A new method for the measurement of meteorite bulk volume via ideal gas pycnometry

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[1] To date, of the many techniques used to measure the bulk volume of meteorites, only three methods (Archimedean bead method, 3-D laser imaging and X-ray microtomography) can be considered as nondestructive or noncontaminating. The bead method can show large, random errors for sample sizes of smaller than 5 cm^3 . In contrast, 3-D laser imaging is a high-accuracy method even when measuring the bulk volumes of small meteorites. This method is both costly and time consuming, however, and meteorites of a certain shape may lead to some uncertainties in the analysis. The method of X-ray microtomography suffers from the same problems as 3-D laser imaging. This study outlines a new method of high-accuracy, nondestructive and noncontaminating measurement of the bulk volume of meteorite samples. In order to measure the bulk volume of a meteorite, one must measure the total volume of the balloon vacuum packaged meteorite and the volume of balloon that had been used to enclose the meteorite using ideal gas pycnometry. The difference between the two determined volumes is the bulk volume of the meteorite. Through the measurement of zero porosity metal spheres and tempered glass fragments, our results indicate that for a sample which has a volume of between 0.5 and 2 cm³, the relative error of the measurement is less than ± 0.6 %. Furthermore, this error will be even smaller (less than $\pm 0.1\%$) if the determined sample size is larger than 2 cm^3 . The precision of this method shows some volume dependence. For samples smaller than 1 cm³, the standard deviations are less than $\pm 0.328\%$, and these values will fall to less than $\pm 0.052\%$ for samples larger than 2 cm³. The porosities of nine fragments of Jilin, GaoGuenie, Zaoyang and Zhaodong meteorites have been measured using our vacuum packaging–pycnometry method, with determined average porosities of Jilin, GaoGuenie, Zaoyang and Zhaodong of 9.0307%, 2.9277%, 17.5437% and 5.9748%, respectively. These values agree well with the porosities of fragments of which have been measured using the Archimedean bead method and 3-D laser imaging. This method also may be applied to the study of rare samples in other fields (e.g., archeology and geology).

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1. Introduction

[2] Porosity is a fundamental physical property of geological materials, which include meteorites. It reflects both the physical environment of formation and the subsequent evolution of the meteorite. Different porosities may influence the internal structure, gravitational field and impact dynamics of asteroids,

and moreover, may affect some other physical properties of asteroids to varying degrees; for example: seismic velocity, cosmogenic nuclide production rates, electric conductivity and so on [Britt et al., 2002]. Moreover, thermal conductivity of meteorite shows a linear correlation with 1/porosity [Opeil et al., 2012]. In planetary science, those interested in modeling crustal thickness require values for the density of the crust [e.g., Wieczorek and Phillips, 1998; Hikida and Wieczorek, 2007; Ishihara et al., 2009], that can be afforded through the study of meteorites. Physical property determinations of meteorites can also provide important constraints in calculations of lithospheric flexure, where both the densities of the crust and the load are variables [e.g., McGovern et al., 2002; Belleguic et al., 2005; Wieczorek, 2008]. The sizes of impact shock craters are largely dependent on the porosity of the target rocks [e.g., Ivanov, 2006]. While, on Mars, crustal porosity is a key factor in the study of subsurface aquifers [Clifford, 1993].

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Figure 1. The meteorite vacuum packaged with balloon.

[3] The study of porosities and densities of meteorites and other extraterrestrial rocks (including presumed meteoritic material representing rocks from Mars and the Moon), therefore, can provide evolutionary clues concerning their parent bodies. Consequently, there have been many studies aimed at the measurement of porosities and densities of meteorites, as well as of lunar rocks [*Consolmagno et al.*, 1998, 2006; Consolmagno and Britt, 1998; Flynn et al., 1999; Britt and Consolmagno, 2000, 2003; Wilkison et al., 2003; Sasso et al., 2009; Macke et al., 2010, 2011; Kiefer et al., 2012].

[4] To date, six methods have been used in the measurement of the bulk volume of a meteorite:

[5] 1. The meteorite is immersed in a certain liquid (such as water, toluene, carbon tetrachloride and isopropyl alcohol) of a known volume [Keil, 1962; Kukkonen and Pesonen, 1983; Terho et al., 1993]. This method, however, is not without problem. For example, surface tensional forces, air trapped within pore space, and fluid penetrated in cracks and pores can lead to much uncertainty in their results. Besides, this method may also lead to the contamination or alteration/ weathering of the meteorites.

