PHARMACOPOEIAL DISCUSSION GROUP

CODE: G-05

NAME: Powder flow

REVISION 1

It is understood that sign-off covers the technical content of the draft and each party will adapt it as necessary to conform to the usual presentation of the pharmacopoeia in question; such adaptation includes stipulation of the particular pharmacopoeia's reference materials and general chapters.

Harmonised provisions:

Provision	EP	JP	USP
Angle of repose	+	+	+
Compressibility index and Hauser ratio	+	+	+
Flow through an orifice	+	+	+
Shear cell methods	+	+	+

+ will adopt and implement; – will not stipulate

Non-harmonised provisions:
None.

Local requirements
None.

European Pharmacopoeia

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POWDER FLOW

The widespread use of powders in pharmaceutics has generated a variety of methods for characterising powder flow. Not surprisingly, scores of references appear in the pharmaceutical literature, attempting to correlate the various measures of powder flow to manufacturing properties. The development of such a variety of test methods was inevitable; powder behavior is multifaceted and thus complicates the effort to characterise powder flow.

The purpose of this chapter is to describe the methods for characterising powder flow that are most frequently used in pharmaceutical applications. In addition, while it is clear that no single and simple test method can adequately characterise the flow properties of pharmaceutical powders, this chapter proposes the standardisation of these test methods.

For testing the powder flow, the four most commonly used methods are described below. Important experimental considerations are identified and recommendations are made regarding standardisation of the methods:

– angle of repose,

– compressibility index (Carr index) or Hausner ratio,

– flow through an orifice,

– shear cell.

In general, any method of measuring powder flow must be practical, useful, reproducible and sensitive, and must yield meaningful results. Replicate determinations are desirable for the determination using any of these techniques. It bears repeating that no simple powder flow method will adequately or completely characterise the wide range of flow properties experienced in pharmaceutical applications. An appropriate strategy may well be the use of multiple standardised test methods to characterise the various aspects of powder flow as needed by the pharmaceutical scientist.

ANGLE OF REPOSE

The angle of repose has been used in several branches of science to characterise the flow properties of solids. Angle of repose is a characteristic related to interparticulate friction, or resistance to movement between particles. Angle of repose test results are reported to be very dependent upon the method used. Experimental difficulties arise due to segregation and consolidation or aeration of the powder as the cone is formed. Despite its difficulties, the method continues to be used in the pharmaceutical industry, and a number of examples demonstrating its value in predicting manufacturing problems appear in the literature.

The angle of repose is the constant, three-dimensional angle (relative to the horizontal base) assumed by a cone-like pile of powder formed by any of several different methods, described briefly below.

Methods for angle of repose

A variety of angle of repose test methods are described in the literature. The most common methods for determining the static angle of repose can be classified based on 2 important experimental variables:

– the height of the 'funnel' through which the powder passes may be fixed relative to the base, or the height may be varied as the pile forms;

– the base upon which the pile forms may be of fixed diameter or the diameter of the powder cone may be allowed to vary as the pile forms.

Variations of the above methods have also been used to some extent in the pharmaceutical applications:

– *drained angle of repose*: this is determined by allowing an excess quantity of powder positioned above a fixed diameter base to 'drain' from the container. Formation of a cone of powder on the fixed diameter base allows determination of the drained angle of repose;

– *dynamic angle of repose*: this is determined by filling a cylinder (with a clear, flat cover on one end) and rotating it at a specified speed. The dynamic angle of repose is the angle (relative to the horizontal) formed by the flowing powder. The internal angle of kinetic friction is defined by the plane separating those particles sliding down the top layer of the powder and those particles that are rotating with the drum (with roughened surface).

Relative ranking of flow for angle of repose

While there is some variation in the qualitative description of powder flow using the angle of repose, much of the pharmaceutical literature appears to be consistent with the classification by Carr[1], which is shown in Table 1. There are examples in the literature of formulations with an angle of repose in the range of 40-50 degrees that manufactured satisfactorily. When the angle of repose exceeds 50 degrees, the flow is rarely acceptable for manufacturing purposes.

Table 1. – *Relative ranking of flow by angle of repose*[2]

Flow property	**Angle of repose (degrees)**
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

Experimental considerations for angle of repose

Angle of repose is not an intrinsic property of the powder, that is to say, it is very much dependent upon the method used to form the cone of powder. On this subject, the existing literature raises these important considerations:

– the peak of the cone of powder can be distorted by the impact of powder from above. By carefully building the powder cone, the distortion caused by impact can be minimised;

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[1] Carr RL. Evaluating flow properties of solids. *Chem. Eng* 1965 ; 72:163-168.

