

## Using Gas Pycnometry to Determine Semisolid Density

Relevant for: food, personal care, cleaning products, slurries, density, pycnometry

Knowing the density of a material enables effortless conversion of mass to volume and vice versa. Many materials that liquid density analyzers cannot accommodate (due to viscosity or solid content), can be handled easily by the Ultracyc 5000. To illustrate the use of the Ultracyc 5000 for these types of materials, measurements on a variety of food, personal care, and cleaning items are reported.



### 1 Introduction

Density is a physical parameter that plays an important role in all material states, whether solid, liquid, or gas. It is measured throughout academia and industry to gain insight into the properties of solid materials. Density is used to:

- Convert between mass and volume
- Ensure sample purity
- Determine concentration of components
- Track the crystal phase structure of materials
- Determine volatile organic content in order to assess the level of drying
- Track tablet void volume and material segregation

True density is the mass of a solid that occupies a unit volume, but excludes the volume of pores and voids within the substance. This is measured by gas pycnometry using a displacement media known to fill both the spaces in between particles as well as any internal pores.

Using Boyle's Law (Equation 1), an application of the ideal gas law assuming a *constant temperature*, the volume of a gas displaced by a sample can be determined through a series of pressure measurements - as long as the volumes of the

chambers used for the measurements are known. To ensure that constant temperature, Ultracyc 5000 instruments employ a Peltier system that maintains temperature from 15 to 50 °C with a stability of  $\pm 0.05^\circ\text{C}$  – the highest level of stability on the market today.

Equation 1: Boyle's Law

$$P_1V_1 = P_2V_2$$

The density measurement of *solids* with gas pycnometry is straightforward. Likewise, the density measurement of *liquids* is straightforward with a device designed to measure the density of liquids. However, there exists an entire class of materials — *semisolids* — that may have a high viscosity or some solid content that does not allow for their easy measurement via such liquid density measurement devices. Semisolids are found in industries ranging from personal care to food and beverage to cleaning products. Semisolid density is important for quality control, material purity, and the packaging process, among other things. Here, we show that the Ultracyc 5000 can quickly, simply, and repeatably assess the density of semisolid materials.

The Ultracyc 5000 instruments feature bi-directional gas flow capability, which allows the gas to expand either from the sample chamber to the reference chamber or the reverse (also referred to as *PowderProtect* mode - Figure 1). This unique feature minimizes the effect of vapor pressure in semisolid materials and provides accurate density measurement even in a high vapor pressure sample [1].

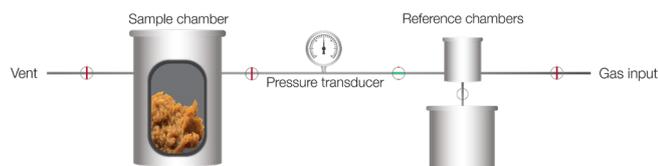


Figure 1: PowderProtect mode illustrated

## 2 Density Measurement

Forty-one samples were measured on the Ultracyc 5000, with the parameters listed in Table 1. Cell size was chosen according to the amount of sample available for the measurement. For best results, the cell should be  $\frac{2}{3}$  to  $\frac{3}{4}$  filled with sample. All measurements in this report were performed in *Reference first* expansion direction, it being more suitable than *Sample first* due to the presence of significant vapor pressure for most of the samples. The Ultracyc 5000 was temperature controlled to 25 °C for each of these measurements. The results are outlined in Table 2.

Table 1: Recommended measurement parameters

Parameter	Setting
Cell size	Appropriate to sample
Gas type	Helium or Nitrogen
Target pressure	18 psig
Flow direction mode	Reference first
Equilibration	Pressure
Preparation mode	Flow, 1 minute
Maximum runs	15
Runs to average	3
End Mode	Automatic, < 0.05%

## 3 Discussion

For semisolid samples, there is an elegant solution to measuring density in cases where devices designed to measure the density of liquids may struggle. Table 2 is separated into categories, and at least three materials per category were measured to illustrate the versatility of the Ultracyc. The cell sizes were either L (large, 131.7 cm<sup>3</sup> nominal volume), M (medium, 48.1 cm<sup>3</sup> nominal volume), or S (small, 10.8 cm<sup>3</sup> nominal volume). Repeatability was calculated as the standard deviation divided by the average of the three density run results. From thick pastes like peanut

butter to liquids like nail polish, gas pycnometry is able to give repeatable results and distinguish between different recipes and consistencies.

