Powders

INTRODUCTION

The widespread use of powders in the pharmaceutical industry has led to a proliferation of test methods for measuring **powder flow** and **density**.

The harmonised chapters in the Pharmacopoeias on Powder Flow (USP Chapter <1174> and Ph.Eur. Chapter 2.9.36) list four well-defined methods for powder testing, aimed at trying to bring about some degree of standardisation within the test methodology:

- Flow through an orifice
- Angle of Repose
- Shear Cell
- Compressibility Index and Hausner Ratio

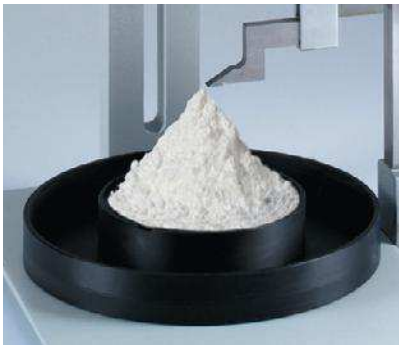
The Flowability Tester BEP2 from Copley Scientific provides a range of options for testing pharmaceutical powders including three of the four methods quoted in the Pharmacopoeias – flow through an orifice, angle of repose and shear cell – in a single, cost effective unit.

The BEP2 is an easy to use, small footprint instrument with interchangeable cylinder, funnel, angle of repose and shear cell attachments.

In addition to providing the test methods detailed in USP <1174> and Ph.Eur. 2.9.36, the unit can also be used to carry out tests described in the separate European Pharmacopoeia Chapter on Flowability 2.9.16.

An optional balance/timer simplifies time vs mass testing.

For compressibility index and Hausner ratio testing, the fourth specified methodology, Copley Scientific offers a series of tapped density testers and the bulk density tester (Scott Volumeter), detailed monographs for which feature in USP Chaper <616> and Ph.Eur. Chapter 2.9.15.



Copley Philosophy			
Robust	✓	Reliable	✓
Easy to use	✓	Compliant	✓

BEP2 with Cylinder Attachment

FLOWABILITY TESTER MODEL BEP2

INTRODUCTION

The Flowability Tester BEP2 has been specifically designed to address the specifications in and comments raised by the **European Pharmacopoeia Chapter 2.9.36** and **US Pharmacopeia Chapter <1174>** on **Powder Flow.**

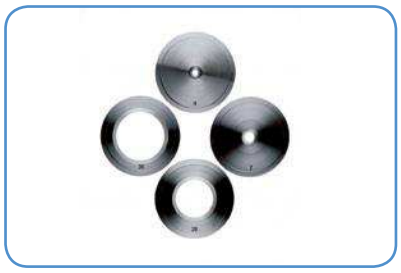
The widespread use of powders in the pharmaceutical industry has led to a proliferation of test methods for measuring powder flow.

The harmonised chapters in the Pharmacopoeias on Powder Flow (USP Chapter <1174> and Ph.Eur. Chapter 2.9.36) list four well-defined methods for powder testing aimed at trying to bring about some degree of standardisation within the existing test methodology:

- Flow through an orifice
- Angle of Repose
- Shear Cell
- Compressibility Index and Hausner Ratio

The Flowability Tester BEP2 from Copley Scientific provides a range of options for testing pharmaceutical powders including three of the four methods quoted in the Pharmacopoeias – flow through an orifice, angle of repose and shear cell – in a single, cost effective unit. In addition to providing the test methods detailed in the harmonised pharmacopoeia chapters, it is also suitable for flowability testing according to Ph.Eur. 2.9.16. An optional balance/timer simplifies time vs mass testing.

The BEP2 is an easy to use, small footprint instrument with interchangeable cylinder, funnel, angle-of-repose and shear cell attachments. A description of each attachment can be found below.



Interchangeable Disks

CYLINDER ATTACHMENT (FLOW THROUGH AN ORIFICE)

Measuring the ability and the time taken for a powder to flow through an orifice of known size is a useful method of quantifying powder flow.

