

Application of the DensiPyc 1000 for Battery Materials

1. Background

Improving the volumetric energy density of rechargeable batteries is a central objective in energy-storage technology development. Achieving this goal depends strongly on the intrinsic properties of the active cathode and anode materials.⁽¹⁾ Among these properties, true density, defined as the mass per unit volume of the material framework excluding open pores, is a critical parameter influencing electrode compaction density and, ultimately, battery volumetric performance. Accurate determination of true density is therefore essential for materials research, process optimization, and performance evaluation.⁽²⁾

True density also reflects fundamental material characteristics such as crystal structure integrity, doping state, and synthesis quality. For example, the true density of an ideal graphite crystal is approximately 2.26 g/cm³, while natural and synthetic graphites typically show lower values depending on their degree of graphitization. As a result, true density serves as an effective indicator of process stability. Deviations from expected values may indicate process fluctuations or raw-material contamination. If such variations remain uncontrolled, they can lead to performance discrepancies among individual cells, accelerated non-uniform aging, and potential safety risks in battery packs.⁽³⁾

For these reasons, true density measurement has become a routine, essential characterization method for battery materials.^(4,5) The gas expansion (volume displacement) method is the most widely adopted technique. The method is based on Boyle's law and typically employs helium as the displacement gas. Helium is selected due to its extremely small kinetic diameter (0.26 nm), which allows penetration into most open and micropores, as well as its chemical inertness and negligible adsorption at ambient temperature. Consequently, the measured volume accurately corresponds to the skeletal volume plus closed pores.

The primary objective of this *AMI Note* is to evaluate the performance of the **DensiPYC 1000** true density analyzer for battery cathode and anode materials and to examine how material microstructure influences measurement stability. The results provide practical guidance for quality control in battery material manufacturing.

2. Experiment

2.1 Instrument calibration

All measurements were performed using the **DensiPYC 1000** true density analyzer from **AMI**. Standard steel calibration spheres were used to determine the reference volume (V_r) and sample cup volume (V_s). This procedure compensates for manufacturing tolerances, valve dead volume, and systematic volumetric errors. Calibration is recommended monthly and is required whenever the test gas or operating temperature is changed.

Instrument stability was verified using the built-in “Quick Validation” function with a reference steel sphere. The results are summarized in Table 1. The relative standard deviation (RSD) of 0.0085% demonstrates excellent volumetric stability and repeatability.

Calibration Sample	Volume Measurements (cm ³)			Average (cm ³)	RSD (%)
Standard steel ball	6.3681	6.3668	6.3672	6.3674	0.0085

Table 1: Calibration of *DensiPyc 1000*

2.2 Sample Measurement

The true density of the cathode materials (FePO₄, LiFePO₄) and anode materials (graphite, silicon-carbon anode, porous carbon) was measured sequentially. All measurements were conducted using the 50 cm³ sample cup equipped with a lid, filled to approximately 80% of its volume. True density measurements were performed sequentially for each cathode and anode material. Every sample was analyzed three consecutive times to assess measurement repeatability. Ultra-high-purity helium (99.999%) served as the displacement gas.

Because gas expansion measurements are sensitive to temperature fluctuations, stable thermal control is essential. All measurements in this study were performed under controlled conditions at 25 °C.

To simulate rapid inspection conditions typical of quality control in industrial environments, no pretreatment (e.g., drying) was applied prior to analysis.

3. Results

Table 2 summarizes the true density results for iron phosphate and lithium iron phosphate.

Cathode Sample	True Density Measurements (g/cm ³)			Average (g/cm ³)	RSD (%)
Iron phosphate (FePO ₄)	2.9834	2.9814	2.9795	2.9814	0.0534
Lithium iron phosphate (LiFePO ₄)	3.4988	3.4972	3.4971	3.4977	0.0223

Table 2: True density values for cathode samples measured with *DensiPyc 1000* from AMI

These results demonstrate excellent repeatability for both cathode materials, indicating stable measurement performance.

The true density results for graphite, silicon-carbon composite, and porous carbon are presented in Table 3.

