2.4.35. Bulk Density of Powders

Published on: 06.06.2024 **Last date for comments:** 17.07.2024

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Manufacturers, regulatory authorities, health authorities, researchers, and other stakeholders are invited to provide their feedback and comments on this draft proposal. Manufacturers are also invited to submit samples of their products to the IPC to ensure that the proposed monograph adequately controls the quality of the product(s) they manufacture. Comments and samples received after the last date will not be considered by the IPC before finalizing the monograph.

Please send any comments you may have on this draft document to lab.ipc@gov.in, with a copy to Dr. Gaurav Pratap Singh (email: gpsingh.ipc@gov.in) before the last date for comments.

Document History and Schedule for the Adoption Process

Description	Details
Document version	1.0
Monograph proposed for inclusion	IP 2026
Tentative effective date of monograph	January, 2026
First draft published on IPC website for public comments	06.06.2024
Draft revision published on IPC website for public comments	-
Further follow-up action as required.	

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Change **to**:

2.4.35. Bulk Density of Powders

This General Chapter has been harmonized with corresponding texts of the European Pharmacopoeia, the Japanese Pharmacopoeia and the United States Pharmacopeia.
Portions of the IP text that and are not part of the PDG harmonized text, are marked with symbols (♦ ♦).

~~**Bulk Density**~~

The bulk density of a powder is the ratio of the ~~mass~~ ♦weight♦ of ~~an untapped~~ a powder sample ~~and~~ to its volume, including the contribution of the interparticulate void volume. Hence, the bulk density depends on the material density and the packing arrangement in the powder bed. Bulk density is commonly expressed in grams per millilitre (1 g/ml = 1 g/cm^3 = 1000 kg/m^3).

~~both the density of powder particles and the spatial arrangement of particles in the powder bed. The bulk density is expressed in grams per ml (g/ml) although the international unit is kilograms per cubic meter (1 g/ml = 1000 kg/m^3) because the measurements are made using cylinders. It may also be expressed in grams per cubic centimeter (g/cm^3).~~
The ~~bulking~~ bulk properties of a powder are dependent upon the preparation, treatment, and storage of the sample, i.e., how it has been ~~was~~ handled. The particles can be packed to have a range of bulk densities~~;~~. Therefore, it is necessary to differentiate the untapped bulk density and tapped bulk density. The tapped and untapped bulk densities are used to evaluate powder flow. A comparison of the tapped bulk and untapped bulk densities can give an indirect measure of the relative importance of the interparticulate interactions influencing the bulk properties of a powder. For comparison specifics, please see section Measures of Powder Compressibility. For additional context, please see general chapter Powder Flow (2.4.48).

~~however, the slightest disturbance of the powder bed may result in a changed bulk density. Thus, the bulk density of a powder is often very difficult to measure with good reproducibility and, in reporting the results; it is essential to specify how the determination was made.~~

Untapped Bulk Density

The untapped bulk density of a powder is determined by ~~either~~ measuring the volume of a known ♦weight♦ of powder sample, ~~that~~ which may have been passed through a sieve, into a graduated cylinder (*Method I*), or by measuring the ~~mass~~ ♦weight♦ of a known volume of powder that has been passed through a volumeter into a cup (*Method II*) or has been introduced into a measuring vessel (*Method III).*

~~*Method I* and *Method III* are favored.~~

The slightest disturbance of the powder bed may result in a changed untapped bulk density, especially for cohesive powders. In these cases, the untapped bulk density is often very difficult to measure with good reproducibility and, in reporting the results, it is essential to specify how the determination was made.

Method I. Measurement in a Graduated Cylinder

Procedure. Pass a quantity of ~~material~~ powder sufficient to complete the test through a sieve with apertures greater than or equal to 1.0 mm, if necessary, to break up agglomerates that may have formed during storage; this must be done gently to avoid changing the nature of the ~~material~~ powder. ~~Into a dry graduated 250-ml cylinder (readable to 2 ml), gently introduce, without compacting,~~ Gently pour approximately 100 g (*m*) of test sample~~(*M*),~~ weighed with 0.1 per cent accuracy, into a dry graduated 250-ml cylinder (readable to 2 ml)~~.~~ Any significant compacting stress should be avoided, for example, by using a funnel or by tilting the graduated cylinder. If necessary ~~Carefully~~ carefully level the powder without compacting, and ~~if necessary, and~~ read the untapped bulk volume ~~unsettled apparent volume~~ (V_0) to the nearest graduated unit. Calculate the untapped bulk density in g per ml by the formula m/V_0. Replicate determinations performed on separate powder samples are desirable. ~~Generally, replicate determinations are desirable for the determination of this property.~~