At the same time, it is important to recognise that the ability of the powder to flow through the orifice can be affected by factors other than the characteristics of the powder itself.

Such factors include the shape and material employed in the construction of the powder container, the diameter and height of the powder bed and the shape of the orifice concerned.

The Pharmacopoeias suggest that the use of a circular cylinder as the powder container encourages powder over powder flow - as opposed to powder over container wall - minimising any effect brought about by differences in the material used to produce the powder container.

As the title suggests, this technique is only suitable for materials that flow, **not** cohesive materials. Assuming this to be the case, then the Pharmacopoeias suggest that providing:

a) The height of the powder bed (the "head") is much greater than that of the orifice
b) The diameter of the opening is greater than six times the diameter of the particles and
c) The diameter of the cylinder is greater than two times the diameter of the opening

then any difference in results brought about by either powder bed or orifice can be considered negligible.

The Cylinder Attachment has been designed to take all of these factors into account.

The **Cylinder Attachment** comprises a stainless steel cylinder measuring 76 mm long x 57 mm i.d. and having a capacity of 200 mL. The bottom of the cylinder is sealed with a collar designed to accept disks having various orifice diameters.

The attachment comes complete with a set of 20 interchangeable stainless steel disks each containing a precision drilled hole in the centre covering the following sizes: 4, 5, 6, 7, 8, 9, 10, 12, 14, 16, 18, 20, 22, 24, 26, 28, 30, 32, 34 and 36 mm. A shutter covers the hole during filling. This can be smoothly removed without vibration to allow the powder to flow through the selected hole.

The Cylinder Attachment can be used in two ways: (a) to carry out **quantitative flowability tests** based on mass vs time or (b) to determine the **intrinsic flowability** of the powder concerned in the form of a flowability index based on comparative measurements.

a) Mass vs Time

Operation is extremely simple.

Select a disk having the appropriate orifice size for the powder concerned (start with 18 mm and work up or down accordingly if size unknown) and secure it to the bottom of the cylinder using the collar provided for this purpose. Adjust the shutter so that the hole in the bottom of the cylinder nozzle is covered.

Introduce the test sample (100 grams unless inappropriate) into the flow cylinder. Now open the shutter and measure the time required for the entire sample to flow out of the funnel using a suitable stopwatch.

Carry out three measurements: express the flow rate results in terms of mass vs time i.e. grams per second.

BEP2 with Cylinder and Balance/Timer Attachments

b) Intrinsic Flowability

The Cylinder Attachment provides a simple and repeatable technique for the determination of powder flow characteristics.

As such it takes into account most of the physical characteristics affecting flowability, such as particle size, shape, fines, unit surface, actual and bulk density, porosity, settling, and electrostatic charge, without a direct quantitative measurement of any of these parameters.

The determination of Intrinsic Flowability is based upon the ability of a powder to fall freely through a hole in a plate. The results are expressed in terms of a Flowability Index given as the diameter (in mm) of the smallest hole the powder falls through freely on three successive attempts.

For new formulations, it is recommended to start with the 18 mm disk. Once the disk is in position and with the shutter in the closed position, a 50 gram sample is introduced into the test cylinder using the funnel provided for this purpose.

After waiting for approx. 30 seconds to allow for any possible formulation of flocculi, the shutter is opened. The test is positive if the powder flows through the hole leaving a residue in the form of an upside-down truncated cone. A powder that flocculates in bulk will, on the other hand, fall abruptly forming a cylindrical cavity. In this instance, as is the case if the powder refuses to flow through the hole, the test is adjudged to be negative.

In the case of a positive result, the test must be repeated with smaller and smaller disk holes until the result is negative. For negative results, increase the size of the disc hole until the test is positive.

The Flowability Index has been used successfully to establish dry powder characteristics prior to setting up filling equipment such as capsule fillers, tablet presses and dry packaging machines, thus avoiding high coefficients of variation.

It can also be used in purchasing specifications to ensure consistent flow characteristics of materials received as well as general quality control procedures.

