

## PNR 12: Consistency of Vaselinum Album and Flavum – Hollow Cone Method (European Pharmacopoeia 2.9.9.)

Relevant for: Pharmaceutical Industry, Cosmetics Industry

The penetrometer PNR 12 is suitable for consistency measurements required in the pharmaceutical industry. Various test kits are available for different pharmaceutical products and standards like the official WHO method European Pharmacopoeia 2.9.9.



### 1 Introduction

This application report gives an example on how to measure vaselinum with Anton Paar's penetrometer PNR 12.

The PNR 12 measures the resistance a material provides against being pierced by a specifically shaped test body. Therefore, a needle-shaped or conical test body is precisely lowered to the surface of the material under test. It will sink into the sample by its own weight for a fixed period of time. This allows a rating of the plasticity or consistency.

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#### 1.1 European Pharmacopoeia 2.9.9

The penetration test according to the European Pharmacopoeia 2.9.9 is an official method, especially

for pharmacy products like vaselinum. A WHO (World Health Organization) document with the detailed description of the whole procedure can be found under: <http://apps.who.int/phint/en/p/docf/> (go to "Supplementary information > Test methods used during development or manufacture > Measurement of consistency by penetrometry").

#### 1.2 Definition

The Penetration Unit [PU] is the consistency of a material expressed as distance in tenth of a millimeter that a specifically shaped test body vertically penetrates a sample during a fixed period of time under constant temperature conditions.

### 2 Instrument

To measure the consistency of vaselinum products, Anton Paar's PNR 12 was used.



Figure 1: PNR 12 with hollow cone

### 3 Accessories

#### Test kit according to European Pharmacopoeia 2.9.9.:

Test Kit, Standard Hollow Cone, ASTM D937, Petrolatum

Consisting of:

- 1 optional hollow cone, 102.5 g brass with steel tip
- 1 plunger, 47.5 g
- 1 sample container, Ø 100 mm × 65 mm



Figure 2: Standard hollow cone test kit

- 2 Petrolatum sample containers, Ø 100 mm × 65 mm

#### Optional:

Pharma Qualification Package “Smart” Document

Consisting of:

- Standard Operating Procedure
- Qualification Instruction
- Design Qualification
- Installation Qualification
- Operation Qualification
- Performance Qualification
- Final Qualification

### 4 Sample

Paraffin:

- Vaselinum Album (white, soft)
- Vaselinum Flavum (yellow, soft)

### 5 Sample and Instrument Preparation

**This Application Report does not replace reading the original standard.**

#### 5.1 Sample Preparation and Filling in Accordance with European Pharmacopoeia 2.9.9.

- Fill the sample bubble-free into three sample containers.
- Create a flat surface.
- The sample should be worked as little as possible during the filling.
- Store the sample at 25 °C ±0.5 °C in a heating cabinet for 24 h, unless otherwise prescribed.

#### 5.2 Instrument Preparation

- The instrument must be located on a level, vibration-free surface.
- Use the adjustable feet to align the device, check with the leveling bulb.
- Hollow cone and plunger must be clean and dry.
- Bring the temperature of the cone to 25 °C ±0.5 °C.

#### 5.3 Settings

- Penetration time: 5 s
- Temperature: 25 °C
- Total weight of test body assembly: 150 g

Parameter	Fixed program 4
Program name	<b>Grease</b>
Operator	Operator 01
Penetration time [1/10 s]	50
Automatic surface detection	No detection
Force sensitivity	4
Automatic temperature control	No sensor, manual
Min. start temperature	0
Max. start temperature	0
Unit	1/10 mm
Limit low	0
Limit high	9999
Wait before	1
Speed down [mm/s]	27
Distance down [mm]	180
Wait on surface [s]	2
Speed up [mm/s]	5
Distance up [mm]	20
Wait after [s]	2
Specials	none

Table 1: Settings for vaselinum

## 6 Measurement

- Select the program “**Grease**”.
- Place the sample container on the penetrometer table.
- Insert the plunger with the hollow cone.
- Press <START>.
- Reset the plunger by gently lifting as far as it goes while pressing the <RELEASE> button.

**Attention: Hold the plunger always by hand before pressing the <RELEASE> button.**

- Press <START>. The slide will move to the basic position. After the waiting time the slide starts to move downwards automatically. With the jog wheel, the speed can be increased (turn clockwise) or decreased (turn counterclockwise).
- Press the jog wheel before the sample surface is reached to stop the movement.
- Place the light source next to the hollow cone so that the shadow or reflection of the tip of the hollow cone can be seen on the surface of the sample.
- Turn the jog wheel clockwise to move the lift down or counterclockwise to move the lift up until the tip of the hollow cone just reaches the

surface. You found the correct position if the tip contacts its shadow or image reflected on the sample surface.

- Press <START>. This releases the plunger with the hollow cone for 5 s.
- After the penetration time the slide will move upwards 20 mm and remains there for 2 s. During this waiting time the sample can drop back into the sample container.
- After this the slide will move back into basic position.
- The result is shown on the display.
- Repeat the measurement with the two remaining samples.

## 7 Cleaning

Wipe the hollow cone with cleaning solvent e.g. ethanol and a tissue between each penetration.

## 8 Results

The result is the average of three penetration readings reported to the nearest whole unit. The test has to be repeated if the individual results differ more than 3 % from the mean value. In this case express the result as the mean and relative standard deviation of the six results.

Sample	1. Test 0.1 mm	2. Test 0.1 mm	3. Test 0.1 mm	Average
Vaselineum Album	244	247	242	<b>244</b>
Vaselineum Album	241	242	239	<b>241</b>
Vaselineum Flavum	226	223	226	<b>225</b>
Vaselineum Flavum	219	218	221	<b>219</b>

Table 2: Example results for vaselinum

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