Anode Sample	True Density Measurements (g/cm³)			Average (g/cm³)	RSD (%)
Graphite	2.2306	2.2302	2.2317	2.2308	0.028
Silicon-carbon	2.0119	2.0126	2.0147	2.0131	0.059
Porous carbon	2.2389	2.2336	2.2360	2.2362	0.097

*Table 3: True density values for anode samples measured with **DensiPyc 1000** from AMI*

The larger RSD observed for porous carbon is attributed to its microstructural characteristics. Porous carbon contains a high concentration of micropores. Although helium molecules are extremely small, gas diffusion into these fine pores occurs slowly, requiring extended equilibration time to reach pressure equilibrium. In addition, weak physical adsorption of helium on the extensive internal surface area may slightly influence the measured volume. Together, these effects lead to increased measurement variability.

For materials with high microporosity, extending the pressure equilibration time or increasing the number of measurement cycles is therefore recommended to improve result stability.

4. Conclusions

True density is an effective indicator for monitoring batch-to-batch consistency and synthesis process stability. Stable true density values contribute to uniform electrode fabrication, reduced cell-to-cell variation, consistent aging behavior, and improved battery safety and cycle life. Routine quality control is best supported through multiple measurements and averaging. Measurement repeatability is also strongly influenced by material microstructure. For microporous materials such as porous carbon, longer equilibration time or additional test cycles are recommended to ensure complete gas penetration and stable results.

After proper calibration, the **DensiPyc 1000**, shown in Figure 1, demonstrates excellent repeatability across a range of battery materials. The **DensiPyc 1000** combines high-precision gas pycnometry with intelligent automation tailored for both research and quality control environments. Its automated sample chamber sealing and intelligent reference-volume management minimize operator-dependent variability, delivering reliable and repeatable true-density measurements critical for evaluating cathode and anode materials. Optional configurations such as integrated

temperature control, in-situ automatic weighing, and vacuum degassing allow the system to adapt to moisture-sensitive or porous battery materials, improving measurement stability and workflow efficiency. Together with user-friendly software, fast test modes, and compliance with international standards (ISO 12154, ASTM, DIN), the **DensiPyc 1000** provides accurate, traceable true-density data essential for monitoring material consistency, optimizing electrode compaction, and supporting high-performance battery development.

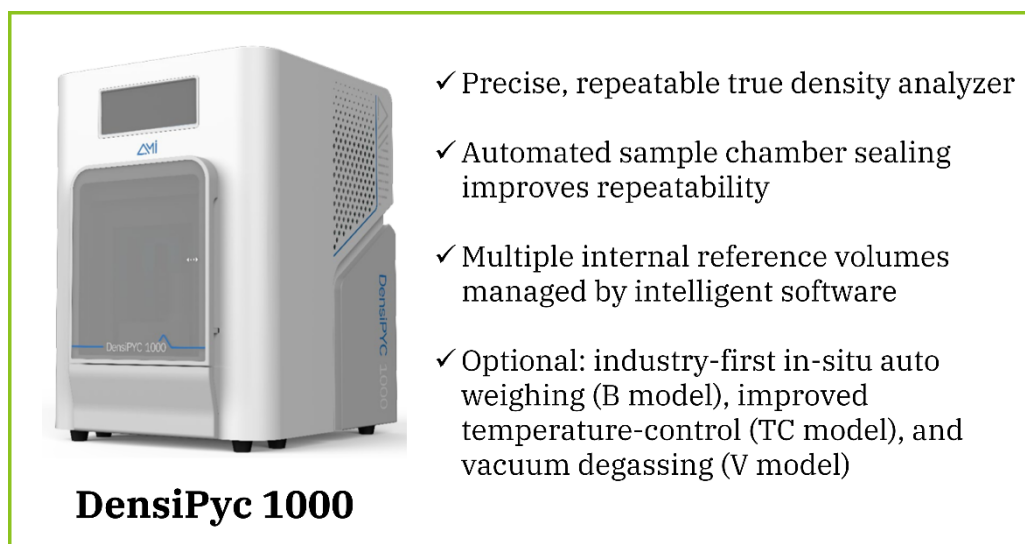


Figure 1: Highlighted features of **DensiPyc 1000** from AMI

5. References

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