Anti-static Grounding Kit for BEP2

BEP2 with Funnel and Balance/Timer Attachments

BEP2 with Funnel Attachment

BEP2 with Funnel and Angle of Repose Attachments

FLOWABILITY TESTER MODEL BEP2

FUNNEL ATTACHMENT (FLOW THROUGH AN ORIFICE)

In certain instances where, for example, the purpose of the test is to simulate flow in a hopper or other production situation, it may be preferable to use a funnel in the form of a truncated cone.

The **Funnel Attachment** is based on the stainless steel flow funnel and nozzle described in the European Pharmacopoeia Chapter 2.9.16 for Flowability. It has a capacity of approx. 400 mL.

The attachment is supplied with three nozzles corresponding to aperture sizes of 10, 15 and 25 mm respectively. Both funnel and nozzles are manufactured from pharmaceutical grade 316 stainless steel. The nozzles can be quickly interchanged using the connecting nut provided for that purpose.

The opening at the bottom of the funnel is secured by means of an adjustable shutter, which is closed during the filling operation. The test is carried out in a similar manner to that of Method A (Mass vs Time) of the cylinder attachment (see page 61).

BALANCE / TIMER ATTACHMENT

By adding a balance and a timer linked to a microswitch located on the shutter mechanism, it is now possible to conduct time vs mass tests using either cylinder or funnel methods without the need for an external stopwatch.

The balance/timer option allows the use of the unit in four modes:

- Determination of the flow time of a predetermined sample weight
- Determination of the flow time of a predetermined sample volume
- Determination of the weight of sample in a predetermined time
- Plot of time against sample weight (weight/time)

Manually Operated Stirrer

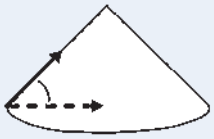
ANGLE OF REPOSE ATTACHMENT

The Angle of Repose is the angle (relative to the horizontal base) of the conical pile produced when a granular material is poured onto a horizontal surface. It is related to the density, surface area and coefficient of friction of the material concerned.

The **Angle of Repose Attachment** comprises a 100 mm diameter circular test platform together with a digital height gauge, having a range of 0-300 mm and an accuracy of 0.03 mm. The test platform has a protruding outer lip in order to retain a layer of powder upon which the cone is formed. Surplus powder is collected in a tray below the test platform.

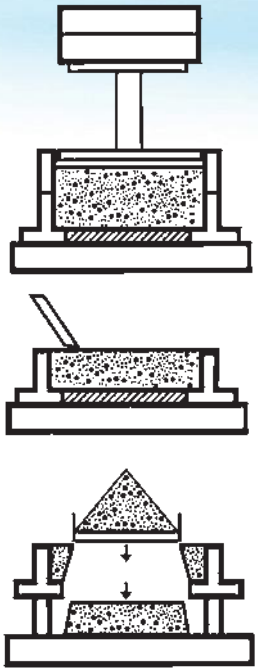
For this particular test, the funnel is normally equipped with a special 10 mm i.d. nozzle mounted 75 mm above the test platform. If necessary, the contents may be stirred to assist in the powder flow (see left).

The tangent of the angle of repose (in degrees) can be determined by reading off the height of the powder cone in mm from the digital display of the height gauge and dividing it by 50. The Table on Page 63 indicates the flow properties associated with corresponding Angles of Repose.



BEP2 with Funnel and Shear Cell Attachments

Shear Cell



FLOWABILITY TESTER MODEL BEP2

SHEAR CELL ATTACHMENT

Shear cell methodology is widely used in the pharmaceutical industry to determine the flow properties of fine grained powders and bulk solids and how they will behave in bins, hoppers, feeders and other handling equipment.

The ability of a material to flow through such devices is dependent on the bulk density of the material and its shear strength.

The **Shear Cell** employed with the BEP2 comprises a cylindrical chamber (manufactured from clear acrylic) measuring 140 mm i.d. and 32.5 mm high and capable of holding 500 mL of the sample. In the floor of the chamber, there is a 100 mm hole, which can be sealed during the consolidation process using an acrylic disk provided for this purpose.

