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Article

# Accurate Density Determination of Various Non-Porous Natural Stones Employing Archimedes' Principle and a New Non-Destructive Liquid Pycnometer for Natural Stones

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## Abstract

In this study we explore various non-destructive methods for the determination of density of 27 non-porous natural stones. Among the methods investigated the most accurate method was found to be the mass-based suspension method that uses Archimedes principle, with costs of equipment less than 20\$. We have used this density measurement method to measure densities of natural stones and copper reference cube in the range of 1.07 – 8.93 g·cm<sup>-3</sup>, for stones that have volumes less than 16.4 cm<sup>3</sup>. The measurement are in excellent agreement with more precise methods that use a 4 decimal place analytical balance. The measurement uncertainty of the method was assessed with a Cu density reference cube and was found to be of the order of 0.1% in measuring the volume of stones with arbitrary shape. Finally, we provide details of the design features of a new liquid-based pycnometer that can measure the density of irregular shape natural stones without the need to form a powder. This pycnometer can also be used to measure density changes in liquids as a function of temperature and solute concentration.

**Keywords:** density; natural stones; level-method; overflow method; mass-based suspension method

## 1. Introduction

Natural stones are used in jewellery, as souvenirs, for decoration and are thought to have metaphysical and healing properties. They are favourite items of collectors and have high market values. Also natural stones with larger dimensions are used as kitchen countertops (e.g. granite, marble, quartzite, soapstone, slate, and limestone) and their density is an important parameter in determining their durability, mechanical properties and therefore market price.[1–3] Most natural stones are not single crystals but rather an aggregate of smaller crystal particles (i.e. grains) and are therefore polycrystalline.[4] Some natural stones are mixtures of grains of different crystalline materials (e.g. granite composed of feldspar, quartz, mica, and amphiboles).[5,6] Also some natural stones are not even crystalline but rather amorphous (e.g. obsidian, opal, and moldavite) so lack a regular arrangement of atoms in a lattice.[7–9] For this reason their structural determination by means of crystallography (i.e. X-ray, neutron diffraction) is sometimes challenging which is the typical method used in mineralogy to determine the density of single crystals.[10–13] It is therefore useful to report the densities and morphology of some common natural stones found in museums and souvenir shops as a means of their identification for collectors and merchants of these stones. Furthermore, it is useful to explore the various methods and techniques to measure the density of

natural stones to quantify which method has the highest precision-to-cost ratio. This approach to evaluate instrumentation in chemistry has not been previously employed and may become a useful parameter in instrument procurement by non-specialised labs.

The concept of density of a material is of high importance in many fields and is therefore thought early on in education. However, to quantify the density of materials is taught at university level courses in chemistry, physics, engineering, materials science, and many other disciplines. Also, the density of materials is assessed in many commercial settings to assess the quality of a material. For example, denser materials have more chemical bonding between the atoms of the material, therefore are expected to have enhanced mechanical properties (e.g. Young's modulus, shear modulus, strain modulus). It also indicates that there is a smaller number of defects in the structure which can affect chemical and mechanical properties of the material. Lastly, for allotropes it is a measure of the void space in a material which is an important parameter in materials design as it indicates the available void space within a material that is available for introducing dopants.

The density of material is defined by the mass of the material divided by the volume of the material. The mass is easier to measure using a scale, however, there has been considerable effort to obtain an accurate measurement of the volume of an object. The volume of a stone can be accurately measured if the stone has a geometric shape (e.g. sphere, cube) but in the case of stones with an arbitrary shape, the volume has to be measured either by submerging the stone into a liquid and measuring the volume of the displaced liquid or by measuring pressure changes in a gas (e.g. Helium), when a known volume of it is introduced inside a closed chamber, that contains the stone, with the use of Boyle's law (i.e.  $P_1V_1 = P_2V_2$ ). In this study we use water as the fluid to measure the volume of twenty-seven natural stones. Water is non-toxic compared to other liquids such as mercury, which has also been used for volume measurements of solids via the displacement method. Therefore it is safe to be used within a teaching lab as well as in educational settings.

There are also digital/optical methods of calculating the volume of a 3D object (e.g. 3D photogrammetry, 3D laser scanning and micro-CT) but none of these can currently approach in accuracy the mass-based method. 3D photogrammetry uses photographs of the object at various angles to reconstruct the shape of the object. [14] This technique has a higher cost (~\$3000), and it has limitations on the size of the object that can be scanned and its transparency. It uses software such as Meshlab (which calculates the volume of the object by summing triangular pyramids that are fitted within the volume of the object). This technique has accuracies of estimating the volume of about 5%.

A more recent technique, 3D laser scanning is generally more accurate than 3D photogrammetry because it uses a laser beam to calculate the distance between a laser beam source and the surface of the object. [15] These distance measurements generate a point cloud that then is used to calculate the volume, which can be as accurate as 2%. The instruments for this technique can cost (\$500-\$3000).

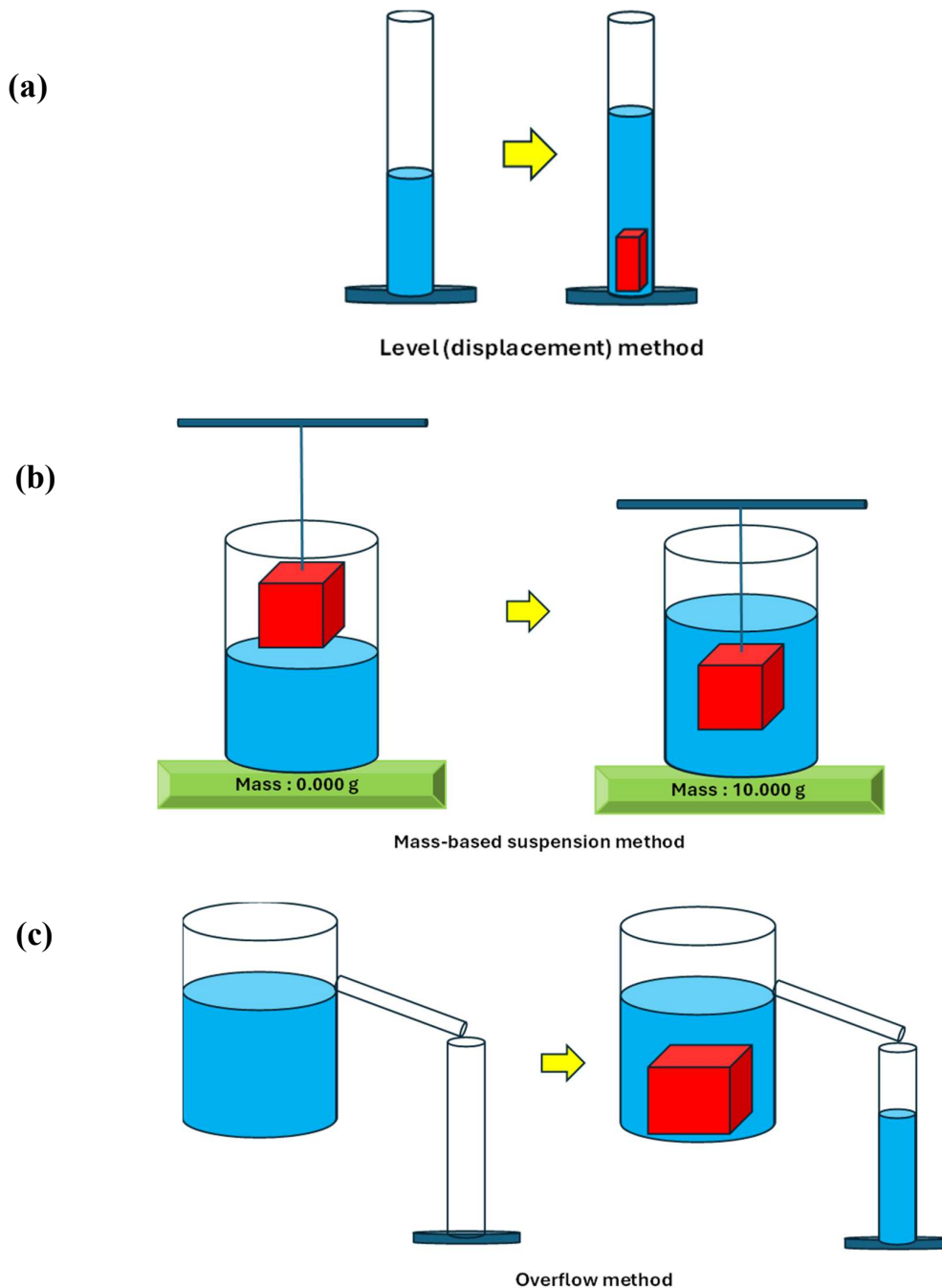
The best and most expensive technique to scan the volume of stones among the digital/optical methods is micro-CT which uses X-ray images (radiographs) and rotates the object through 360 degrees and then uses the 2D images to digitally reconstruct a 3D volume. This technique has the advantage of being able to reveal the internal and external structure of an object so it will take into account if there is porosity within the micrometre range within the stone [16] and has accuracy of the volume to about 1%. This technique is expensive with some instruments sold in the range of \$200,000 to over \$1,000,000 and is therefore only used in specialised labs.

