# Powders

#### **Background: Powders**

A pharmaceutical powder is a dry, bulk solid composed of many very fine particles normally classified dependent on particle size between coarse and very fine (see USP Chapter <811> Powder Fineness).

Whilst powders themselves are not widely employed as a dosage form in their own right, they are often used in the preparation of other forms such as tablets, capsules and inhaled products and are frequently added to other ingredients to make semisolids such as creams, ointments and pastes.

It is this widespread use of powders in the pharmaceutical industry that has led to a proliferation of test methods for measuring powder flow and density.

USP <1174> and Ph. Eur. 2.9.36 list four well-defined methods for powder testing proposed to bring some degree of test methodology standardisation:

- · Flow through an orifice
- · Angle of repose
- · Shear cell
- · Compressibility index and Hausner ratio

# Powders: Flowability

Powder flow depends on several factors, some of which relate to the powder material and some to the actual manufacturing processes, its ability, for example, to flow from a container (hopper, funnel, cylinder, etc.) or its compressibility in forming a tablet. The Pharmacopoeias recommend three methods for testing powder flow:

### 1. 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 powders. As the name suggests, this technique is only applicable to free-flowing powders, not cohesive materials. Methods vary but can be classified on the basis of three basic experimental variables:

- (a) Type of container employed (hopper, funnel, cylinder, etc.)
- (b) Size and shape of the orifice employed
- (c) Method of measuring powder flow rate

### 2. Angle of Repose

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 and indicative of flow in accordance with the values shown in the table overleaf.



# Powders: Flowability

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	

#### 3. Shear Cell

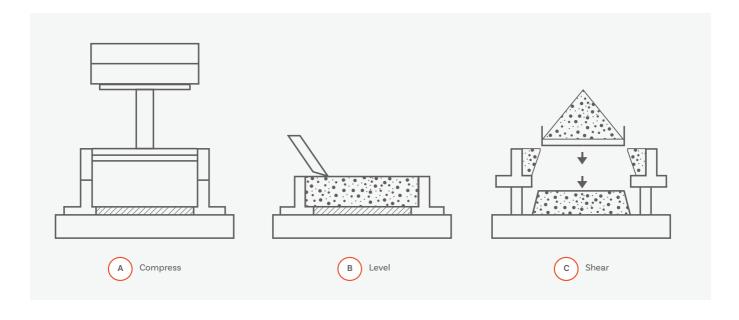
Measures the force required to shear a circular disk through a prepared sample of bulk material.

It comprises 2 stages:

- (a) Sample consolidation (bulk density measurement)
- (b) Failure inducement (shear strength)

Shear Cell methodology 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 powder to flow through such devices is dependent on the bulk density of the material concerned and its shear strength.



# Powders: Bulk and Tapped Density

The bulk density of a powder is expressed in terms of grams per mL by dividing the weight of a given "untapped" powder sample by its volume.

It is important to ensure that no settlement occurs during preparation such that the density of the powder concerned is "as poured" and therefore includes the contribution made by the volume of the interparticulate void.

Tapped Density, on the other hand, is the density attained after "tamping down". This is normally measured using an instrument that lifts and the drops a measuring cylinder or similar vessel containing the powder through a fixed distance.

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

### **Compressibility Index and Hausner Ratio**

Measures of the ability of the powder to flow and its compressibility can now be given in the form of (a) the Hausner Ratio or (b) the Compressibility Index" (see below).

#### a. Hausner Ratio

= Tapped Density/Bulk Density

### b. Compressibility Index

Tapped Density - Bulk Density = \_\_\_\_\_ x 100 Tapped Density

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.

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
3 - 37	Very poor	1.46 - 1.59
>38	Very, very poor	> 1.60

# Powders: Flowability Testers

The Flowability Tester BEP2 has been specifically designed to address the specifications in and comments raised by the Ph. Eur. and USP on Powder Flow.

The Flowability Tester BEP2 from Copley 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.



#### BEP2 with Cylinder Attachment

Comprising a stainless-steel cylinder measuring 76 mm long x 57 mm and a capacity of 200 mL, each attachment comes complete with 20 interchangeable stainless-steel disks each with a precision drilled hole between 4 mm and 36 mm.

The cylinder attachment can be used in two different ways:

- i. For quantitative flowability tests based on mass vs time
- ii. To determine the intrinsic flowability of a powder via a flowability index based on comparative measurements.



