cooling is even more marked than in the case of homogeneous paraffins. The repeatability and reproducibility of the test results are to a large extent dependant on strict adherence to the provisions of this Standard.