In this paper we evaluate various inexpensive yet accurate empirical methods to evaluate the density of natural stones. This is particularly important for educators that would like to assess this physical parameter using inexpensive equipment in activities that enhance experiential learning. Also, collectors can use our method to measure the density of various natural stones in combination with their morphological examination. In addition, a calibration protocol is outlined, and how to calculate the accuracy and precision of these techniques. Finally, we propose a design of a new chemistry glassware that can determine the density of natural stones in a non-destructive way using the displacement of fluids in a thin, transparent tube with well-defined geometric parameters. This

glassware can also be used to study density changes in liquids as function of temperature and solute concentration, in addition to density changes due to the interaction of liquids with solids.

## 2. Materials and Methods

Typically, there are three methods that the density of a material can be measured in chemistry, physics, engineering and materials science. The first method is the level method (i.e. displacement) method. The second method is the overflow method, and the third method is the mass-based suspension method that employs Archimedes' principle to measure the volume of a stone. These three methods are depicted in the following simplified schematic.



**Scheme 1.** Simplified schematic of the (a) level (displacement) method, (b) overflow method and (c) mass-based suspension method used to determine the density of natural stones used in this study.

Archimedes' principle states that the buoyant force of a partially or fully submerged object in a fluid is given by

$$F_b = \rho \cdot g \cdot V \quad (1)$$

Where  $F_b$  is the buoyant force,  $\rho$  is the density of the liquid,  $g$  is the acceleration due to gravity and  $V$  is the volume of the submerged object or the volume of the displaced fluid. This force is exerted onto the scale due to Newton's third law (action-reaction law) when the stone is submerged into water (see Scheme 1(c)).

So, the mass measured on the scale is

$$m_{scale} = \frac{F_b}{g} \quad (2)$$

Combination of equations 1 and 2 yields that the mass measured on the scale is

$$m_{scale} = \rho \cdot V_{stone} \quad (3)$$

And since the fluid we used was water at 20 °C which has a density of 0.9982 g·cm<sup>-3</sup> the volume of the stone is given by,

$$V_{stone} = \frac{m_{scale}}{\rho_{water}} \quad (4)$$

Lastly the density of the stone is calculated by measuring the mass of the stone ( $m_{stone}$ ) on the scale and dividing by the volume of the stone ( $V_{stone}$ ) measured using Archimedes principle, given by

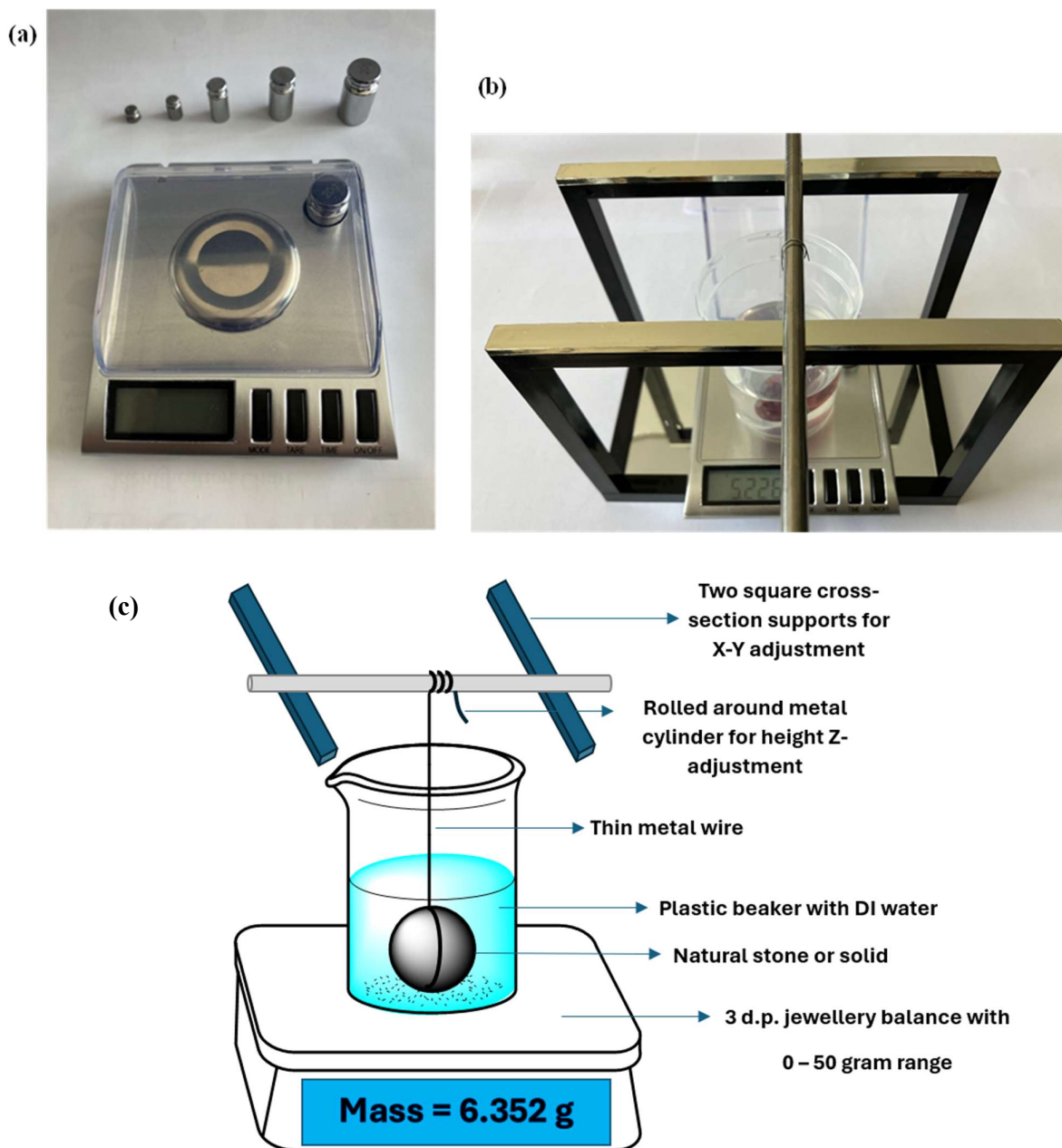
$$\rho_{stone} = \frac{m_{stone}}{V_{stone}} \quad (5)$$

