PNR 12: Consistency of Ointment and Cream

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 creams with Anton Paar's penetrometer PNR 12.

Due to its easy handling and convertibility the PNR 12 has gained a wide scope of applications. It is suitable for products like creams manufactured by the cosmetics or the pharmaceutical industry.

The European Pharmacopoeia 2.9.9. is an official method especially for pharmacy products like ointment. 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").

1.1 Advantages of a Micro Cone Kit Compared to a Hollow Cone Kit

- Small sample volume - Micro cup ≈4 mL (petrolatum cup ≈275 mL); ideally suited for samples of limited availability
- Easy air bubble-free filling is possible
- Shorter tempering time due to the small volume
- Easy cleaning / low solvent consumption therefore environmentally friendly
- Better reproducibility and repeatability
- Wide consistency range
- Most pharmacy producers prefer the micro cone method for ointment and cream

1.2 Benefit

The consistency is not only important for the product packing but even more for the application and handling.

For example the spreading behavior:

- The more liquid a cream (lotion) the faster it will be absorbed which is for example useful for sun protection products.
- A more solid product would stay longer on the skin surface which might be desirable for ointment that should cover an open wound.

1.3 Definition

The Penetration Unit [PU] is the consistency of a material expressed as distance in tenth of a millimeter.

2   Instrument

To measure the consistency of ointment and cream products, Anont Paar's PNR 12 was used.

Figure 1: Penetrometer PNR 12

3   Accessories

Various test kits are available for different standards or manual and automatic surface detection. The choice of the accessories depends mainly on the consistency. Cream is a very soft sample therefore we a light plunger and test cylinder is needed.

In case of electrical conductive samples the PNR 12 offers an automatic surface detection.

Example Accessories:

- Plunger, 10 g, 107 mm length, aluminum
- Test cylinder, Ø 10 mm, 15 g, stainless steel
- Sample container, Ø 55 mm × 70 mm

Figure 2: Test cylinder (front), plunger (middle) and sample container (back)

or

- Pharma test kit, micro cone, acc. to Klein (incl. micro-cone KLEIN, 7 g, and plunger 16.8 g)

Figure 3: Pharma test kit, Micro Cone, acc. to Klein

Optional:

- Temperature sensor for PNR 12
- Circulator bath set CD-B27
- Transfer dish

4   Sample and Instrument Preparation

A correct preparation of a measurement is important for gaining the best result. How to prepare samples and instrument is explained below.

4.1   Sample Preparation and Filling

It is highly recommended to create an own preparation procedure if no standard is available.

It is important that the sample and instrument preparation is always the same.

Here an example:

- Pour the sample bubble-free into three sample containers.
- Create a flat surface.
- The sample should be worked as little as possible during the filling.
- Temper the sample to 25 °C for 2 h.

4.2   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.

- The test cylinder and the plunger must be clean and free from water.
- Install the electric surface detector for conductive samples.
- Small sample containers have to be centered against the test cylinder.
- In big sample containers several tests can be conducted. Please consider the required distance between the measuring points and the measuring point and the wall of the sample container to be >10 mm.

4.3 Calibration

Calibration of distance, time and Pt100 is possible.

For detailed information please refer to the manual.

5 Example Settings for a Test Kit as Shown in Figure 2

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

Other settings are possible but it is very important to have the same settings for all comparable samples.

For example the penetration time could be shortened if the penetration value is still too high although the overall weight of the penetration assembly is as low as possible. In this case you can also lower the temperature.

Parameter	Fixed program 3	Fixed program 4
Program name	**Cond Sample**	**Grease**
Operator	Operator 01	Operator 01
Penetration time	50	50
Automatic surface detection	Electric sensor	No detection
Force sensitivity	**4**	**4**
Automatic temperature control	No sensor, off	No sensor, manual
Min. start temperature	0	0
Max. start temperature	0	0
Unit	1/10 mm	1/10 mm
Limit low	0	0
Limit high	9999	9999
Wait before	1	1
Speed down	**12 mm/s**	**27 mm/s**
Distance down	180 mm	180 mm
Wait on surface	2 s	2 s
Speed up	5 mm/s	5 mm/s
Distance up	20 mm	20 mm
Wait after	2 s	2 s
Specials	C-Value (80 g)	NLGI greases

Table 1: Example settings for creams

6 Measurement

There are two possibilities for the surface detection when testing the consistency of creams with the PNR 12. The manual and the automatic surface detection are explained below.

6.1 With Manual Surface Detection

- Select the program "**Grease**".
- Place the sample container on the penetrometer table.
- Insert the plunger with the test cylinder.
- 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.
- Position the light source next to the test cylinder so that the shadow or reflection of the tip of the test cylinder can be seen on the surface of the sample.
- Turn the jog wheel clockwise to move the slide down or counterclockwise to move the slide up until the tip of the test cylinder just reaches the surface. You found the correct position if the tip touches its shadow or image reflected on the sample surface.
- Press <START>.
  This releases the plunger with the test cylinder 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 this procedure at least three times with different samples.
- Use a clean test cylinder for each determination.

6.2 With Automatic Surface Detection

Please keep in mind that the automatic surface detection by the electronic sensor is only possible with conductive samples.

In this case, the sample container has to be isolated against the PNR 12 table with a suitable material, e.g. a plastic foil.

- Select the program "**Cond Sample**".
- Place the sample container on the penetrometer table.
- Insert the hook of the electric surface detector in the sample or assemble it outside on the metallic sample container.
- Insert the plunger and test cylinder.
- 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 until the surface is reached.

- After penetration time slide will move upwards 10 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 this at least three times with fresh samples.
- Use a clean perforated disk for each determination.

7 Cleaning

Wipe the test cylinder with a cleaning solvent e.g. ethanol and a tissue between each penetration.

8 Results

The result is the average of three penetration results reported to the nearest whole unit.

Sample	1. Test result in 0.1 mm	2. Test result in 0.1 mm	3. Test result in 0.1 mm	Average in 0.1 mm
1	110	113	114	112
2	113	115	111	113
3	111	110	112	111

Table 2: Example results of a cream

The automatic detection by electronic sensor is only possible with conductive samples.

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