If the powder density is too low or too high, such that the test sample has an untapped ~~apparent~~ bulk volume of ~~either~~ more than 250 ml or less than 150 ml, it is not possible to use 100 g of powder sample. In this case ~~Therefore,~~ a different amount of powder is ~~has to be~~ selected as the test sample, such that its untapped ~~apparent~~ bulk volume is

between 150 ml ~~to~~ and 250 ml (i.e. untapped bulk ~~apparent~~ volume greater than or equal to 60 per cent of the total volume of the graduated cylinder); the weight of the test sample is specified in the expression of results.

For test samples having an ~~apparent~~ untapped bulk volume between 50 ml and 100 ml, a 100 ml graduated cylinder readable to 1 ml can be used; the volume of the graduated cylinder is specified in the expression of results.

Method II. Measurement in a Volumeter

Apparatus. The apparatus[1] (Fig. 2.4.35-1) consists of a top funnel fitted with a 1.0-mm sieve~~. The funnel is~~ mounted over a baffle box containing four glass baffles ~~plates~~ over which the powder slides and bounces as it passes. At the bottom of the baffle box is a funnel that collects the powder and allows it to pour into a cup ~~of specified capacity~~ mounted directly below it. The cup may be cylindrical (25.00 ± 0.05 ml volume with an ~~inside~~ internal diameter of ~~30.00 ± 2.00~~ 29.50 ± 2.50 mm) or ~~a square~~ cubical (16.39 ± ~~0.2~~ 0.05 ml volume~~) with inside dimensions of 25.4 ± 0.076 mm).~~

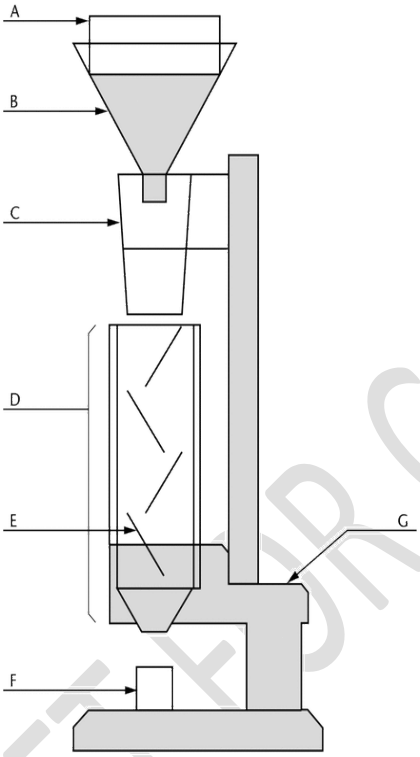

Fig. 2.4.35-1: Volumeter. (A) 1.0-mm sieve; (B) powder funnel; (C) loading funnel; (D) baffle box; (E) glass baffle; (F) cup; (G) stand.

Procedure. Allow an excess of powder to flow through the apparatus into the sample receiving cup until it overflows, using a minimum of 25 cm^3 of powder with the cubical ~~square~~ cup and 35 cm^3 of powder with the cylindrical cup. Carefully, scrape excess powder from the top of the cup by smoothly moving the edge of the reclined spatula blade across ~~of a spatula perpendicular to and in contact with~~ the top surface of the cup, taking care to keep the spatula tilted backwards ~~perpendicular~~ to prevent packing or removal of powder from the cup. Remove any ~~material~~ powder from the ~~sides~~ side of the cup, and determine the ♦weight♦, ~~*M* (*m*),~~ of the powder to the nearest 0.1 per cent. Calculate the untapped bulk density in grams per mililitre using ~~, in g/ml, by~~ the formula:

$$\frac{m}{V_0}$$

~~in which~~(where V_0 is the volume~~, in ml,~~ of the cup). Replicate determinations performed on separate powder samples are desirable. Record the average of three determinations using three different powder samples.