For these methods we have used an inexpensive jewellery scale (~\$15-20), which measures a maximum mass of 50.000g with a precision of 0.001g (see Figure 1) according to the manufacturer. The accuracy of the scale was assessed with a calibration set chrome plating scale weights set that included the following weights: 1g, 2g, 5g, 10g, 20g and 50g, shown in Figure 1(a). The experimental setup for the mass-based suspension method using the jewellery scale is shown in Figure 1(b). In this, the natural stone is suspended from a horizontal beam using a 0.20 mm in diameter metal wire, measured with a digital metal caliper with an accuracy of 0.01 mm. The length of the wire that is submerged in the liquid is about 5 cm, which results in an error of the volume of the submerged stone of 1.57×10<sup>-3</sup> cm<sup>3</sup>. This corresponds to an error in the determination of the volume of the natural stone using Archimedes' principle that is of the order of the accuracy (1×10<sup>-3</sup> g) of the jewellery scale. Using this method, the natural stone is preserved as there is no requirement to drill a hole to suspend the stone by other means or to make a powder of the stone. This method can also be used to support stones of any shape and a large range of masses, depending on the range of the scale, making the method very versatile and applicable to a broad range of sample dimensions and densities. During the measurement, the natural stone is fully submerged in deionized water, without touching the bottom or the side walls of a 120 ml capacity polypropylene (PP) sample vial, without a lid. The use of a plastic rather than a glass beaker is to reduce the mass exerted on the jewellery scale, which has a measuring range of 1-50 g, which enhances the mass range of natural stone specimens that can be measured. Also, the size of the sample vial is such that it fits exactly on to the measuring platform of the jewellery scale. With this experimental setup we were able to measure the density of natural stones that had volumes smaller than 10 cm<sup>3</sup>, which is the typical size of natural stones commercially available.

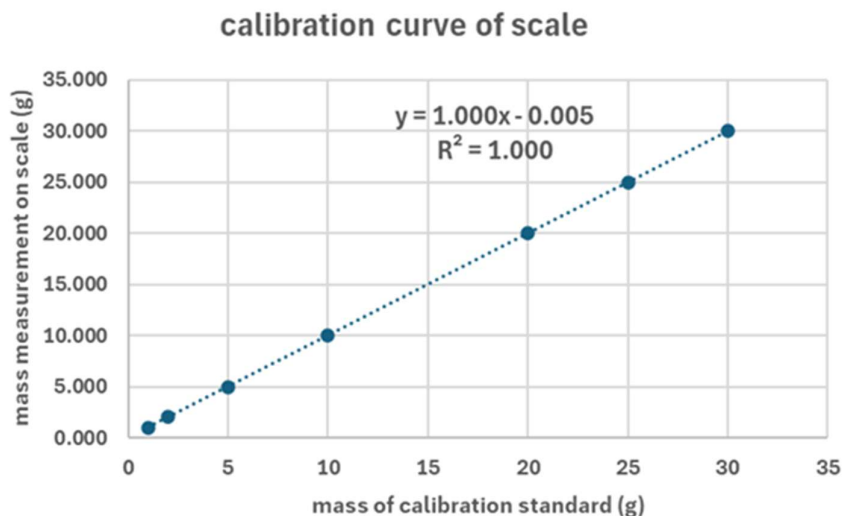
To test the accuracy of the mass-based suspension method using a three d.p. jewellery scale we have also measured the densities with four d.p. accuracy using a closed system and an analytical balance (Fisher analytical series FAS224/E). For these measurements we have used deionised water 18.2 MΩ·cm (Elga – Pure lab chorus). The pressure inside the lab was about 759 Torr, measured with an analogue barometer. The pH of deionised water was seven within the time of the experiment (10-15 sec) and the temperature of water, once equilibrated with the lab temperature, was 21.9 °C. The rate of evaporation of water inside the lab was slightly higher than under ambient conditions and was of the order of 0.0005 g·min<sup>-1</sup>.

Therefore, all measurements with the mass-based suspension method were made within 10-15 sec, and this was possible as the use of the metal wire made the adjustment of height and lateral position of the stone very fast, minimising therefore the loss of water mass due to evaporation. Additionally, we used a closed system to minimise the effect of water evaporation. The measurements of densities using a four d.p. analytical balance within a lab environment were only slightly affected by the negative pressure (-0.1 Torr). The agreement between the method developed in this manuscript using the jewellery scale and the more accurate analytical balance measurements were found to be in excellent agreement for the twenty-seven natural stones, with an average percent error of 0.3% error, between the two methods. Also, the uncertainty of the density measurement with the mass-based suspension method due to the error in the determination of the volume with a jewellery balance is of the order of 0.1% error, which was assessed with a copper density reference cube ( $d = 2.54$  cm and 99.95% purity).

The systematic error of the jewellery balance was corrected with the use of a linear calibration curve using standard weights within the measured range (0-30 g) of the balance. The calibration curve of the jewellery scale (see Figure 2) showed that the correlation between chrome-plated standard masses and the measured mass on the scale is perfectly linear ( $R^2 = 1.000$ ) within the range of 1 – 30 g. Also, the equation of the trendline from linear regression suggests that this correlation between theoretical and empirical mass is linear with a small correction factor of 0.005 g, which must be added to the measured masses on the jewellery scale to take into account the systematic error of the jewellery balance. We note that when this correction is employed via calibration of the jewellery scale, this instrument can be as accurate as other 3 d.p. analytical balances that have much higher costs. Additionally, the maximum standard deviation observed in the mass measurements is 0.002 g with an average value of the standard deviation of all measurements of 0.0004 g. This suggests that the jewellery scale is accurate to  $0.001 \text{ g} \pm 0.0004 \text{ g}$  once the correction factor of 0.005 g has been added to every individual mass measurement. On this scale the calibration of the mass was initially done at 20 g and 40 g using the standard weights that were supplied with the scale. However, to evaluate the accuracy of this calibration method we have used chrome plating scale weights from a different supplier to obtain the calibration curve data which are presented in Table 1. The temperature was stable throughout the experiment. Also the use deionised water rather than distilled water is a more accurate option as dissolved ions would alter the density of water, which would affect the volume measurement of this method. The instrument drift in measured masses is negligible under ambient conditions but at negative atmospheric pressure inside a lab, there is a drift in the measured mass which is dictated by the evaporation rate of water. So the suggested method for inexpensive density measurements of natural stones is appropriate for ambient conditions of pressure, temperature and humidity.



**Figure 1.** (a) Jewellery scale with 3 d.p. accuracy and a set of chrome plating scale weights used for calibration of the scale. (b) Experimental setup for the suspension method determining density. (c) Simplified figure of the mass-based suspension method with the jewellery balance to determine the densities of natural stones (Figure partially drawn in ChemDraw professional).