[6] 2. The meteorite is cut directly into more easily measured shapes [e.g., *Yomogida and Matsui*, 1981]. Again, this method is a destructive one causing irreparable damage to the meteorite samples and may, in addition, create some cracks and fractures that weren't already present.

[7] 3. The sample is packed with clay of a certain volume into a known shape whose total volume is more easily determined [e.g., Matsui et al., 1980; Yomogida and Matsui, 1983]. However, this method may again contaminate the meteorite.

[8] 4. Three-dimensional laser imaging is used. This method can be used in measuring meteorites as small as 0.55 cm^3 , with a precision of better than 1% [e.g., *Herd et al.*, 2003; Smith et al., 2006; McCausland et al., 2011]. This method, however, is difficult to apply in the measurement of the bulk volume of a large number of meteorites, due to the cost,

time and effort involved in the measurements [Britt and Consolmagno, 2003].

[9] 5. X-ray microtomography also provides a 3-D image of the sample, but the instrumentation is very expensive and requires qualified operators [e.g., Friedrich et al., 2008; McCausland et al., 2010; Wittmann et al., 2011] to produce good results.

[10] 6. The Archimedean bead method is used. This is the most commonly used method for measuring meteorite bulk volume as it is considered neither destructive nor sample contaminating [Consolmagno and Britt, 1998; Britt and Consolmagno, 2003; Wilkison et al., 2003; Coulson et al., 2007; Sasso et al., 2009; Macke et al., 2010, 2011; Kiefer et al., 2012]; it is also cheap and relatively straightforward to do. The measurement error, with this method, is typically less than 2% when a large meteorite is under investigation, but errors will significantly increase as the size of the meteorite diminishes (e.g., volumes of $3-5$ cm³ or less [*Macke*] et al., 2010]). The measurement errors of meteorite smaller than $\overline{3}$ cm³, while never accurately ascertained, apparently will be too large to reflect the exact porosity of the sample. This raises the important question and problem we aim to address in this paper, that many meteorite samples are small, almost exclusively in the range of only a few grams and that typically have a volume of around 1 cm^3 , meaning they are typically outside of the capability of the Archimedean bead method.

[11] Hence, it is necessary to establish a rapid, nondestructive, noncontaminating, highly accurate and reproducible method for the measurement of the bulk volume of meteorites, particularly those of a small sample size. Based upon this situation, this paper introduces a high-accuracy method for measuring the bulk volume of meteorites, which overcomes the problems outlined above.

2. Method

[12] To determine the porosity of meteorite usually requires measurement of the bulk volume of a given meteorite and the grain volume. Grain volume can be determined accurately via ideal gas pycnometry, such as utilizing a Quantachrome Ultrapycnometer (accuracy <0.03%, repeatability <0.015%). Thus, the accuracy of porosity measurement for a meteorite depends principally on the accuracy of the bulk volume measurement of this sample. Here, we provide the following steps outlining the procedure to determine the bulk volume of meteorites with a relatively high accuracy.

[13] 1. A meteorite sample of a specific mass is placed into a high-quality, thin-walled balloon. The balloon neck is then attached to a vacuum pump and sealed so as to prevent atmosphere from penetrating back into the balloon. The vacuum pump is started and the air inside of the balloon evacuated, making the balloon fit closely to the surface of meteorite (Figure 1).

[14] 2. The balloon-packaged meteorite is put into sample chamber, where the volume (V_{m-b}) of the balloon-packaged meteorite and the encasing balloon as a whole can be determined, following the same process of the grain volume measurement, outlined above.

