

**PHARMACOPOEIAL DISCUSSION GROUP****CORRECTION****CODE: G-02****NAME: Bulk density of powders****(Correction 1 of Rev. 4 signed on 20 January 2023)**

*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.*

**Item to be corrected:**

- Figure 3 dimension changed from "3mm ± 0.2 mm" to "14 mm± 2 mm"

**Harmonised provisions:**

<b>Provision</b>	<b>EP</b>	<b>JP</b>	<b>USP</b>
Introduction	+	+(1)	+
Untapped bulk density	+	+	+
Tapped bulk density	+	+	+
Measures of powder compressibility	+	+	+

(1) "For additional context, please see general chapter G-05 Powder Flow." not included in the JP.

+ will adopt and implement; - will not stipulate

**Non-harmonised provisions:**

None

**Local requirements**

<b>EP</b>	<b>JP</b>	<b>USP</b>
None	None	None

G-02

Revision 4 Correction 1

October 2023

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## G-02 BULK DENSITY OF POWDERS

1  
2 The bulk density of a powder is the ratio of the mass of a powder sample to its volume,  
3 including the contribution of the interparticulate void volume. Hence, the bulk density  
4 depends on the material density, and the packing arrangement in the powder bed. Bulk  
5 density is commonly expressed in grams per millilitre ( $1 \text{ g/mL} = 1 \text{ g/cm}^3 = 1000 \text{ kg/m}^3$ ).  
6 The bulk properties of a powder are dependent upon the preparation, treatment and storage  
7 of the sample, i.e. how it has been handled. The particles can be packed to have a range  
8 of bulk densities. Therefore, it is necessary to differentiate the untapped bulk density and  
9 tapped bulk density.  
10 The tapped and untapped bulk densities are used to evaluate powder flow. A comparison  
11 of the tapped bulk and untapped bulk densities can give an indirect measure of the relative  
12 importance of the interparticulate interactions influencing the bulk properties of a powder.  
13 For comparison specifics, please see section *Measures of powder compressibility*. For  
14 additional context, please see general chapter *G-05 Powder Flow*.

### Untapped bulk density

15  
16  
17 The untapped bulk density of a powder is determined by measuring the volume of a  
18 known mass of powder sample, which may have been passed through a sieve, in a  
19 graduated cylinder (Method 1), or by measuring the mass of a known volume of powder  
20 that has been passed through a volumeter into a cup (Method 2) or has been introduced  
21 in to a measuring vessel (Method 3).  
22 The slightest disturbance of the powder bed may result in a changed untapped bulk  
23 density, especially for cohesive powders. In these cases, the untapped bulk density is often  
24 very difficult to measure with good reproducibility and, in reporting the results, it is  
25 essential to specify how the determination was made.

#### METHOD 1: MEASUREMENT IN A GRADUATED CYLINDER

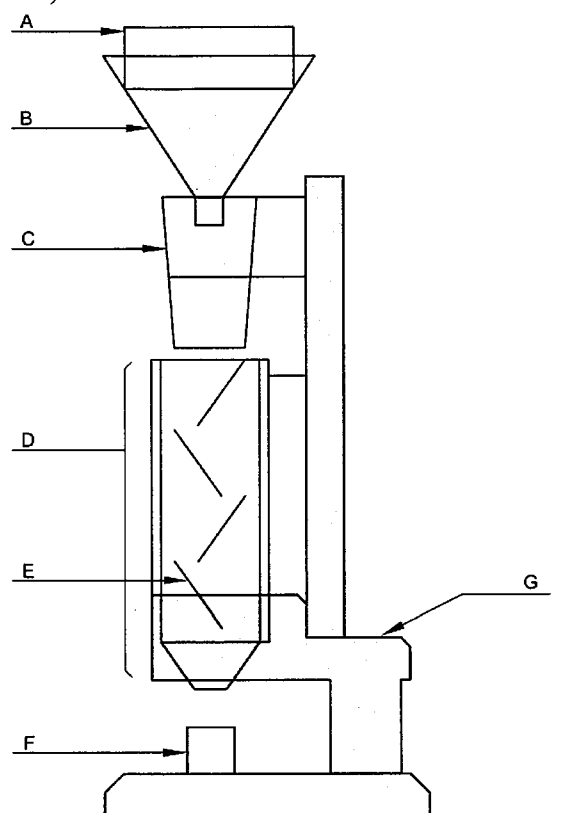
27 *Procedure.* Pass a quantity of powder sufficient to complete the test through a sieve with  
28 apertures greater than or equal to 1.0 mm, if necessary, to break up agglomerates that may  
29 have formed during storage; this must be done gently to avoid changing the nature of the  
30 powder. Gently pour approximately 100 g ( $m$ ) of the test sample, weighed with 0.1 per  
31 cent accuracy, into a dry graduated 250 mL cylinder (readable to 2 mL). Any significant  
32 compacting stress should be avoided, for example, by using a funnel or by tilting the  
33 graduated cylinder. If necessary, carefully level the powder without compacting, and read  
34 the untapped bulk volume ( $V_0$ ) to the nearest graduated unit. Calculate the untapped bulk  
35 density in grams per millilitre using the formula  $m/V_0$ . Replicate determinations  
36 performed on separate powder samples are desirable.