**Figure 2.** Calibration curve of jewellery scale with chrome scaling weights.

**Table 1.** Calibration curve data for the measurement of mass between 0-30 g on a jewellery scale along with mean value of the mass and standard deviation reported for each measurement.

mass of calib. stand. (g)	Mass measurement on scale			Mean (g)	Stand. (g)
	measur. 1 (g)	measur. 2 (g)	measur. 3 (g)		
1	0.996	0.995	0.996	0.996	0.001
2	1.996	1.995	1.993	1.995	0.002
5	5.000	5.000	5.000	5.000	0.000
10	9.995	9.994	9.993	9.994	0.001
20	20.000	20.000	20.000	20.000	0.000
25	25.006	25.006	25.006	25.006	0.000
30	30.000	30.000	30.000	30.000	0.000

\* the mass of 25g was 20g standard plus 5g standard and the mass of 30g was the 20g standard

The dimensions of these natural stones were such that the overflow method was not found to be an accurate method for the determination of densities of natural stones with volumes less than 10 cm<sup>3</sup>. We find that the accuracy of the overflow method can be improved by measurement of the mass of the overflowed water on the jewellery scale rather than the volume of the overflowed water in a graduated cylinder as shown in Scheme 1(b). The overflow method was evaluated with two commercially available overflow vessels shown in Figure 3. One was a 500 ml capacity borosilicate displacement vessel and the second a 500 ml tin displacement vessel, and the volume of the stone was measured by measuring the mass of the displaced water collected in a beaker placed on the jewellery scale. One advantage of using the overflow vessel was that we could measure the volume of larger natural stones, whereas with the graduated cylinder only small in dimensions specimens could be measured. In general, the use of the displacement vessel gave more accurate readings for the volume, especially when the mass of the displaced water was measured. One drawback of the borosilicate displacement vessel was that the side tube inner diameter was 4.8 mm and therefore some displaced water would remain in the glass tube. We have therefore used the tin displacement

vessel with a side tube inner diameter of 11.5 mm to report volume of stones when the stones had volumes that exceeded 10 cm<sup>3</sup>. However, these results are not reported due to errors introduced using the overflow method due to the small volume (< 10 cm<sup>3</sup>) of the natural stone specimens examined in this study. We will later provide the design details of a liquid pycnometer that avoids any inaccuracies due to water retention in the overflow tube, due to water capillary effect, and its tendency to adhere strongly to glass.

The third method we used to measure the density of these natural stones was the level (i.e. displacement) method (Scheme 1(a)). In this method the density is determined by changes in the height of the water level inside a graduated cylinder made from borosilicate glass. In this cylinder water forms a meniscus at the surface, but we have also examined the use of graduated cylinders by Clementoni which are made from a plastic in which water has a flat surface due to the absence of strong adhesive forces of water with the cylinder walls. This makes the measurement of the height of water more accurate and precise as there is an absence of a meniscus, the height of which may be difficult to observe. For consistency among the measured volumes in the level method the volume of most natural stones was measured using a 100 ± 1 ml graduated cylinder (here we are able to measure volumes to within 0.5 ml by observation of the meniscus that is in-between two lines) in which deionised water was poured and the bottom of the meniscus of water was observed. We have also used a 250 ± 2 ml graduated cylinder for the volume measurement of one stone with larger dimensions. However, this method was not found perfectly accurate in the determination of the volume of natural stones, as there was a great uncertainty in the measurement of the water level in the graduated cylinder. So, this method was only used for one natural stone that had larger dimensions and therefore could not be measured with the jewellery scale.

The natural stones for which we determined the density is given in Table 2, along with their common name, their dominant chemical formula, and the mineral name used commonly in mineralogy.

**Table 2.** List of label, chemical formula and name of natural stones investigated for their density.

Label	Name (common)	Correct mineral name	Dominant formula	Key citation
a	Carnelian	Chalcedony (var. of Quartz)	SiO <sub>2</sub> (with Fe <sup>3+</sup> inclusions)	USGS Quartz entry [17]
b	Clear quartz	α-Quartz	SiO <sub>2</sub>	[17]
c	Blue banded agate	Blue lace agate (Chalcedony)	SiO <sub>2</sub> + Al-Fe-Mn oxides	[18]
d	Nummite	Amphibole mix (Anthophyllite-Gedrite)	(Mg,Fe) <sub>7</sub> Si <sub>8</sub> O <sub>22</sub> (OH) <sub>2</sub>	[19]
e	Snowflake obsidian	Volcanic glass with cristobalite spherulites	Amorphous SiO <sub>2</sub> + Al <sub>2</sub> O <sub>3</sub> + FeO	[20]
f	Malachite	Malachite	Cu <sub>2</sub> CO <sub>3</sub> (OH) <sub>2</sub>	USGS Malachite [21]
g	Rose quartz	Rose Quartz	SiO <sub>2</sub> (Ti-Fe-Mn impurities)	[17]
h	Nephrite jade	Actinolite-Tremolite solid solution	Ca <sub>2</sub> (Mg,Fe) <sub>5</sub> Si <sub>8</sub> O <sub>22</sub> (OH) <sub>2</sub>	[22]
i	Petrified wood	Silicified wood (Quartz + minor calcite)	SiO <sub>2</sub> ± CaCO <sub>3</sub>	[7]

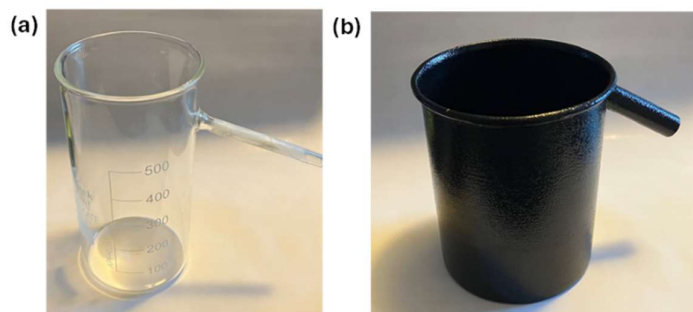
j	Brecciated jasper	Jasper (opaque Quartz)	SiO <sub>2</sub> with Fe-oxide cement	[18]
k	Dalmatian jasper	Igneous rhyolite (spotty quartz-feldspar)	(Na,K)AlSi <sub>3</sub> O <sub>8</sub> + SiO <sub>2</sub>	[23]
l	Turritella agate	Fossiliferous Chalcedony	SiO <sub>2</sub> + CaCO <sub>3</sub> shells	[24]
m	Aquamarine	Beryl	Be <sub>3</sub> Al <sub>2</sub> Si <sub>6</sub> O <sub>18</sub>	USGS Beryl [25]
n	Green aventurine	Quartz with fuchsite mica	SiO <sub>2</sub> + K(Al,Cr) <sub>2</sub> (Si <sub>3</sub> AlO <sub>10</sub> )(OH) <sub>2</sub>	[26]
o	Pink agate	Dyed Chalcedony	SiO <sub>2</sub>	[18]
p	Brecciated jasper	seej	SiO <sub>2</sub>	[18]
q	Opalite (man-made)	Opalite glass	SiO <sub>2</sub> ·nH <sub>2</sub> O (amorphous)	[27]
r	Yellow ocean jasper	Orbicular Chalcedony	SiO <sub>2</sub> + Fe-oxides	[18]
s	Fuchsite	Fuchsite	K(Al,Cr) <sub>2</sub> (Si <sub>3</sub> AlO <sub>10</sub> )(OH) <sub>2</sub>	[26]
t	Orange moonstone	Orthoclase feldspar	(Na,K)AlSi <sub>3</sub> O <sub>8</sub>	[28]
u	Shiva lingam	Crypto-crystalline Quartz/Hematite	SiO <sub>2</sub> + Fe <sub>2</sub> O <sub>3</sub>	[29]
v	Amber	Fossilised tree resin	(C <sub>10</sub> H <sub>16</sub> O) <sub>n</sub>	[30]
w	White quartz	α-Quartz	SiO <sub>2</sub>	[17]
x	Yellow fluorite	Fluorite	CaF <sub>2</sub>	USGS Fluorite [31]
y	Green fluorite	Fluorite	CaF <sub>2</sub>	[31]
z	Blue fluorite	Fluorite	CaF <sub>2</sub>	[31]
aa	Red jasper	Hematitic Jasper	SiO <sub>2</sub> + Fe <sub>2</sub> O <sub>3</sub>	[18]