[15] 3. After the balloon-packaged meteorite is taken from the sample chamber of the pycnometer, we cut the balloon and extract the meteorite. Then, the total volume (V_{s-b}) of

Table 1. Results of Metal Spheres' Measurement

Sample	Actual Volume (cm ³)	Volume Overestimate $(\%)$
Case $1-1$	1.0729	-0.289
Case $1-2$	1.0729	0.065
Case $1-3$	1.0729	-0.401
Case $1-4$	1.0729	0.587
Case $1-5$	1.0729	0.084
Case $1-6$	1.0729	0.130
Case $1-7$	1.0729	-0.168
Average		0.001 ± 0.328
Case $2-1$	1.1689	0.265
Case $2-2$	1.1689	-0.128
Case $2-3$	1.1689	0.034
Average		$0.057 + 0.198$
Case $3-1$	2.1504	0.056
Case $3-2$	2.1504	-0.014
Case $3-3$	2.1504	0.033
Case $3-4$	2.1504	0.070
Case $3-5$	2.1504	0.140
Case $3-6$	2.1504	0.088
Average		0.061 ± 0.052

the broken balloon and a metal sphere with a known volume (V_s) is determined by putting these both together in the sample chamber.

[16] 4. The bulk volume of the meteorites (V_b) can then be calculated by use of

$$
V_b=V_{m-b}-\left(V_{s-b}-V_s\right)
$$

The uncertainties of this method mainly lie in the process of measuring V_{m-b} and V_{s-b} .

[17] In this study, the measurements of bulk volume and grain density were all made using a Micro-ultrapyc 1200e pycnometer, produced by the Quantachrome Company. The gas used was nitrogen. The target pressure of the each measurement is 1.34 bars (19.5 psig). The precision of the pycnometer is determined as ± 0.0001 cm³. The mass of the samples were measured on a BSA224S-CW (BT224S) Sartorius electronic analytical balance with an accuracy of ± 0.1 mg. The vacuum pump used in this study is a rotary vane pump. The flow rate of the pump is 1 cfm and the ultimate vacuum is 10 Pa.

3. Determination of Errors

[18] In order to evaluate the accuracy and precision of this method, zero-porosity material is needed to work as standards. This kind of sample has an identical grain volume to that of its bulk volume. Two groups of experimental samples were selected in this experiment, which include metal spheres with smooth surfaces and tempered glass fragments with irregular shapes and rough surfaces. Due to the size limitation of the sample chamber of the Micro-ultrapyc 1200e pycnometer, the volume of the experimental samples could not exceed 3 cm^3 . It can be predicted, however, that higher accuracy and more precise data can obtained if larger size samples (i.e., greater than 5 cm³) are used, coupled with a larger sample chamber.

3.1. Metal Spheres

[19] The volumes of three metal spheres used in this work are 1.0729 , 1.0775 and 0.0960 cm^3 , respectively. Three different combinations are measured during the experimental runs. In case 1 a single metal sphere with a volume of 1.0729 cm³ is used. For case 2 there are two spheres with volumes of 1.0729 and 0.0960 cm³. Spheres volumes of 1.0729 and 1.0775 cm³ comprise case 3. Following the four steps described in section 2, the three cases were measured seven, three and six times, respectively. The results of the various sample runs, measured by vacuum packaging-pycnometry method, are presented in Table 1. For the seven measurements of case 1, the relative errors ranged from -0.401% to 0.587% with an average of 0.001%, and a standard deviation of $\pm 0.328\%$. Case 2 was measured three times. The relative errors ranged from -0.128% to 0.265% and the average value is 0.057% with a standard deviation of $\pm 0.198\%$. Case 3 was measured six times. The relative errors ranged from -0.014% to 0.140% with an average value of 0.061% and standard deviation of ± 0.052 %. This method does, however, exhibit volume dependence (Figure 2): larger samples have a smaller standard deviation than smaller ones.

3.2. Tempered Glass

[20] Because fragments of tempered glass have complex shapes, which are quite similar to the shapes of many meteorites, these fragments were chosen for the purpose of establishing potential measurement errors in the analysis of meteorite samples. Fifteen samples (Figure 3), with volumes varying from 0.4948 cm^3 to 2.036 cm^3 (see Table 2), under identical experimental conditions to that used in measuring the metal spheres, were determined. The results (Table 2) indicate that all the relative errors of measurement of tempered glass fragments for volumes between approximately 0.5 cm³ and 2 cm³ are within ± 0.5 %. Figure 4 shows the actual volume and corresponding deviation of 15 samples measured after vacuum packaging. One can see that relative errors decrease as sample volumes increase. As in the case of the metal spheres, this indicates certain volume dependence for this method. For optimum results (within the limited size of the sample chamber of the pycnometer) a sample that closely matches that of the chamber should be chosen.