[1]The apparatus (the Scott Volumeter) conforms to the dimensions in ISO 3923-2:1981 or ASTM B329. ASTM B329–14.

Method III. Measurement in a Vessel

Apparatus. The apparatus consists of a 100-ml cylindrical stainless steel vessel ~~of stainless steel~~ with dimensions as specified in Fig. 2.4.35-2.

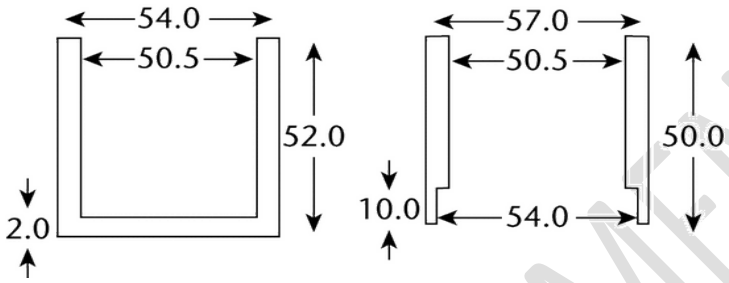

Fig. 2.4.35-2: Measuring Vessel (Left) and Cap (Right)

Dimension in millimetres

Procedure. Pass a quantity of powder sufficient to complete the test through a 1.0 mm sieve, if necessary, to break up agglomerates that may have formed during storage, and allow the obtained sample to flow freely into the measuring vessel until it overflows. Carefully scrap the excess powder from the top of the vessel as described ~~for~~ under *Method II.* Determine the ♦weight♦ ~~(M_0)~~ (m_0) of the powder to the nearest 0.1 per cent by subtracting ~~subtraction of~~ the previously determined ~~mass~~ ♦weight♦ of the empty measuring vessel. Calculate the untapped bulk density in grams per millilitre ~~(g/ml)~~ ~~by~~ using the formula ~~$M_0/100$~~ $m_0/100$~~,~~. Replicate determinations performed on separate powder samples are desirable. ~~and record the average of three determinations using three different powder samples.~~

Tapped Bulk Density

The tapped bulk density is an increased bulk density attained after mechanically tapping a receptacle ~~container~~ containing the powder sample. The ~~T~~tapped bulk density is obtained by mechanically tapping a graduated ~~measuring~~ cylinder or vessel containing ~~a~~ the powder sample. After ~~observing~~ recording the initial untapped bulk volume (V_0) and weight (m_0) of the powder sample, ~~powder volume or weight,~~ the ~~measuring~~ graduated cylinder or vessel is mechanically tapped, and volume or ♦weight♦ readings are taken until little further volume or ♦weight♦ change is observed as described in the method. The mechanical tapping is achieved by raising the graduated cylinder or vessel and allowing it to drop ~~under its own weight~~ a specified distance ♦under its own weight♦ by ~~either~~ one of three methods as described below. Devices that rotate the graduated cylinder or vessel during tapping may be preferred to give a more leveled surface after tapping. ~~minimize any possible separation of the mass during tapping down.~~

Method I - Measurement in a Graduated Cylinder

Apparatus. The apparatus (Fig. 2.4.35-3) consists of the following:

– A 250-ml graduated cylinder (readable to 2 ml) with a ~~mass~~ ♦weight♦ of 220 ± 44 g.
– A ~~settling~~ tapping apparatus capable of producing~~, in 1~~ per minute, ~~either~~ ~~nominally 250 ± 15 taps from a height of 3 ± 0.2 mm, or~~ nominally 300 ± 15 taps from a height of 14 ± 2 mm. The support for the graduated cylinder, with its holder, has a ~~mass~~ ♦weight♦ of 450 ± 10 g.