### 3. Results and Discussion

In general, the measurement of mass is more accurate than the measurement of volume. Every lab nowadays has four d.p. analytical balance with can measure masses to within  $\pm 0.0001$  g. There are also specialised labs that use  $\pm 0.000001$  g microbalance which achieve even higher accuracy. This is the reason that for example pipettors used in chemistry, biology and biomedical labs are calibrated using a mass-based method, where the mass of water is measured rather than the volume and then using the equation  $V = m / \rho$  (where V is the volume,  $\rho$  the density of water and m the mass of water) the accuracy of the pipettor can be estimated and also calibrated in some pipettors.[14]

The measurement of volume by displacement of a fluid in graduated cylinder is less accurate as a 100 ml graduated cylinder (i.e. volumetric cylinder) has an accuracy of 1 ml. This means that a rock of a typical natural stone which has a volume of 5 - 10 cm<sup>3</sup> there will be a 10 - 20% error in the volume determination by use of the water displacement method. It has therefore been suggested that a mass-

based method where the mass of the displaced water is measured using an overflow vessel is more accurate and precise (see Figure 3). [32]



**Figure 3.** Picture of commercially available (a) borosilicate overflow vessel (500 ml) and (b) tin overflow vessel (500 ml) that can measure the volume of an object with irregular shape by water overflow and collection of the water in a graduated cylinder or in a beaker that is on a scale.

Another study has compared the level (i.e. displacement method) to the overflow and suspension method based on Archimedes' principle and found that the latter is the most accurate among the three methods in determining the volume of an object for density determination.[33] The % errors reported in this study for volume determination were  $1.7 \pm 8.5\%$  for the level (i.e. displacement) method,  $0.09 \pm 3.0\%$  for the overflow method and  $0.04 \pm 0.43\%$  for the suspension method.[33]

Natural stone densities vary between  $1.05 \text{ g/cm}^3$  for amber to  $19.32 \text{ g/cm}^3$  for gold. This suggests that their density in combination with morphological characterisation (colour, patterns and shape of natural stones) useful for collectors and educators that seek a quick method to determine the kind of natural stone and as an activity in the chemistry/physics/materials science lab. We therefore determine accurate densities and provide high-clarity pictures of the morphological characteristics of a collection of natural stones shown in Figure 4.

### 3.1. Densities of Natural Stones

We find that the water overflow method using a 500 ml borosilicate or tin overflow vessel gives inconsistent results for displaced water due to the small volume ( $< 10 \text{ cm}^3$ ) of the natural stones studied. These results are not reported because for smaller volume stones (e, g, n, l, j, k, l, m, n, p, r, s, t, v, w) this method did not result in any overflow. Currently, the availability of 100 ml overflow vessels, which would have been more appropriate for the volume of the natural stones measured here, is not commercially available. The only natural stone that gave reliable results with the overflow method was specimen w which had a volume of  $16.1 \text{ cm}^3$ . Also, some natural stones seemed to just change the surface tension of water (or even the density) and the additional volume of the natural stone had no effect on the height of the water meniscus, resulting in a lack of overflow when the natural stone was added to the liquid. This is definitely an area that could undergo further investigation as the change of the density of a fluid due to the interaction with a solid, have not been previously reported. Such investigations may become possible with the new liquid pycnometer we offer in the last section of this work.





**Figure 4.** Various natural stones for which the densities were determined in this study. (a) Carnelian, (b) Clear quartz, (c) Blue banded agate, (d) Nummite, (e) Snowflake obsidian, (f) Malachite, (g) Rose quartz, (h) Nephrite jade, (i) Petrified wood, (j) Brecciated jasper, (k) Dalmatian jasper, (l) Turritela agate, (m) Aquamarine, (n) Green aventurite fluorite, (o) Pink agate, (p) Brecciated jasper, (q) Opalite, (r) Yellow ocean jasper cabochon, (s) Fuchsite, (t) Orange moonstone, (u) Shiva lingam, (v) Amber, (w) White quartz, (x) Yellow fluorite, (y) Green fluorite, (z) Blue fluorite, (aa) Red jasper.

The level method (i.e. water displacement method) gave relatively accurate results for the small stones with an average percent error given by the following equation,

$$\text{average \% error} = \frac{1}{n} \sum_i^n \frac{|\rho_1 - \rho_2|}{\rho_2} \cdot 100 \quad (6)$$

where  $n$  is the number of measurements,  $\rho_1$  the density of the natural stone measured with the less accurate method and  $\rho_2$  the density measured with the more accurate method.

The percent error of the mass-based suspension method using the three d.p. jewellery scale was calculated using the following equation,

$$\% \text{ error} = \frac{|\rho_1 - \rho_2|}{\rho_2} \cdot 100 \quad (7)$$

where  $\rho_1$  is the density measured with the mass-based suspension method and  $\rho_2$  the density of 99.95% purity Cu reference cube, at 300K and 1 bar pressure.

Using equation 1 we can assess by what degree density measurements of the level method differ from the mass-based suspension method. This average % error was found to be 9.3% for natural stones in the range of 2 - 50 g. The level method also requires a volumetric cylinder in addition to the jewellery scale whereas the mass-based suspension method requires only a plastic beaker, the jewellery scale, and a small diameter wire. We therefore are in agreement with the earlier study by W. H. Stephen [33] that the mass-based suspension method is the most accurate among the three methods in determining the density of objects with irregular shape and this is due to the higher accuracy of mass measurement compared to volume measurement by the instruments used in this study.

As the measurements of the volume of the Cu (d = 1 inch) reference cube revealed that the suspension method had an inherent error of the order of 0.1% which is due to the inaccuracies in the measurement of the volume of the object in water. So, for a natural stone with 5 cm<sup>3</sup> volume the error in the volume measurement would be 0.005 cm<sup>3</sup>. This suggest that densities with this method with the jewellery scale should be reported up to two d.p.. Densities using the level method were reported to two d.p. as we could read volumes that have a precision of 0.5 ml on the 100 ml graduated cylinder. To confirm the validity of our density measurements, we have also measured the densities of the various natural stones using a four d.p. analytical balance in a closed system, and we report these densities as reference values of the natural stones examined.

In Table 3 we tabulate the measured densities for the level method, the mass-based suspension method using a three d.p. jewellery scale and the mass-based suspension method using the four d.p. analytical scale. The agreement of the two later methods is 0.3% (average % error) based on the average percent error which indicates that the density measurement protocol developed is accurate even with the use of a relatively inexpensive jewellery balance.