#### **BEP2** with Funnel Attachment

The stainless-steel flow funnel is designed to simulate flow in a hopper or other similar production situation.

Supplied with 10 mm, 15 mm and 25 mm aperture quick-change nozzles it is manufactured from pharmaceutical grade 316 stainless steel. Testing is carried out with an easy-to-use shutter.



- BEP2 with Funnel and Balance/Timer
  Attachments
- B BEP2 with Cylinder and Balance/Timer Attachments

Remove the hassle of a stopwatch by adding our balance and timer attachment to the shutter mechanism to simplify and streamline mass vs time tests including:

- a) Flow time of a predetermined sample weight
- b) Flow time of a predetermined sample volume
- c) Weight of a sample in a predetermined time
- d) Time against sample weight (weight/time).



#### BEP2 with Funnel and Shear Cell Attachments

The Copley cylindrical shear cell attachment is ideal for determining flow properties based on the bulk density and shear strength of fine-grained powders and bulk solids.

Providing important information on a given material's behaviour in bins, hoppers, feeders and other handling equipment, the Copley shear cell enables precise control of test parameters for a wide range of flowability measurements.

#### BEP2 with Funnel and Angle of Repose Attachments

Providing a simple method for assessing the friction characteristics of powders, the Copley angle of repose attachment is ideal for predicting manufacturing issues associated with resistance to movement between particles.

Comprises a 100 mm diameter circular test platform together with a digital gauge. Powder heights of up to 300mm can be easily read from the clear gauge display. Dividing this value by 50 calculates the angle of repose.





Manually operated Stirrer for Funnel Attachment









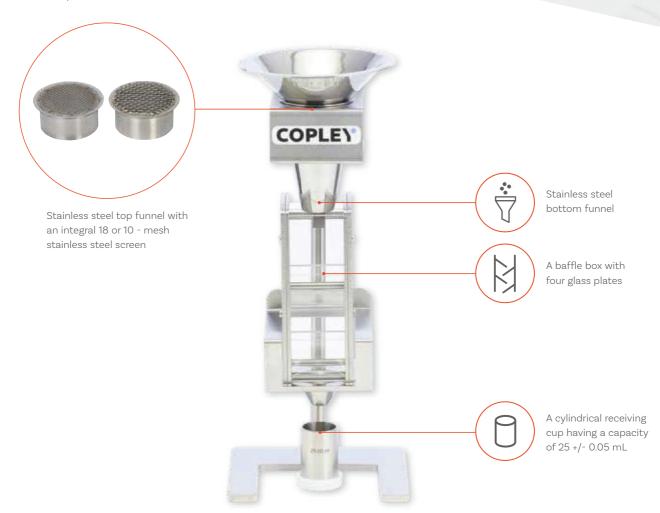
### Flowability

Cat. No.	Description
1650	Flowability Tester Model BEP2 Stand and Upright
1651	Cylinder Attachment
1652	Funnel Attachment
1656	Manually operated Stirrer for Funnel Attachment
1653	Balance/Timer Attachment
1654	Angle of Repose Attachment*
	*Requires the Funnel Attachment (Cat No. 1652) to operate
1655	Shear Cell Attachment*
	*Requires the Funnel Attachment (Cat No. 1652) to operate
1657	Anti-Static Grounding Kit for BEP2
1658	IQ/OQ Documentation Pack
1659	Qualification Tools
1660	Re-calibration of Qualification Tools

# Powders: Bulk Density Tester

The Bulk Density Tester (Scott Volumeter) is described in Ph. Eur. and USP and is designed for measuring the bulk density of fine powders and similar products.

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 Scott Volumeter obviates this problem.



#### Scott Volumeter

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)

## Powders: Tapped Density Testers

A high reliability solution for routine measurement of the tapped density of powders, granules and similar products, the Copley JVi testers are the only single tapped density systems on the market that offer all three methods specified in Eur. Ph. and USP.

Available with either one or two test stations, the JVi tapped density series provides detailed reports, including Hausner ratio calculations for a wide range of industrial standards.