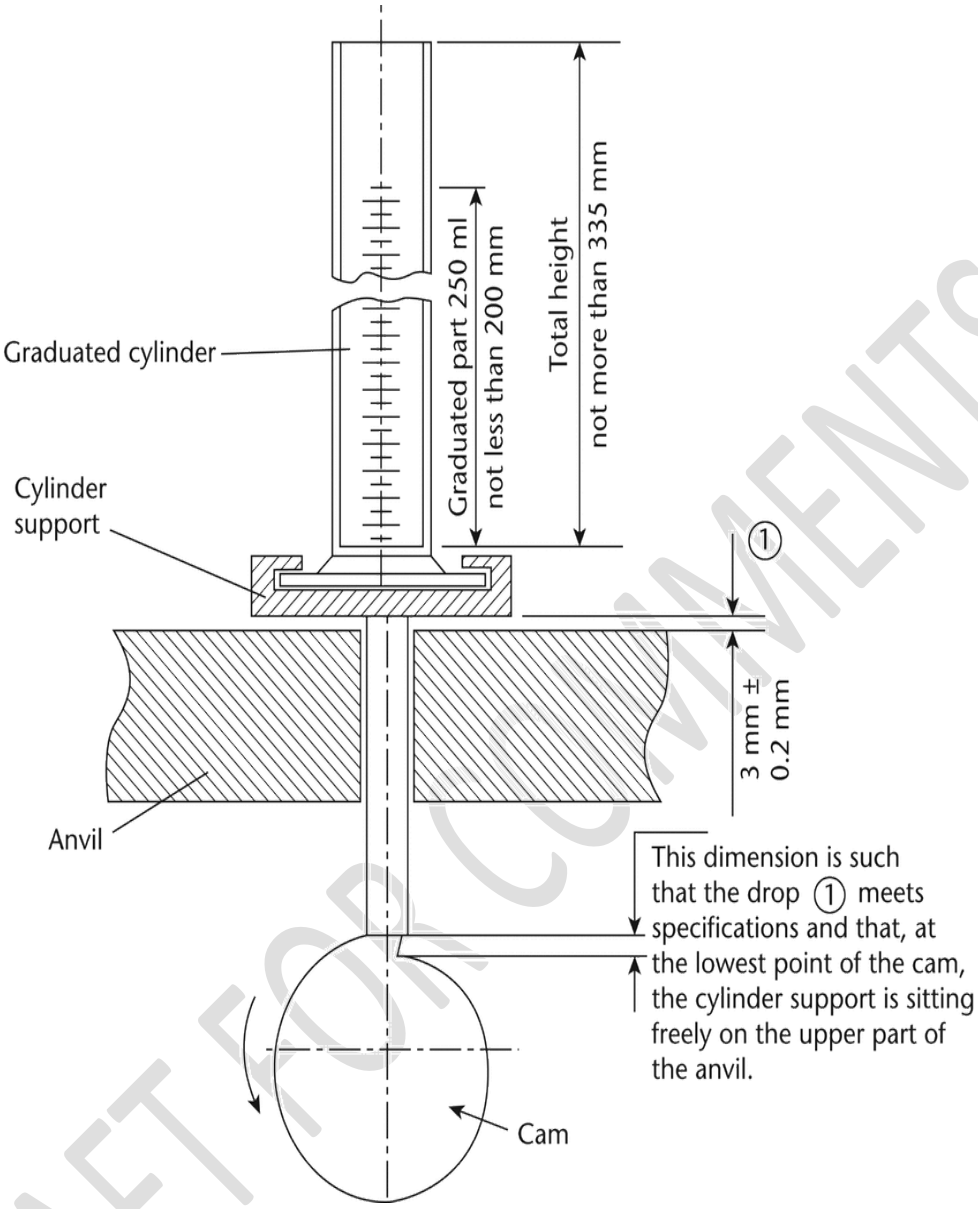

Fig. 2.4.35-3: ~~Settling~~ Tapping device for Powder Samples

Dimension in millimetres

Procedure. Proceed as described above for the determination of the untapped bulk volume (V_0). Secure the graduated cylinder in the ~~holder~~ support. Carry out 10, 500, and 1250 taps on the same powder sample and read the corresponding volumes V_{10}, V_{500}, and V_{1250} to the nearest graduated unit. If the difference between V_{500} and V_{1250} is less than or equal to 2 ml, V_{1250} is the tapped bulk volume. If the difference between V_{500} and V_{1250} exceeds 2 ml, repeat in increments of, for example, ~~such as~~ 1250 taps, until the difference between ~~succeeding~~ successive measurements is less than or equal to 2 ml. Fewer taps may be appropriate for some powders, when validated. Calculate the tapped bulk density in grams per millitre ~~(g/ml)~~ using the formula $m/$~~V_F~~-V_f ~~in which~~ (where ~~V_F~~-V_f is the final tapped bulk volume). Replicate determinations are desirable for the determination of this property. Specify the drop height with the results.