Polycrystallinity is expected to reduce the density of natural stones compared to the density of single crystals. For most specimens, the main crystal structure was amorphous silicon dioxide, which is known to have a lower density than quartz; however, depending on the second phase in the solid, some stones that were primarily silica (SiO<sub>2</sub>) had higher densities compared to  $\alpha$ -quartz. We aim to explore this aspect in a subsequent study more detailed study in which also temperature effects could be addressed using the new liquid pycnometer for which we offer the design details.

**Table 3.** Label, measured mass, average measured mass (av. m), standard deviation ( $\sigma$ ) of measured mass and calculated density of the various natural stone specimen listed in Table 2 using the level method ( $Q_1$ ) and the mass-based suspension method using the jewellery scale ( $Q_2$ ) and the mass-based suspension method using the analytical 4 d.p. balance ( $Q_3$ ). Values are also compared to literature values for the density ( $Q_{lit}$ ). The picture of the various natural stones is given in Figure 4.

Label	$m_1$	$m_2$	$m_3$	av. m	$\sigma$	$V_{stone}$	$m_{stone}$	$Q_1$	$Q_2$	$Q_3$	%	
	(g)	(g)	(g)	(g)	(g)	(cm <sup>3</sup> ) 4	(g)	(g/c m <sup>3</sup> ) <sup>1</sup>	(g/ cm m <sup>3</sup> ) <sup>2</sup>	(g/c m <sup>3</sup> ) <sup>5</sup>	err	$Q_{lit}$ (g/cm <sup>3</sup> )
Carnelian	5.22	5.225	5.224	5.220	0.00	5.230	13.68	2.27	2.6	2.61	0.0	2.64 [17]
	7								1	23		
Clear quartz	7.03	7.036	7.032	7.029	0.00	7.041	15.51	2.38	2.2	2.19	0.3	2.648 [17]
	3								0	52		
Blue banded agate	5.61	5.618	5.620	5.614	0.00	5.624	14.60	2.42	2.5	2.58	0.1	2.60 [18]
	9								9	89		

<b>Nummite</b>	5.79 3	5.794	5.795	5.789	0.00 1	5.799	13.76 8	2.75	2.3 7	2.36 58	0.2	3.01(anthophyllite) [19]
<b>Snowflake obsidian</b>	2.82 0	2.821	2.821	2.816	0.00 1	2.821	6.617	2.20	2.3 4	2.33 84	0.0	2.38 ± 0.05 (obsidian) [7]
<b>Malachite</b>	7.89 4	7.895	7.893	7.889	0.00 1	7.903	30.61 8	3.82	3.8 7	3.86 41	0.1	4.03 [21]
<b>Rose quartz</b>	2.48 6	2.484	2.485	2.480	0.00 1	2.484	6.591	2.63	2.6 4	2.64 11	0.0	2.65 [17]
<b>Nephrite jade</b>	2.64 5	2.643	2.644	2.639	0.00 1	2.644	7.062	2.82	2.6 6	2.65 54	0.2	2.95 ± 0.05 [22]
<b>Petrified wood</b>	2.46 2	2.463	2.462	2.457	0.00 1	2.462	6.557	2.62	2.6 5	2.64 69	0.2	2.60 [34]
<b>Brecciated jasper</b>	3.50 4	3.505	3.507	3.500	0.00 2	3.507	10.17 5	2.54	2.8 9	2.88 74	0.2	2.60 [18]
<b>Dalmatian jasper</b>	3.88 0	3.879	3.878	3.874	0.00 1	3.881	10.13	2.53	2.6 0	2.60 43	0.0	2.58 [23]
<b>Turritella agate</b>	3.53 7	3.536	3.535	3.531	0.00 1	3.537	9.206	3.06	2.6 0	2.58 83	0.3	2.60 [24]
<b>Aquamarin e</b>	3.29 5	3.299	3.297	3.292	0.00 2	3.298	8.237	2.35	2.4 9	2.48 57	0.2	2.641 [25]
<b>Green aventurine</b>	4.00 0	4.000	4.000	3.995	0.00 0	4.002	12.71 8	3.17	3.1 7	3.16 67	0.1	2.65 [26]
<b>Pink agate</b>	9.07 6	9.075	9.074	9.070	0.00 1	9.086	23.53 7	2.93	2.5 9	2.58 38	0.1	2.60 [18]
<b>Brecciated jasper</b>	2.56 9	2.565	2.567	2.562	0.00 2	2.567	8.217	3.28	3.1 9	3.19 23	0.1	2.60 [18]
<b>Opalite</b>	4.00 0	3.995	3.996	3.992	0.00 3	3.999	9.632	2.40	2.4 0	2.40 06	0.1	2.20 (glass) [27]
<b>Yellow ocean jasper</b>	3.34 0	3.342	3.340	3.336	0.00 1	3.342	8.858	2.52	2.6 4	2.63 72	0.2	2.60 [18]
<b>Fuchsite</b>	4.63 3	4.632	4.631	4.627	0.00 1	4.635	12.55 1	2.66	2.7 0	2.69 62	0.2	2.88 [26]
<b>Orange moonstone</b>	2.20 8	2.207	2.208	2.203	0.00 1	2.207	10.48 3	2.75	4.7 3	2.65 43	0.0	2.56 [35]
<b>Shiva lingam</b>	2.38 7	2.386	2.386	2.381	0.00 1	2.386	8.028	2.67	3.3 5	2.78 91	0.2	2.70 [29]
<b>Amber</b>	1.65 8	1.657	1.656	1.652	0.00 1	1.655	1.784	0.67	1.0 7	1.07 99	0.8	1.05 [30]
<b>White quartz</b>	- -	-	-	-	-	16.1	49.40 6	2.90	3.0 7	2.62 79	4.4	2.648 [17]
<b>Yellow fluorite</b>	9.25 0	9.252	9.249	9.245	0.00 2	9.262	25.23 1	3.14	2.7 2	2.72 04	0.0	3.18 [31]

<b>Green fluorite</b>	8.00 9	8.008	8.005	8.002	0.00 2	8.017	21.81 9	2.72	2.7 2	2.71 42	0.2	3.18 [31]
<b>Blue fluorite</b>	9.74 3	9.741	9.740	9.736	0.00 2	9.754	26.41 3	2.63	2.7 1	2.70 41	0.0	3.18 [31]
<b>Red jasper</b>	1.88 8	1.889	1.888	1.883	0.00 1	1.887	5.225	2.61	2.7 6	2.76 25	0.3	2.60 [18]
<b>Cu ref.</b>	16.370	16.36 3	16.36 1	16.370	0.00 5	16.42 6	146.6 7				0.1 7	8.929
<b>d<sup>6</sup> (cm)</b>	2.542	2.540	2.541		0.00 1	16.40 6						8.940

<sup>1</sup> Displacement of water was used to measure the volume of the natural stone using eqn.4.

<sup>2</sup> Mass-based suspension method employing Archimedes principle.

<sup>3</sup> This natural stone specimen mass exceeded the measuring range of the jewellery scale so the level method was used instead

<sup>4</sup> Density of water used to calculate  $V_{\text{stone}} 0.9982 \text{ g}\cdot\text{cm}^{-3}$  at 20 °C (UKAS ISO/IEC17025 and ISO Guide 34 certified).

