

# Protocol

## Helium pycnometry

### 1. Method

This method measures the volume of gas (Helium) displaced by a known mass of powder, and gives the true density of the material. The sample must be completely dried. Around 1-5 g of powder is necessary.

### 2. Equipment

- Instrument: Micromeritics Accupyc 1330 (more info from [http://www.particle.dk/methods/Helium Pycnometry.htm](http://www.particle.dk/methods/Helium%20Pycnometry.htm));
- Analytical balance (precision 0.1 [mg]);
- Spatula for powder samples.

### 3. Protocol

- The sample should be dried at 200 °C, during 1 hour (this can be adjusted depending on the sensitivity of the sample), in air or vacuum (depending on sample).
- Press the white key on the keyboard (2<sup>nd</sup> function) then “Zero”, then “Enter”. The blank measurement (volume of the empty cell) takes about 1 minute.
- When the calibration is done, weigh the mass of the empty cell using the analytical balance (accuracy 0.1 [mg]) and carefully write down the result  $W_C$  [g] (approximate mass = 2.0700 [g])
- With the spatula add a sufficient amount of powder into the cell (1-5 g) and carefully write down the result  $W_T$  [g]
- The mass of the powder is calculated from:

$$W_P = W_T - W_C \text{ [g]}$$

- Put the cell with the powder in the instrument, and close it
- Press the white key on the keyboard (2<sup>nd</sup> function) then “Analyse”
  - o “Sample ID”: type the sample number then press “Enter”;
  - o “Mass”: type the sample mass  $W_p$ , then press “Enter”;
  - o press “Enter”;
  - o press “Enter”
  - o One standard measurement takes 45 [min].
- Once the measurement has started, open “Accupyc.ht” on the connected computer
  - o Go to the “Transfert” menu, select “Capturer le texte”, then Browse to select the folder in which the measurement will be saved. Name the sample [Powder-Lotn°-Accupyc-Experimentn°-Operator.txt](#), then OK.
  - o Once the measurement is finished, go to the “Transfert” menu, select “Capturer le texte”, and “Arrêter”
- Once the measurement is finished and saved, remove the powder from the cell, wash it with water and ethanol and dry at 60 [°C] in the oven.

#### 4. Presentation of the results, data storage, and data treatment

##### Data storage

- Copy the file [Powder-Lotn°-Accupyc-Experimentn°-Operator.txt](#) Go to \\Ltpc40\powderfiles. Copy the folder *Powderfiles*. Paste it in your project folder, and change its name into [Powder-Lotn°](#).
- Paste the TXT file in the folder [Project/Powder-Lotn°/Accupyc/Data](#).

##### Data treatment

- Go to \\Ltpc40\powderfiles. In the folder [Project/Powder-Lotn°](#), open the Excel sheet "Powdersheet.xls"
- Click on the *Accupyc* button, and follow the instructions given in the Excel sheet

##### Remarks:

- One can use the density given by the machine and calculated from the sample weight ( $W_p$ ).
- Or one can also calculate the real density for each run from:

$$d = W_p / \text{Volume}$$

Thus for each run, one can see the trend of the density over the repetitions (~10). If it is not stable, the measurement has to be repeated, or the sample drying has to be improved.