37 If the powder density is too low or too high, such that the test sample has an untapped  
38 bulk volume of more than 250 mL or less than 150 mL, it is not possible to use 100 g of  
39 powder sample. In this case, a different amount of powder is selected as the test sample,  
40 such that its untapped bulk volume is between 150 mL and 250 mL (i.e. untapped bulk  
41 volume greater than or equal to 60 per cent of the total volume of the graduated cylinder);  
42 the mass of the test sample is specified in the expression of results.

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

## 46 METHOD 2: MEASUREMENT IN A VOLUMETER

47 *Apparatus.* The apparatus<sup>1</sup> (Figure 1) consists of a top funnel fitted with a 1.0 mm sieve,  
 48 mounted over a baffle box containing 4 glass baffles over which the powder slides and  
 49 bounces as it passes. At the bottom of the baffle box is a funnel that collects the powder  
 50 and allows it to pour into a cup mounted directly below it. The cup may be cylindrical  
 51 (25.00 ± 0.05 mL volume with an internal diameter of 29.50 ± 2.50 mm) or cubical  
 52 (16.39 ± 0.05 mL volume).



- 53
- |                   |                 |
|-------------------|-----------------|
| A. 1.0 mm sieve   | E. glass baffle |
| B. powder funnel  | F. cup          |
| C. loading funnel | G. stand        |
| D. baffle box     |                 |

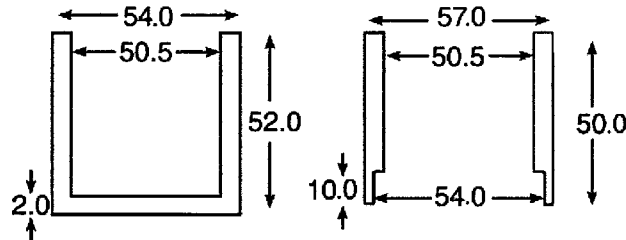
54 Figure 1. – *Volumeter*

55  
 56  
 57  
 58 *Procedure.* Allow an excess of powder to flow through the apparatus into the sample  
 59 receiving cup until it overflows, using a minimum of 25 cm<sup>3</sup> of powder with the cubical  
 60 cup and 35 cm<sup>3</sup> of powder with the cylindrical cup. Carefully, scrape excess powder from  
 61 the top of the cup by smoothly moving the edge of a reclined spatula blade across the top  
 62 surface of the cup, taking care to keep the spatula tilted backwards to prevent packing or  
 63 removal of powder from the cup. Remove any powder from the side of the cup and  
 64 determine the mass (*m*) of the powder to the nearest 0.1 per cent. Calculate the untapped  
 65 bulk density in grams per millilitre using the formula  $m/V_0$  (where  $V_0$  is the volume of the  
 66 cup). Replicate determinations performed on separate powder samples are desirable.

<sup>1</sup> The apparatus (the Scott Volumeter) conforms to the dimensions in ISO 3923-2:1981  
 or ASTM B329-14.

## 67 METHOD 3: MEASUREMENT IN A VESSEL

68 *Apparatus.* The apparatus consists of a 100 mL cylindrical stainless steel vessel with  
69 dimensions as specified in Figure 2.



70

71 Figure 2. – *Measuring vessel (left) and cap (right)*  
72 *Dimensions in millimetres*

73

74 *Procedure.* Pass a quantity of powder sufficient to complete the test through a 1.0 mm  
75 sieve, if necessary, to break up agglomerates that may have formed during storage, and  
76 allow the obtained sample to flow freely into the measuring vessel until it overflows.  
77 Carefully scrape the excess powder from the top of the vessel as described under  
78 Method 2. Determine the mass ( $m_0$ ) of the powder to the nearest 0.1 per cent by  
79 subtracting the previously determined mass of the empty measuring vessel. Calculate  
80 the untapped bulk density in grams per millilitre using the formula  $m_0/100$ . Replicate  
81 determinations performed on separate powder samples are desirable.

82

### Tapped bulk density

83 The tapped bulk density is an increased bulk density attained after mechanically tapping  
84 a receptacle containing the powder sample.