The test is based on measuring the force required to shear a circular disk through a prepared sample of bulk material. It comprises two stages: (a) sample consolidation (bulk density measurement) and (b) failure inducement (shear strength).

The sample is first subjected to a consolidated load such that the bulk density of the material can be determined – ideally, this should be similar to the loads experienced by the material in practice. Alternatively, a standard reference can be employed e.g. 10 kg.

The acrylic disc sealing the bottom of the test cell is now removed and load steadily applied to the test sample by pouring sand through a funnel into a container of appropriate proportions resting on top of the sample until such time as the sample fails (shears).

The results should be expressed in terms of bulk density, shear strength and if appropriate, estimate of device outlet required.

Flow Properties & Angle of Repose

Flow Property	Angle of Repose
Excellent	25 - 30
Good	31 - 35
Fair - aid not needed	36 - 40
Passable - may hang up	41 - 45
Poor - must agitate, vibrate	46 - 55
Very poor	56 - 65
Very, very poor	> 66

Cat. No.	Description
1650	Flowability Tester Model BEP2 Stand and Upright
1651	Cylinder Attachment (Flow through an Orifice)
1652	Funnel Attachment (Flow through an Orifice)
1656	Manually operated Stirrer for Funnel Attachment
1653	Balance/Timer Attachment
1654	Angle of Repose Attachment*
1655	Shear Cell Attachment*
1657	Anti-static Grounding Kit for BEP2
1658	IQ/OQ Documentation Pack
1659	Qualification Tools

* Requires the Funnel Attachment (Cat.No.1652) to operate

BULK DENSITY TESTERS

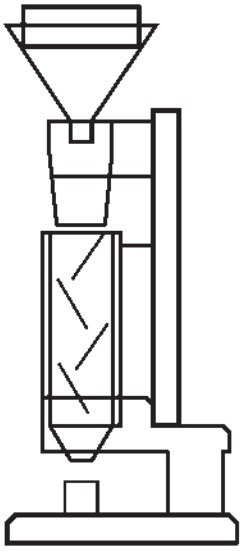
The bulk density of powders can be extremely difficult to measure since the slightest disturbance may result in a change in the results.

This is the result of the relationship between the particles that constitute the powder bulk. This same relationship affects the ability of the powder to flow.

The **bulk density** of a powder may be described as the density of the powder "as poured" into a measuring vessel.

Tapped density, on the other hand, is the density attained after "tamping down". This is normally measured using an instrument that lifts and then drops a measuring cylinder containing the powder through a fixed distance (see the Tapped Density Tester described on Page 65).

A comparison of the bulk and tapped densities of powders can give an indication of the type of interaction present between the various particles making up the powder mass and hence provide an index of powder flowability (see ***Compressibility Index*** and ***Hausner Ratio*** described on the next page).



Schematic of Scott Volumeter

THE SCOTT VOLUMETER

The **Bulk Density Tester (Scott Volumeter)** is described in **USP Chapter <616> Method 2** and **European Pharmacopoeia Chapter 2.9.34** and is designed for measuring the **bulk density of fine powders** and similar products.

CONSTRUCTION

The apparatus comprises:

- A stainless steel top funnel having an integral 18-mesh stainless steel screen
- A baffle box containing four glass baffle plates over which the powder slides and bounces as it passes
- A stainless steel bottom funnel to direct the powder into the receiving cup
- A cylindrical receiving cup having a capacity of 25 +/- 0.05 mL
- A stand to support the apparatus and its constituent parts.

An alternative funnel having an integral 10-mesh screen is available on request.