<sup>5</sup> Density of water at 21.9 °C was  $0.9977956 \text{ g}\cdot\text{cm}^{-3}$  (CRC handbook of physics and chemistry) 18.2 M $\Omega$ -cm DI water ELGA (Pure lab chorus).

<sup>6</sup> The dimensions of the copper reference cube were measured with a 3 d.p. digital caliper (Draper Expert) and the density of deionised water was taken as  $0.99656 \text{ g cm}^{-3}$  (300K, 1 bar). The density of 99.95% w/w copper at 300K is  $8.930 \text{ g cm}^{-3}$  ([https://www.engineeringtoolbox.com/copper-density-specific-heat-thermal-conductivity-vs-temperature-d\\_2223.html](https://www.engineeringtoolbox.com/copper-density-specific-heat-thermal-conductivity-vs-temperature-d_2223.html)) The mass of the Cu ref. cube was measured with a 2 d.p. accuracy professional digital scale (146.67 g) and was taken as the average of three measurements (146.66 g, 146.68 g and 146.67 g).

<sup>7</sup> Percent error of the mass-based suspension method with a 3 d.p. jewellery scale calculated from Eqn 7.

The volume in the density determination was either from the level method or from the changes in mass of the suspension method due to Archimedes principle. The densities determined by the later method show higher precision and accuracy which is reflected in the significant figures reported for these measurements.

If we compare the accuracies of the densities determined by the simple and inexpensive suspension method, we reach the conclusion that this technique is accurate and cost-effective. We therefore recommend that educators, collectors, and merchants of natural stones use this technique to identify natural stones in combination with morphological characterisation. This will reduce the use of incorrect names for these minerals on sites that commercialise these materials. By using a more accurate digital balance of four d.p., we find that the average percent error (% err.) between the suspension method using the three d.p. jewellery balance and the more accurate four d.p. balance by Fisher analytical series FAS224/E is only 0.3%. However, the average percent error of the level method and the suspension method using the jewellery balance was 9.2%. This suggests that the methodology developed with the suspension method and the jewellery balance is accurate to the two d.p. with an average percent error of 0.3% which could potentially further improve by measuring only natural stones that have volumes smaller than  $10 \text{ cm}^3$ . This is evident through the measurement of the copper reference cube which had an inherent of the technique is assessing the volume of the cube of only 0.1%.

The uncertainty of the measurement of the density using the 3 d.p. jewellery balance was assessed using a copper density reference cube ( $d = 1 \text{ inch} = 2.54 \text{ cm}$ ) to validate the accuracy of the volume measurement of the stone. To increase the accuracy of the volume determination of this geometric cube, we have used a digital calliper with 0.001 cm accuracy. This resulted in a volume of the reference cube of  $16.406 \text{ cm}^3$ . This was remarkably close to the volume of the metal cube measured from the mass-based suspension method on a three d.p. digital jewellery balance, which was found

to be  $16.421 \text{ cm}^3$ . So, the differences in volume between the exact geometric shape of the reference cube and the volume calculated from the mass-based suspension method are of the order of  $0.01 \text{ cm}^3$ . From this one measurement of the copper density reference cube, we find that the uncertainty of the technique in measuring densities with the three d.p. jewellery scale is of the order of 0.1%. This suggests that the densities with the three d.p. jewellery scale can be reported with accuracy up to two d.p.. Densities with 2 d.p. accuracy are commonly reported in mineralogy reports so the method developed with the jewellery balance could essentially also be used in mineralogy when the density of non-porous natural stones is determined.

### 3.2. Gas pycnometry – Non-Destructive Density of Solids via Boyle’s Law

Gas pycnometers calculate the actual volume of a solid by measuring pressure changes ( $\Delta P$ ) that happen when helium with a known mass expands from a reference chamber into a sample chamber of unknown volume. The sample volume and density can be obtained from the  $\Delta P$  reading with the ideal gas law ( $P \cdot V = \text{constant at constant } T$ ) without immersing the sample in liquid or making a powder from it. Helium is preferred for this application because its tiny kinetic diameter of 0.26 nm enables it to access most open porosity spaces within a solid, while remaining unable to pass through crystalline lattice structures. The commercial tools automate the expansion process by using highly accurate pressure transducers ( $\pm 0.0001 \text{ psi}$ ) and chambers with precise temperature control ( $\pm 0.01 \text{ K}$ ). Table 4 summarises current models and typical costs of such highly specialised instruments.

**Table 4.** Gas Pycnometer Models: Sample Capacity, Accuracy and Market Price.

Model	Sample volume range	Stated precision ( $\text{g cm}^{-3}$ )	Typical price (USD)*
Micromeritics AccuPyc II 1340	0.1–135 $\text{cm}^3$	$\pm 0.0001$	5 k (refurbished)–18 k (new) [36]
Anton Paar Ultrapyc 5000	0.5–100 $\text{cm}^3$	$\pm 0.0002$	18–25 k [37]
OEM bench-top units (China)	1–50 $\text{cm}^3$	$\pm 0.001$ – $0.002$	1–3 k [38]

### 3.3. Precision-to-Cost Comparison

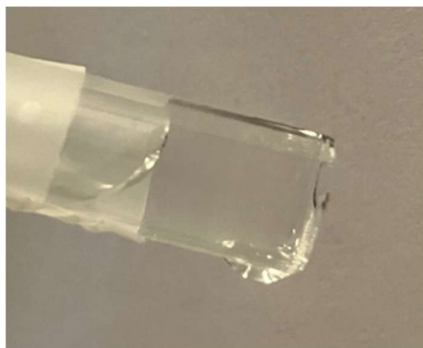
We have performed a brief market search on instruments that can measure the density of stones in a non-destructive way to make a comparison with the mass-based suspension method suggested in this work. We calculate the ratio between precision of the instrument per the cost of the instrumental setup and find the following results.

- Mass-based suspension method using a jewellery scale (this work):  $\pm 0.01 \text{ g cm}^{-3} / \$20 \Rightarrow 5.0 \times 10^{-4}$  precision-units per \$.
- AccuPyc II (new):  $\pm 0.0001 \text{ g cm}^{-3} / \$18,000 \Rightarrow 5.6 \times 10^{-9}$  precision-units per \$.

A higher precision-per-cost ratio makes an instrument more accessible and practical for educators, collectors and even mineralogists who need an affordable method to measure stone densities. While gas pycnometers provide hundred-fold greater absolute precision, the mass-based suspension method using a jewellery scale a precision-to-cost ratio that is 5-fold greater than gas pycnometry instruments, demonstrating its value for educators and routine gem identification.

### 3.4. Future Directions in Improving the Measurement of Densities of Natural Stones Using Glassware

Based on our study, we estimate that an overflow vessel with a square cross-section tube emerging from the main vessel would dispense water more accurately, as the water meniscus perimeter would be co-linear with the opening of the square tube. Currently the small circular diameter tube used in commercially available borosilicate overflow vessels of 500 ml capacity retains some of the overflowed water inside the tube due to the formation of a double meniscus at the tip of the overflow tube (see Figure 5) due to adhesive forces of water with the walls of the glass tube (e.g. capillary effect).