Ph. Eur. and USP Compliant



Adjustable stroke frequency control for accelerated or high sensitivity testing



Intuitive touchscreen control to simplify operation



One to two test station unit configurations



Supports Methods 1, 2 and 3



Extensive data reporting output options



Integrated calculation of bulk density, tapped density, Hausner ratio and compressibility index



Option to automate and remotely control JVi systems



# JVi Series: Key Features



### JVi Series: Touchscreen User Interface

















#### **Kev Features:**

- Intuitive menu structure enables users to locate features quickly and easily
- Easy-set user-configurable test parameters:

  Test speed
  - Number of tans
  - Tost duration
- Status of 'Actual' v 'Set' test parameters clearly displayed throughout testing
- Test progress bar provides clear and constant indication on run status

- Resistive touchscreen interface can be operated with gloves on
- Hygienic wipe-clean screen
- High productivity easy system set-up and operation minimises training burden.
- · Calculation of:
  - **Bulk Density**
  - Tapped Density
  - Hausner Ratio
  - Compressibility Index





### Reporting

Extensive data output options are available as standard, including direct reporting to a printer or PC.

#### Reported parameters

· Speed (taps per minute)

Set

Average

Maximum

Minimum

Number of taps

Set

Actual

Calculations

Start weight/volume (g/mL) End weight/volume (g/mL) Bulk Density (g/mL)

Tapped Density (g/mL)

Hausner Ratio

Compressibility Index (%)







### Compliance & Maintenance



- ✓ Certificate of compliance to Ph. Eur./USP provided as standard
- ✓ Comprehensive IQ/OQ/PQ documentation packages and toolkits available

# Choose your JVi Tapped Density System



**Cat Number** 

1631

**Test Station Capacity** 

1 Cylinder

**Eur. Ph and USP Test Methods Supported** 

1, 2, 3

Unit Dimensions (w x d x h)

260 x 347 x 562 mm (with 250 mL cylinder)



**Cat Number** 

1632

**Test Station Capacity** 

2 Cylinders

Eur. Ph and USP Test Methods Supported

1\*, 2, 3

\* Only one station available for this method

Unit Dimensions (w x d x h)

260 x 347 x 562 mm (with 250 mL cylinders)



Acoustic Cabinet reduces the noise level produced by the JVi series unit



JV 200i with Method 3 platform

JVi Seri	es: Tec	hnical	Speci	ficat	ions

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Pharmacopoeial Compliance	Ph. Eur. 2.9.34 USP <616>
User Interface	Resistive touchscreen
Test Duration Parameters	Time or Taps
Testing Run Time	Up to 99 hours, 59 minutes, 59 seconds
Test Station Capacity	Up to 2 stations (1 left, 1 right)
Data Output	RS 232 USB Type A (for a USB printer) USB Type B (for communications with a PC)
Methods Supported Details	Method 1: 14 mm @ 300 taps/min  Method 2: 3 mm @ 250 taps/min  Method 3: 3 mm, 14 mm - 50/60 taps/minute
Alarm	End of testing (audible)

### JVi Series

Cat. No.	Description	Cat. No.	Description
1631-1	Tapped Density Tester JV 100i - Method 1, 250 mL Cylinder	1604	250 mL Measuring Cylinder (spare)
1631-2	Tapped Density Tester JV 100i - Method 2, 250 mL Cylinder	1605	100 mL Measuring Cylinder (option)
1631-3	Tapped Density Tester JV 100i - Method 3 - 3mm Drop	1635	Tapped Density Method 3 Cup
1631-4	Tapped Density Tester JV 100i - Method 3 - 14mm Drop	1643	Platform for use with 250 mL Cylinder - Method 1
1632-2	Tapped Density Tester JV 200i - Method 2, 250 mL Cylinders	1644	Platform for use with 250 mL Cylinder - Method 2
1632-3	Tapped Density Tester JV 200i - Method 3 - 3mm Drop	1641	Platform for use with 100 mL Cylinder - Method 1
1632-4	Tapped Density Tester JV 200i - Method 3 - 14mm Drop	1642	Platform for use with 100 mL Cylinder - Method 2
		1645	Platform for use with Cup - Method 3 - 3mm Drop
1603	IQ/OQ/PQ Documentation Pack	1646	Platform for use with Cup - Method 3 - 14mm Drop
1616	Qualification Tools		
1617	Re-calibration of Qualification Tools	1636	Acoustic Cabinet for JV100i / 200i
		1609	50 mL Measuring Cylinder Kit
		1610	25 mL Measuring Cylinder Kit
		1611	10 mL Measuring Cylinder Kit
		1612	5 mL Measuring Cylinder Kit