For example, one can easily distinguish between different types of pasta sauce by measuring the density. Basil & garlic pasta sauce has an average density of 1.0388 g/cm<sup>3</sup> with 0.00005 (0.005%) repeatability, whereas the mushroom pasta sauce has a slightly higher density of 1.0461 g/cm<sup>3</sup> with 0.00005 (0.005%) repeatability. Even smaller density differences are easily and accurately detected - traditional tomato pasta sauce has a density of 1.0498 g/cm<sup>3</sup>.

Chunky peanut butter (1.0901 g/cm<sup>3</sup>), which has pieces of whole nuts mixed into it, can be distinguished from creamy peanut butter (1.2003 g/cm<sup>3</sup>), which has no nut pieces, by density. Using gas pycnometry in this way on the Ultracyc 5000, it is easy to perform a quality control check or distinguish between samples that otherwise appear identical.

Regardless of the semisolid, the procedure to obtain density is the same: quick and easy. Cleanup is especially easy; after measurement, simply dispose of the sample and rinse the cell with soapy water. For samples that are sticky or hard to clean, a disposable cell liner is available.

## 4 Conclusions

The Ultracyc 5000 is an excellent instrument for measuring the density of semisolids and liquids with suspended particles (slurries), samples that are difficult or impossible to measure with devices designed to measure the density of liquids. Highly accurate and repeatable measurements ensure that small differences in the density of the various constituents and final product can be detected easily and confidently. The unique features of the Ultracyc 5000 such as: bi-directional airflow expansion, Peltier temperature control, and the easy and repeatable lid locking mechanism, all make it an essential instrument in the manufacturing plant for quick and accurate streamline testing and in research and development applications.

## 5 References

1. [www.anton-paar.com](http://www.anton-paar.com), I15IA004EN-A Application Report: **Density and Percent Solids of a Slurry** Anton Paar. [Online] April 2020. [www.anton-paar.com](http://www.anton-paar.com)

## 6 Results

Table 2: Semisolids run on Ultrapyc 5000

Category	Sample	Cell Size	Container Type	Consistency	Density (g/cm <sup>3</sup> )			Average (g/cm <sup>3</sup> )	Repeatability (g/cm <sup>3</sup> )
					Run 1	Run 2	Run 3		
<b>1- Foods</b>									
<b>A. Condiments</b>									
	Yellow mustard	L	Squeeze bottle	Smooth thick liquid	1.0557	1.0554	1.0554	1.0555	0.00013
	Dijon mustard	L	Squeeze bottle	Grainy thick liquid	1.0896	1.0892	1.0894	1.0894	0.00015
	Stoneground mustard	L	Squeeze bottle	Coarse thick liquid	1.0903	1.0909	1.0904	1.0905	0.00024
<b>B. Sauces</b>									
	Basil & garlic pasta sauce	L	Glass jar	Chunky liquid	1.0389	1.0388	1.0388	1.0388	0.00005
	Mushroom pasta sauce	L	Glass jar	Chunky liquid	1.0461	1.0460	1.0461	1.0461	0.00005
	Traditional tomato pasta sauce	L	Glass jar	Chunky liquid	1.0498	1.0498	1.0499	1.0498	0.00004
<b>C. Canned goods</b>									
	Baked beans	L	Aluminum can	Thick lumpy liquid	0.6943	0.6942	0.6942	0.6942	0.00007
	Creamed corn	L	Aluminum can	Thick lumpy liquid	1.0478	1.0478	1.0479	1.0478	0.00004
	Cranberry sauce with berries	L	Aluminum can	Thick lumpy liquid	1.1790	1.1788	1.1788	1.1789	0.00008
<b>D. Salad dressings</b>									
	Chunky blue cheese salad dressing (Brand A)	L	Squeeze bottle	Thick lumpy liquid	0.9975	0.9976	0.9977	0.9976	0.00008
	Chunky blue cheese salad dressing (Brand B)	L	Squeeze bottle	Thick lumpy liquid	1.0043	1.0040	1.0047	1.0043	0.00029
	Creamy cheese salad dressing (Brand A)	L	Squeeze bottle	Thick smooth liquid	1.0097	1.0095	1.0095	1.0096	0.00009
<b>E. Squeeze tubes</b>									
	Peanut butter	M	Squeeze bottle	Thick paste	1.1142	1.1130	1.1129	1.1134	0.00053
	Tomato paste	M	Squeeze bottle	Thick paste	1.1373	1.1378	1.1380	1.1377	0.00026
	Wasabi paste	M	Squeeze bottle	Thick paste	1.2000	1.2003	1.2007	1.2003	0.00024
<b>F. Nut butters</b>									
	Crunchy peanut butter	M	Plastic jar	Thick chunky paste	1.0904	1.0900	1.0899	1.0901	0.00020
	Creamy cashew butter	M	Glass jar	Thick paste	1.1183	1.1179	1.1179	1.1180	0.00017
	Creamy peanut butter	M	Plastic jar	Thick paste	1.2004	1.2003	1.2002	1.2003	0.00007
<b>G. Fruit preserves</b>									
	No sugar added marmalade	M	Glass jar	Thick gel	1.2671	1.2673	1.2676	1.2673	0.00016
	Jelly (no seeds)	M	Glass jar	Thick gel	1.3135	1.3131	1.3178	1.3148	0.00162
	Jam (with fruit chunks and seeds)	M	Glass jar	Thick gel	1.3145	1.3144	1.3145	1.3145	0.00004