85 The tapped bulk density is obtained by mechanically tapping a graduated cylinder or  
86 vessel containing the powder sample. After recording the initial untapped bulk volume  
87 ( $V_0$ ) and mass ( $m_0$ ) of the powder sample, the graduated cylinder or vessel is mechanically  
88 tapped, and volume or mass readings are taken until little further volume or mass change  
89 is observed as described in the method. The mechanical tapping is achieved by raising the  
90 graduated cylinder or vessel and allowing it to drop a specified distance under its own  
91 mass, by one of 3 methods as described below. Devices that rotate the graduated cylinder  
92 or vessel during tapping may be preferred to give a more levelled surface after tapping.

## 93 METHOD 1: MEASUREMENT IN A GRADUATED CYLINDER

94 *Apparatus.* The apparatus (Figure 3) consists of the following:

- 95 – a 250 mL graduated cylinder (readable to 2 mL) with a mass of  $220 \pm 44$  g;
- 96 – a tapping apparatus capable of producing, per minute, nominally  $300 \pm 15$  taps from a  
97 height of  $14 \pm 2$  mm. The support for the graduated cylinder, with its holder, has a mass  
98 of  $450 \pm 10$  g.

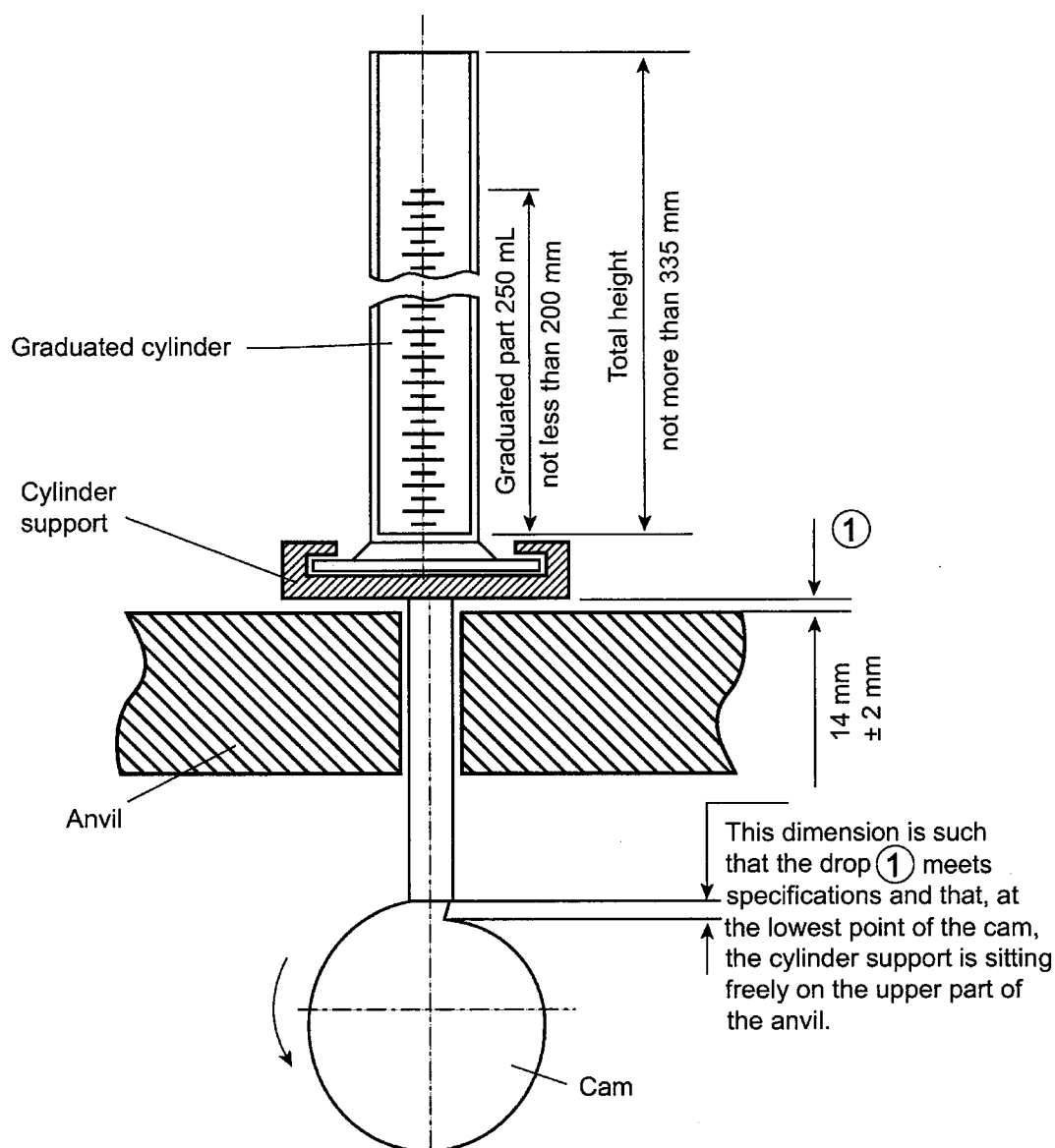


Figure 3. —Tapping device for powder samples  
Dimensions in millimetres

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100

101

102

103 *Procedure.* Proceed as described above for the determination of the untapped bulk  
 104 volume ( $V_0$ ). Secure the graduated cylinder in the support. Carry out 10, 500 and  
 105 1250 taps on the same powder sample and read the corresponding volumes  $V_{10}$ ,  $V_{500}$  and  
 106  $V_{1250}$  to the nearest graduated unit. If the difference between  $V_{500}$  and  $V_{1250}$  is less than or  
 107 equal to 2 mL,  $V_{1250}$  is the tapped bulk volume. If the difference between  $V_{500}$  and  $V_{1250}$   
 108 exceeds 2 mL, repeat in increments of, for example, 1250 taps, until the difference  
 109 between successive measurements is less than or equal to 2 mL. Fewer taps may be  
 110 appropriate for some powders, when validated. Calculate the tapped bulk density in grams  
 111 per millilitre using the formula  $m/V_f$  (where  $V_f$  is the final tapped bulk volume).  
 112 Replicate determinations are desirable for the determination of this property. Specify  
 113 the drop height with the results.

114 If available sample amount is insufficient for an untapped volume of 150 mL, use a  
 115 reduced amount and a suitable 100 mL graduated cylinder (readable to 1 mL) weighing

116 130 ± 16 g and mounted on a support weighing 240 ± 12 g. The untapped volume of  
 117 the sample should be between 50 mL and 100 mL. If the difference between  $V_{500}$  and  
 118  $V_{1250}$  is less than or equal to 1 mL,  $V_{1250}$  is the tapped bulk volume. If the difference  