~~Generally, replicate determinations are desirable for the determination of this property. Specify the drop height with the results. If it is not possible to use a 100 g test sample,~~ If the available sample amount is insufficient for an untapped volume of 150 ml, use a reduced amount and a suitable 100-ml graduated cylinder (readable to 1 ml) weighing 130 ± 16 g and mounted on a ~~holder~~ support weighing 240 ± 12 g. The untapped volume of the sample should be between 50 ml and 100 ml. If the difference between V_{500} and V_{1250} is less than or equal to 1 ml, V_{1250} is the tapped bulk volume. If the difference between V_{500} and V_{1250} exceeds 1 ml, repeat in increments of, for example, 1250 taps, until the difference between successive measurements is less than or equal to 1 ml. The modified test conditions are specified in the expression of the results.

Method II - Measurement in a Volumeter

~~Apparatus and~~ Procedure. Proceed as directed under *Method I* except that the mechanical tester provides a fixed drop of 3 ± 0.2 mm at a nominal rate of 250 ± 15 taps per minute.

Method III - Measurement in a Vessel

~~Apparatus and~~ Procedure. Proceed as directed in *Method III ~~– Measurement in a Vessel~~* for measuring the untapped bulk density using the measuring vessel equipped with the cap shown in Fig. 2.4.35-2~~Figure 2~~. The measuring vessel with the cap is lifted 50–60 times per minute by the use of a suitable tapped density tester. Carry out 200 taps, remove the cap, and carefully scrape excess powder from the top of the measuring vessel by smoothly moving the edge of a reclined spatula blade across the top surface of the cup, taking care to keep the spatula tilted backward to prevent packing or removal of powder from the vessel. Determine the ♦weight♦ (m) of the powder to the nearest 0.1 per cent by subtracting the previously determined ♦weight♦ of the empty measuring vessel. ~~as described in Method III – Measurement in a Vessel for measuring the bulk density~~ . Repeat the procedure using 400 taps. If the difference between the two ~~masses~~ ♦weights♦ obtained after 200 and 400 taps exceeds 2 per cent, repeat the test ~~carry out a test~~ using 200 additional taps until the difference between ~~succeeding~~ successive measurements is less than 2 per cent. Calculate the tapped bulk density in grams per millilitre ~~(g/ml)~~ using the formula ~~MF~~m_f/100 (where ~~MF~~-m_f is the final tapped ~~mass~~ ♦weight♦ of powder in the measuring vessel). Replicate determinations performed on separate powder samples are desirable. ~~Record the average of three determinations using three different powder samples.~~ The test conditions, including tapping height are specified in the expression of the results.

Measures of Powder Compressibility

Because the interparticulate interactions influencing the ~~bulking~~ bulk properties of a powder ~~are~~ also ~~the interactions that~~ interfere with powder flow, a comparison of the untapped bulk and tapped bulk densities can give an indirect measure of the relative importance of these interactions in a given powder. Such a comparison is often used as an index of the ability of the powder to flow, for example the Compressibility Index or the Hausner Ratio ~~as described below~~.

The Compressibility Index and Hausner Ratio are measures of the propensity of a powder to be compressed as described above. ~~As such, they are measures of the powder's ability to settle, and they permit an assessment of the relative importance of interparticulate interactions. In a free-flowing powder, such interactions are less significant, and the bulk and tapped densities will be closer in value. For poorer flowing materials, there are frequently greater interparticle interactions, and a greater difference between the bulk and tapped densities will be observed. These differences are reflected in the *Compressibility Index* and the *Hausner Ratio*.~~

Compressibility Index. ~~Calculate by the formula:~~

$$\frac{100\,(V_0 - V_f)}{V_0}$$

~~Where,~~ V_0 = ~~unsettled apparent volume~~ untapped bulk

~~V_F~~-V_f = final tapped ~~volume~~ bulk

Hausner Ratio

$$\frac{V_0}{V_f}$$

Depending on the powder ~~material,~~ the compressibility index can be determined using V_{10} instead of V_0. If V_{10} is used, it is clearly stated with the results.