18-mesh and 10-mesh screen filter inserts

Scott Volumeter

MODE OF OPERATION

1) Weigh the empty receiving cup and place it in position
2) Slowly pour the powder through the upper funnel until it overflows the receiving cup. (Note: Use a minimum of 35 cm^3)
3) Level the top of the receiving cup with a spatula such that it is completely full being careful not to compress or shake the powder
4) Re-weigh the receiving cup and its contents
5) Calculate the bulk density in terms of grams per mL by dividing the weight of the powder by the volume of the cup

Cat. No.	Description
6301	Scott Volumeter with 18-mesh screen (USP <616> Method 2)
6302	Alternative filter insert with 10-mesh screen
6303	Volume Certification of the Receiving Cup
6305	Spare Receiving Cup
6306	Spare Set of Glassware (4 x Baffles + 1 Front and Rear Plate)

TAPPED DENSITY TESTERS

The Tapped Density Testers Series JV has been designed to measure the tapped density of powders, granules and similar products in accordance with **Methods 1 and 2** of **USP Chapter <616>** and **European Pharmacopoeia Chapter 2.9.34**

This technique is particularly useful in powder flowability studies and also in determining the amount of settlement during transit in order to optimise pack sizes e.g. washing powders.

Tapped density is achieved by mechanically tapping a measuring cylinder (i.e. raising the cylinder and allowing it to drop the specified distance of 3 +/- 0.2 mm under its own weight) containing the sample under test.

Two versions of the tester (JV 1000 and JV 2000) are available dependent on the number of test stations required (one or two). Both versions utilise 250 mL measuring cylinders as standard; however, 100 mL cylinders (and smaller) together with appropriate platforms are also available if required.

Both of the instruments concerned are equipped with membrane keypads for setting the number of strokes or time and an LCD screen to set the appropriate parameters and monitor the progress of the test.

MODE OF OPERATION

The mode of operation is identical on both models.

Weigh out a predetermined amount of the sample, say 100 g +/- 0.1%, place it in the graduated cylinder provided and note the unsettled volume. Secure the graduated cylinder to the test platform of the tester using the bayonet fitting provided for this purpose.

Unless otherwise specified, set the number of taps via the membrane keypad on the front of the instrument to 500 and operate the device making a note of the resulting tapped volume.

JV 1000 with 1 x 250 mL Measuring Cylinder

JV 2000 with 1 x 100 mL and 1 x 250 mL Measuring Cylinder

Repeat this operation for a further 750 taps noting the volume once again. Continue repeating the test in increments of 1250 taps until the difference in tapped volume is less than 2%. Note the final reading.

The tapped density in grams per mL can now be calculated by dividing the sample weight by the final tapped volume.

Measures of the ability of the powder to flow and its compressibility can now be given in the form of the Hausner ratio (Tapped Density/Bulk Density) and the Compressibility Index ((Tapped Density - Bulk Density/Tapped Density) x 100).

In a free flowing powder, inter-particulate interaction is less significant and unsettled and tapped densities will be closer in value. In poorly flowing powders, the inverse is to be expected. It follows that the closer the Hausner ratio is to 1, the better the flow. Powders with poor flow generally have a ratio of greater than 1.25.

A special **acoustic cabinet** capable of reducing the noise level of the volumeter from about 80 db to 58 db is available on request. The tapped density testers measure 280 x 250 x 670 mm (w x d x h).

Scale of Flowability

Compressibility Index (%)	Flow Character	Hausner Ratio
< 10	Excellent	1.00 - 1.11
11-15	Good	1.12 - 1.18
16-20	Fair	1.19 - 1.25
21-25	Passable	1.26 - 1.34
26-31	Poor	1.35 - 1.45
32-37	Very poor	1.46 - 1.59
> 38	Very, very poor	> 1.60

Cat. No.	Description
1601	Tapped Density Tester JV 1000 (1 x 250 mL Cylinder)
1602	Tapped Density Tester JV 2000 (2 x 250 mL Cylinders)
1603	IQ/OQ/PQ Documentation Pack
1616	Qualification Tools
1604	250 mL Measuring Cylinder (spare)
1605	100 mL Measuring Cylinder (option)
1605A	Special Platform for use with 100 mL Cylinder (option)
1606	Acoustic Cabinet