 119 between  $V_{500}$  and  $V_{1250}$  exceeds 1 mL, repeat in increments of, for example, 1250 taps,  
 120 until the difference between successive measurements is less than or equal to 1 mL.  
 121 The modified test conditions are specified in the expression of the results.

## 122 METHOD 2: MEASUREMENT IN A VOLUMETER

123 *Procedure.* Proceed as directed under Method 1 except that the mechanical tester provides  
 124 a fixed drop of  $3 \pm 0.2$  mm at a nominal rate of  $250 \pm 15$  taps per minute.

## 125 METHOD 3: MEASUREMENT IN A VESSEL

126 *Procedure.* Proceed as described under Method 3 for measuring the untapped bulk  
 127 density, using the measuring vessel equipped with the cap shown in Figure 2.  
 128 The measuring vessel with the cap is lifted 50-60 times per minute by the use of a suitable  
 129 tapped density tester. Carry out 200 taps, remove the cap and carefully scrape excess  
 130 powder from the top of the measuring vessel by smoothly moving the edge of a reclined  
 131 spatula blade across the top surface of the cup, taking care to keep the spatula tilted  
 132 backwards to prevent packing or removal of powder from the vessel. Determine  
 133 the mass ( $m$ ) of the powder to the nearest 0.1 per cent by subtracting the previously  
 134 determined mass of the empty measuring vessel. Repeat the procedure using 400 taps. If  
 135 the difference between the 2 masses obtained after 200 and 400 taps exceeds 2 per cent,  
 136 repeat the test using 200 additional taps until the difference between successive  
 137 measurements is less than 2 per cent. Calculate the tapped bulk density in grams per  
 138 millilitre using the formula  $m_f/100$  (where  $m_f$  is the final tapped mass of powder in  
 139 the measuring vessel). Replicate determinations performed on separate powder samples  
 140 are desirable. The test conditions, including tapping height, are specified in the expression  
 141 of the results.

## 142 Measures of powder compressibility

143 Because the interparticulate interactions influencing the bulk properties of a powder also  
 144 interfere with powder flow, a comparison of the untapped bulk and tapped bulk densities  
 145 can give an indirect measure of the relative importance of these interactions in a given  
 146 powder. Such a comparison is often used as an index of the ability of the powder to flow,  
 147 for example the compressibility index (Carr index) or the Hausner ratio.

148 The compressibility index and Hausner ratio are measures of the propensity of a powder  
 149 to be compressed as described above. Compressibility index:

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

150

$V_0$  = untapped bulk volume;

151

$V_f$  = final tapped bulk volume.

152 Hausner Ratio :

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

153

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