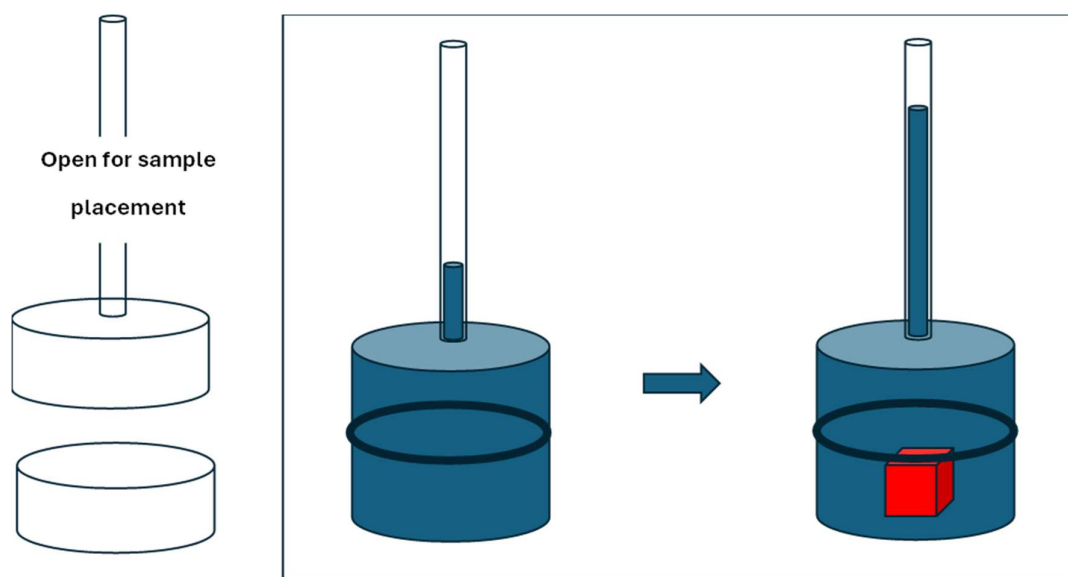
**Figure 5.** Double meniscus formed by water adhesion to glass tube walls.

This capillary effect would be limited using a 1 x 1 cm square overflow tube that would be large enough to eliminate the formation of the double meniscus at the overflow tube. Also, the availability of inexpensive jewellery scales with a higher measuring range would significantly improve versatility of the mass-based suspension method.

In the chemistry laboratory the density of liquids and solid powders is usually measured using a liquid pycnometer which was an invention of Dmitri Ivanovich Mendeleev and the German glassworker, Heinrich Geissler in 1959. A method to measure the density of metals and gemstones was first described in the book of the Iranian polymath scholar al-Beruni (Abu al-Rayan Muhammad ibn Ahmad al-Biruni, 973 - after 1050 A.D.) with the title 'The book most comprehensive in knowledge of precious stones' (Translation by Hakim Mohammad Said, No. 66, ISBN 969-8016-28-7, Pakistan Hijra Council). In this book he used the hydrostatic method of finding the specific gravity and density of metals and gemstones with precision with a conical instrument in which he placed a certain mass of a solid (100 Mithqāl = 425 g) and measured the mass of overflowed water through a semi-circular tube that was pierced to reduce the water capillary effect, which was called the 'Conical instrument of Abū al-Rayān'.

In the liquid pycnometer currently used the weight of the liquid displaced by the solid is determined by subtracting the weight of the pycnometer and liquid from the weight of the pycnometer, solid, and liquid. This weight difference is the liquid that was displaced from the closed pycnometer and using the density of water one can calculate the volume of the solid inside the pycnometer. This technique although works very well for liquids and powdered solids has the limitation that it is a destructive method for natural stones as they would have to be powdered, to fit in the liquid pycnometer. Therefore in this study we suggest the design features of a liquid pycnometer that is non-destructive for solids. This liquid pycnometer could use the level method but with improved design and glassware that is currently possible to manufacture.

We suggest that the level method could be improved using a sealed compartment that would fit the natural stone in a non-destructive way and then this compartment with a small diameter graduated cylinder at the top would increase the accuracy of the volume measurement. A simplified device is shown in Figure 6 which shows the liquid pycnometer in an open and closed position. It also shows the level of the displaced liquid inside the capillary tube at the top of the pycnometer.



**Figure 6.** Simplified schematic of a proposed inexpensive pycnometer for natural stones to which a known mass of water is added first without the stone using an accurate syringe or pipettor (e.g. 1ml) and then the same amount of water is added with the stone inside the pycnometer.

The volume of the water inside the top graduated cylinder could be measured by a geometric equation that correlates the inner diameter of the tube to the change of height of water (or other fluid) inside the tube using the geometry equation of a cylinder,

$$V = \pi r^2 h \quad (8)$$

The inner diameter of the glass tube could be measured very accurately with a caliper that has a typical accuracy of  $\pm 0.01$  mm, which would result in an accuracy of the volume of the order of  $10^{-7}$   $\text{cm}^3$ , assuming that the height of liquid is measured visually with an accuracy of 1 mm. Also, thin glass tubes can be made to have accurate diameters of  $\pm 0.005$  mm error in the diameter value which could potentially increase even further the accuracy of such a device. This would be an improved design of the liquid pycnometer currently used which is destructive and needs the solid sample to be made in a powder, first, to measure its volume. Making a solid natural stones significantly increases the surface area of the solid that interacts with the liquid phase, which could enhance error in density measurements due to leak of elements in the natural stone, into the aqueous phase, which could potentially alter the density of water due to ion hydration.

Such a device would also make possible to study the expansion of liquids as a function of temperature or due to changes of solute concentration, making it a versatile analytical technique.

#### 4. Conclusions

We have used a simple and inexpensive mass-based suspension method to determine the densities of twenty-seven natural stones. The densities are reported to an accuracy of four d.p. and can be used as a reference for accurate densities of non-porous natural stones. We find the use of a jewellery scale with three d.p. is accurate enough to determine densities of natural stones with equipment costs of about 20\$ to two d.p. accuracy. Densities in the range of  $1.07 - 8.93$   $\text{g cm}^{-3}$  have been measured with this method. The measurement uncertainty of the method developed is of the order of 0.1% error when compared to the density of a Cu density cube reference material. This technique can readily be used by mineralogists, educators, and collectors of natural stones to perform easier identification of natural stone samples, using a physical parameter such as density in addition to morphological characterisation. This study can also be used as an activity in the chemistry, physics, and materials science lab where students use inexpensive instrumentation to identify the densities of various rocks and natural stones and compare the accuracy of various techniques used. Finally, we

provide the design details of a new liquid pycnometer which can measure the density of rocks, natural stones, and other solid object of irregular shape non-destructively, with inexpensive glassware and study the effect of temperature and solute concentration on the densities of liquids.

**Supplementary Materials:** The following supporting information can be downloaded at the website of this paper posted on Preprints.org.

**Author Contributions:** Conceptualization, C.D.Z.; methodology, C.D.Z.; validation, C.D.Z., D.D. and E.L.; formal analysis, C.D.Z., D.D. and E.L.; investigation, C.D.Z., D.D. and E.L.; resources, C.D.Z.; data curation, C.D.Z, D.D. and E.L.; writing-original draft preparation, C.D.Z. and E.L.; writing—review and editing, C.D.Z., E.L. and D.D.; project administration, C.D.Z; funding acquisition, C.D.Z. Authors have read and agreed to the published version of the manuscript. All authors have read and agreed to the published version of the manuscript.

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**Data Availability Statement:** The original data presented in the study are openly available in ResearchGate with the (title: Accurate Densities of 27 Natural Stones Using the Mass-based Suspension Method) following DOI 10.13140/RG.2.2.25704.38403/1 (DOI 10.13140/RG.2.2.25704.38403).

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