3.3. Measurement of Meteorites

[21] In order to compare the results of our new method and the literature data from other methods, bulk volumes of nine fragments from four ordinary chondrites (Jilin, GaoGuenie,

Figure 2. Volume discrepancy for the measurement of metal sphere.

Figure 3. Fifteen fragments of tempered glass used in measurement of actual volume and volume overestimates.

Zaoyang and Zhaodong) which vary in shape, size, and surface roughness were measured using ideal gas pycnometry following the procedures outlined in section 2. The grain density was also measured using the same pycnometer. The bulk densities and porosities of these samples were calculated and listed together with the grain densities, grain volumes and bulk volumes in Table 3. Some corresponding values of different fragments of the same meteorites, as determined using other methods are also listed in Table 3.

[22] The average bulk density for six Jilin fragments is $3.\overline{4586} \pm 0.0015$ g/cm³. This value is in agreement with the bulk density $(3.49 \pm 0.03 \text{ g/cm}^3)$ of a 33.56 g piece of Jilin measured by the Archimedean bead method [Macke, 2010], and slightly higher than the corresponding values (3.41 \pm 0.03 g/cm³, 3.398 g/cm³) of 62.35 g and 73.4 g fragments measured using the Archimedean bead method [Kohout et al., 2008; Beech et al., 2009]. However, Wilkison and Robinson [2000] give an average bulk density (measured again using the Archimedean bead method) for a 20.61 g fragment of Jilin

Table 2. Results of Tempered Glass Measurement

Sample	Mass (g)	Actual Volume (cm ³)	Volume Overestimate $(\%)$
T G-1	1.2147	$0.4866 + 0.0005$	-0.185
$TG - 2$	1.7898	$0.7215 + 0.0007$	0.083
$TG-3$	1.9345	$0.7845 + 0.0015$	-0.490
T G-4	2.2619	$0.9150 + 0.0008$	0.120
$TG-5$	2.3193	0.9392 ± 0.0006	-0.458
TG-6	2.3908	$0.9643 + 0.0017$	0.270
$TG-7$	3.0505	$1.2309 + 0.0021$	0.203
$TG-8$	3.1234	$1.2609 + 0.0012$	-0.357
$TG-9$	3.1488	$1.2702 + 0.0007$	-0.260
$TG-10$	3.1880	$1.2770 + 0.0008$	-0.031
$TG-11$	3.6167	1.4547 ± 0.0009	0.213
$TG-12$	3.7238	$1.4992 + 0.0005$	0.060
$TG-13$	4.1679	$1.6716 + 0.0013$	0.341
$TG-14$	4.7991	1.9332 ± 0.0006	-0.062
$TG-15$	5.0649	2.0367 ± 0.0007	0.093

bulk density is somewhat higher $(\sim 3.5\%)$ than the bulk density measured via our method. The average grain density of the six fragments of Jilin is 3.7910 ± 0.0298 g/cm³, this value agrees very well with the grain density $(3.78 \pm$ 0.04 $g/cm³$) as determined by *Beech et al.* [2009] for a 62.35 g fragment of Jilin. The porosities of the six fragments of Jilin ranged from 7.8620% to 11.3715% (average 9.0307 \pm 1.2954). These values also agree well with the porosities of fragments of Jilin as measured using the Archimedean bead method [Kohout et al., 2008; Beech et al., 2009].

as 3.58 ± 0.06 g/cm³ [*Wilkison and Robinson*, 2000]. This

[23] Only one 3.5107 g fragment of GaoGuenie has been measured in this study, the grain density, bulk density and porosity are 3.6893 ± 0.0050 g/cm³, 3.5813 ± 0.0080 g/cm³ and 2.9277%, respectively. The bulk density measured using our method is only 1.9% lower than the bulk density measured using 3-D laser imaging method; this also was using a small fragment with a volume of merely 1.19 cm³. The bulk

Figure 4. Volume discrepancy for the measurement of tempered glass.

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^aThe range of the values had been averaged.