Category	Sample	Cell Size	Container Type	Consistency	Density (g/cm <sup>3</sup> )			Average (g/cm <sup>3</sup> )	Repeatability (g/cm <sup>3</sup> )
					Run 1	Run 2	Run 3		
<b>H. Cake frosting/Icing</b>									
	Coconut pecan	M	Plastic tub	Thick, stiff paste	1.1723	1.1718	1.1718	1.1720	0.00085
	Sugar-free vanilla	M	Plastic tub	Thick, stiff paste	1.2481	1.2466	1.2455	1.2467	0.00020
	Vanilla	M	Plastic tub	Thick, stiff paste	1.3197	1.3195	1.3191	1.3194	0.00019
	Cream cheese	M	Plastic tub	Thick, stiff, chunky paste	1.3197	1.3195	1.3194	1.3195	0.00009
<b>2- Non-foods</b>									
<b>A. Personal care</b>									
	Hair gel	L	Squeeze bottle	Gel	1.0077	1.0072	1.0070	1.0073	0.00029
	Exfoliating scrub	L	Pump bottle	Thin gel	1.0259	1.0260	1.0255	1.0258	0.00021
	Body wash/Shower gel	L	Squeeze bottle	Thin gel	1.0272	1.0271	1.0268	1.0270	0.00017
	Aluminum-free green deodorant	M	Plastic twist-up tube	Rigid gel	1.0419	1.0422	1.0425	1.0422	0.00024
	Regular white deodorant	M	Plastic twist-up tube	Rigid gel	1.0820	1.0812	1.0811	1.0814	0.00037
	Clear gel deodorant	M	Plastic twist-up tube	Thin gel	1.1231	1.1236	1.1245	1.1237	0.00052
	Striped paste/gel toothpaste	S	Plastic tube	Thick paste	1.3594	1.3594	1.3594	1.3594	0.00000
	White paste toothpaste	S	Plastic tube	Thick paste	1.3677	1.3680	1.3681	1.3679	0.00012
	Green gel toothpaste	S	Plastic tube	Thick gel	1.3843	1.3841	1.3834	1.3839	0.00028
<b>B. Cosmetics</b>									
	Gel nail polish	S	Glass bottle	Smooth liquid	0.9628	0.9669	0.9755	0.9684	0.00546
	Clear nail polish	S	Glass bottle	Smooth liquid	0.9768	0.9783	0.9866	0.9806	0.00440
	Metallic nail polish	S	Glass bottle	Smooth liquid	1.1682	1.1723	1.1872	1.1759	0.00694
<b>C. Household goods</b>									
	Brass polish	M	Squeeze bottle	Smooth liquid	1.1715	1.1736	1.1759	1.1737	0.00153
	Cooktop cleaner	M	Squeeze bottle	Thick liquid	1.2292	1.2299	1.2310	1.2300	0.00060
	Multi-purpose cleaner	M	Squeeze bottle	Thick liquid	1.2809	1.2813	1.2817	1.2813	0.00025
	Leather shoe polish	S	Aluminum can	Wax	0.8584	0.8589	0.8584	0.8586	0.00027
	Leather shoe scuff cover	S	Squeeze bottle	Thin liquid	1.0238	1.0240	1.0258	1.0245	0.00088
	Leather cleaner	S	Aluminum can	Wax	1.0257	1.0259	1.0261	1.0259	0.00016