– the nature of the base upon which the powder cone is formed influences the angle of repose. It is recommended that the powder cone be formed on a 'common base', which can be achieved by forming the cone of powder on a layer of powder. This can be done by using a base of fixed diameter with a protruding outer edge to retain a layer of powder upon which the cone is formed.

Recommended procedure for angle of repose

Form the angle of repose on a fixed base with a retaining lip to retain a layer of powder on the base. The base must be free of vibration. Vary the height of the funnel to carefully build up a symmetrical cone of powder. Care must be taken to prevent vibration as the funnel is moved. The funnel height is maintained at approximately 2-4 cm from the top of the powder pile as it is being formed in order to minimise the impact of falling powder on the tip of the cone. If a symmetrical cone of powder cannot be successfully or reproducibly prepared, this method is not appropriate. Determine the angle of repose by measuring the height of the cone of powder and calculating the angle of repose, α, from the following equation:

$$\tan(\alpha) = \frac{\text{height}}{0.5 \times \text{base}}$$

COMPRESSIBILITY INDEX AND HAUSNER RATIO

The compressibility index (Carr index) and the closely related Hausner ratio may predict powder flow characteristics as being affected by e.g. , size and shape, material density, surface area, moisture content, and cohesiveness of powder. The compressibility index and the Hausner ratio are calculated from the untapped and tapped bulk density or untapped and tapped bulk volume of a powder. For additional information see *G-02 Bulk Density of Powders.*

Methods for compressibility index and Hausner ratio

While there are some differences in the method of determining the compressibility index and Hausner ratio, the basic procedure is to measure the untapped bulk volume, (V_0), and the final tapped bulk volume, (V_f), of the same powder sample after tapping the powder until no further volume changes occur. The compressibility index and the Hausner ratio are calculated as follows:

$$\text{Compressibility Index} = 100 \times \frac{V_0 - V_f}{V_0}$$

$$\text{Hausner Ratio} = \frac{V_0}{V_f}$$

Alternatively, the compressibility index and Hausner ratio may be calculated using measured values of untapped bulk density ($\rho_{untapped}$) and tapped bulk density (ρ_{tapped}) as follows:

$$\text{Compressibility Index} = 100 \times \frac{\rho_{tapped} - \rho_{untapped}}{\rho_{tapped}}$$

$$\text{Hausner Ratio} = \frac{\rho_{tapped}}{\rho_{untapped}}$$

In a variation of these methods, the rate of consolidation is sometimes measured rather than, or in addition to, the change in volume that occurs on tapping. For the compressibility index and the Hausner ratio, a commonly reported relative ranking of flow is given in Table 2.

Table 2. – *Relative ranking of flow by compressibility index and Hausner ratio*[2]

Compressibility index (per cent)	**Flow character**	**Hausner ratio**
1-10	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

Compressibility index and Hausner ratio are not intrinsic properties of the powder, that is to say, they are dependent upon the methodology used. Several important considerations affecting the determination of the untapped bulk volume, V_0, the final tapped bulk volume, V_f, the untapped bulk density, $\rho_{untapped}$, and the tapped bulk density, ρ_{tapped}, are the following:

– the diameter and the mass of the graduated cylinder used with its holder,

– the number of times the powder is tapped to achieve the tapped bulk density,

– the apparatus drop height,

– the mass of powder used in the test,

– rotation of the sample during tapping.

FLOW THROUGH AN ORIFICE

The flow of a powder depends upon many factors, some of which are particle-related and some related to the process. Monitoring its ability to flow through an orifice (by assessing the "arching diameter", the orifice diameter at which the powder arches and is no longer able to discharge) and its flow rate have been used to measure powder flow. Of particular significance is the utility of monitoring flow continuously, since pulsating flow patterns have been observed even for free-flowing powders. Changes in flow rate as the container empties can also be observed. Empirical equations relating flow rate to the diameter of the opening, particle size, and particle density have been determined. Whereas assessing the arching diameter of a powder may be used for cohesive and free-flowing powders, determining the flow rate through an orifice is useful only with free-flowing powders.

The flow rate through an orifice is generally measured as the mass per time flowing from any of a number of types of containers (cylinders, funnels, hoppers). Measurement of the flow rate can be in discrete increments or continuous.