^bThe mass range of 14 fragments of GaoGuenie meteorite. aThe range of the values had been averaged. bThe mass range of 14 fragments of GaoGuenie meteorite.

density and porosity measured using our method are in the range of corresponding values measured by the method of immersing samples in fluid [Beech et al., 2009]. The bulk density of a 21.0 g piece of GaoGuenie reported in Macke's doctoral study [*Macke*, 2010] is 3.55 ± 0.005 g/cm³, which is in agreement with our value.

[24] A 3.1769 g fragment of Zaoyang meteorite has been measured using our modified bead method. The bulk density, grain density and porosity are 3.1721 ± 0.0069 g/cm³, 3.8472 ± 0.0612 g/cm³ and 17.5437%, respectively. The grain density and bulk density are all slightly higher than the corresponding values reported by Macke [2010], though the porosity is comparable.

[25] Different from the three H5 ordinary chondrite meteorites outlined above is the Zhaodong meteorite; it is a L4 ordinary chondrite. For this meteorite we measured a 5.0099 g sample. The bulk density, grain density and porosity are determined as 3.3938 ± 0.0069 g/cm³, 3.6095 ± 0.0069 0.0017 g/cm³ and 5.9748%, respectively. Compared with the corresponding values measured by Macke [2010], the bulk density obtained by our method is slightly higher, which also made the porosity slightly higher.

4. Discussion

[26] The measurements of metal spheres and tempered glass indicate that vacuum packaging-pycnometry method is a simple, nondestructive and reliable method for measuring the bulk volume of meteorites and other rock. The test of metal spheres indicates that the method exhibits certain volume dependence. The larger samples have higher precision (see Figure 2). The measurement of 15 tempered glass fragments also shows a tendency of volume dependence of this method.

[27] Noncontaminating techniques are very important in the determination of the physical properties of meteorites, many of which are located in museums and are of exceptional value to science. While our method is essentially one that is noncontaminating, in this study, latex balloons that can be purchased "off the shelf" at any store were used. As such, it is possible that outgassed hydrocarbons, etc., from the latex could have been forced into the meteoritic sample during the evacuation process. For those researchers interested in organic chemistry in meteorites, such contamination might not be negligible. In order to avoid this problem, curation-grade balloons should be used in meteorites bulk volume measurement. One feature that was not closely examined in our studies is the influence that surface roughness of a meteorite may have on the measurement of bulk volume. This is something that could be looked at in the future.

[28] While it is unfortunate, the fragments of Jilin, GaoGuenie, Zaoyang and Zhaodong measured in this study are not the same fragments determined previously by other scientists; because of this, our results may reflect not only differences in measurement accuracy and precision for the chosen method but also slight differences in determined meteorite physical properties. For example, Beech et al. [2009] noted that there is variability in determined meteorite physical properties (bulk density, grain density and porosity) for the H5 chondrites GaoGuenie and Jilin. As such, our results (that include another H5: Zaoyang) provide additional evidence the variation in the bulk density values applicable to multiple fragments from the same meteorite fall. What is interesting and important, moreover, is that we can achieve meaningful results for the density and porosity of small fragments of meteorites and other geological materials (i.e., those samples of less than 1.5 cm^3 in bulk volume) through this nondestructive and simple method.

5. Conclusions

[29] Measurement of the bulk volume of meteorites with the pycnometer-balloon vacuum packing method can allow for the collection of high accuracy and precise results for small meteorite samples. When the vacuum packagingpycnometry method is used to measure a sample with a volume of between 0.5 and 2 cm^3 , the relative error will less than 0.6%, and much less (i.e., below 0.1%) if the method is used to measure a sample larger than 2 cm^3 in size. It is clear that relative errors decrease as the samples' volume increases. This new method is quite efficient in measuring samples smaller than 5 cm^3 , which cannot be achieved when using the Archimedean bead method. Because of limitations in the sample chamber size of Micro-ultrapyc 1200e pycnometer, samples larger than 3 cm^3 could not been measured. However, we have reason to believe that the samples which are larger than 3 cm³ will have an even greater accuracy and precision for porosities determinations in this way. The measurements of bulk volumes of Jilin, GaoGuenie, Zaoyang and Zhaodong fragments indicate that the method is suitable as a nondestructive and noncontaminating test for small meteorites. As with the Archimedean bead method, this method can be applied in the study of small, valuable materials in other fields, such as archeology, geology, etc.

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