Methods for flow through an orifice

There are a variety of methods described in the literature. The most common for determining the flow through an orifice can be classified based on 3 important experimental variables:

– the type of container used to contain the powder. Common containers are cylinders, funnels, and hoppers from production equipment;

– the size and shape of the orifice used. The orifice diameter and shape are critical factors in determining powder flow;

– the method of measuring powder flow rate. Flow rate can be measured continuously using an electronic balance with some sort of recording device (strip chart recorder, computer). It can also be measured in discrete samples (for example, the time it takes for 100 g of powder to pass through the orifice to the nearest tenth of a second or the amount of powder passing through the orifice in 10 s to the nearest tenth of a gram).

Variations in methods for flow through an orifice

Either mass flow rate or volume flow rate can be determined. Mass flow rate is the easier of the methods, but it biases the results in favour of high-density powders. Since die fill is volumetric, determining volume flow rate may be preferable. A vibrator is occasionally attached to facilitate flow from the container, however, this appears to complicate interpretation of results. A moving orifice device has been proposed to more closely simulate rotary press conditions. The minimum diameter orifice through which powder flows can also be identified.

No general scale is available because flow rate is critically dependent on the method used to measure it. Comparison between published results is difficult.

Experimental considerations for flow through an orifice

Flow through an orifice is not an intrinsic property of the powder. It is very much dependent upon the methodology used. The existing literature points out several important considerations affecting these methods:

– the diameter and shape of the orifice,

– the type of container material (metal, glass, plastic),

– the diameter and height of the powder bed.

Recommended procedure for flow through an orifice

Flow rate through an orifice can be used only for powders that have some capacity to flow. It is not useful for cohesive powders. Provided that the height of the powder bed (the 'head' of powder) is much greater than the diameter of the orifice, the flow rate is virtually independent of the powder head. It is advisable to use a cylinder as the container, because the walls of the container must have little effect on flow. This configuration results in flow rate being determined by the movement of powder over powder, rather than powder along the wall of the container. Powder flow rate often increases when the height of the powder column is less than twice the diameter of the column. The orifice must be circular and the cylinder must be free of vibration. General guidelines for dimensions of the cylinder are as follows:

– diameter of the opening greater than 6 times the diameter of the particles,

– diameter of the cylinder greater than twice the diameter of the opening.

Use of a hopper as the container may be appropriate and representative of flow in a production situation. It is not advisable to use a funnel, particularly one with a stem, because flow rate will be determined by the size and length of the stem as well as the friction between the stem and the powder. A truncated cone may be appropriate, but flow will be influenced by the powder-wall friction coefficient, thus, selection of an appropriate construction material is important.

For the opening in the cylinder, use a flat-faced bottom plate with the option to vary orifice diameter to provide maximum flexibility and better ensure a powder-over-powder flow pattern. Rate measurement can be either discrete or continuous. Continuous measurement using an electronic balance can more effectively detect momentary flow rate variations.

SHEAR CELL METHODS

In an effort to put powder flow studies and hopper design on a more fundamental basis, a variety of powder shear testers and methods that permit a more thorough and precisely defined assessment of powder flow properties have been developed. Shear cell methodology has been used extensively in the study of pharmaceutical powders. From these methods, a wide variety of parameters can be obtained, including the yield locus representing the shear-stress to normal-stress relationship at incipient flow, the angle of internal friction, the unconfined yield strength, powder cohesion, and a variety of related parameters such as the flow function coefficient. Because of the ability to control experimental parameters more precisely, flow properties can also be determined as a function of consolidation load, time, and other environmental conditions. These methods have been successfully used to determine critical hopper and bin dimensions.

Methods for shear cell

One type of shear cells corresponds to translational shear cells which are split horizontally, forming a shear plane between the stationary and the moveable portion of the shear cell. After powder bed consolidation in the shear cell (using a well-defined procedure), the force necessary to shear the powder bed is determined. Translational shear cells may have a cylindrical shape or a rectangular box shape.

A second type of shear cells corresponds to rotational shear cells. These include cylindrical shape and annular shape cells. Their design offers some advantages over the translational shear cell design, including the need for less material. A disadvantage, however, is that because of their design, the powder bed is not sheared as uniformly because material on the outside of the rotational shear cell is sheared more than material in the inner region.

All of the shear cell methods have their advantages and disadvantages, but a detailed review is beyond the scope of this chapter. As with the other methods for characterising powder flow, many variations are described in the literature. A significant advantage of shear cell methodology in general is a greater degree of experimental control.

Recommendations for shear cell

The many existing shear cell configurations and test methods provide a wealth of data and can be used very effectively to characterise powder flow. They are also helpful in the design of equipment such as hoppers and bins. Because of the diversity of available equipment and experimental procedures, no specific recommendations regarding methodology are presented in this chapter. It is recommended that the results of powder flow characterisation using shear cell methodology include a complete description of equipment and methodology used.