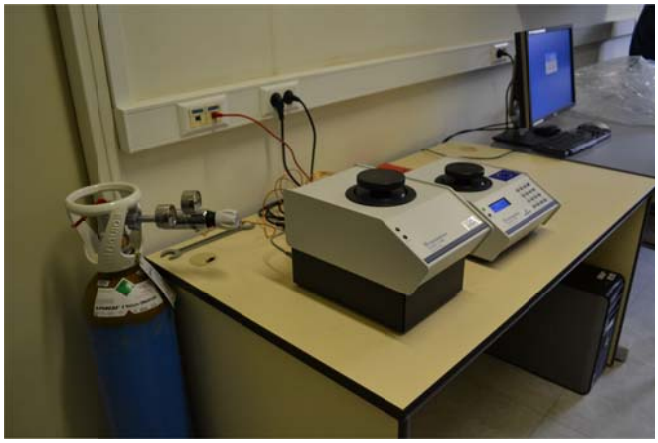
Connectivity measurements

Meaning

Connectivity measurements allow obtaining the percentage of isolated vesicle versus the connected ones. These measurements deliver first-order information on the outgassing capacity (i.e., potential for gas loss) of the magma near fragmentation (Klug et al. 2002; Formenti and Druitt 2003; Giachetti et al. 2010, Shea et al 2011, 2012).

Methodology



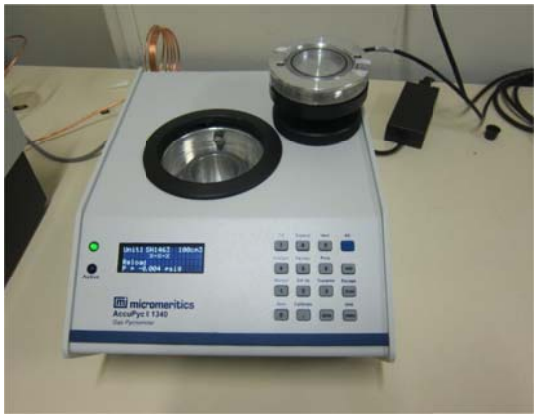


Figure 1 Left:

These measurements are obtained using an AccuPyc II 1340 Gas Displacement Helium Pycnometer (Figure 1). This instrument performs measurement of the skeletal volume of powders and solids that fill sample chambers of 10-100-350 cm^3 (samples from 1x1; 1.8x3.9; 4.6x6; 5.8x13.9 cm, being the first number the diameter and the second the length of the sample). This is well adapted for vesicularity measurements because the helium can enter even the smallest vesicles. The total volume of the sample (solids+vesicles; V_{sam}) is obtained from the glass sphere method on scoria and pumice of irregular shape. Measurement with the pycnometer gives the volume V_{meas} of the solid phases (glass+crystals) plus the volume of any isolated vesicles. The connected vesicularity (X_c) is given by:

$$X_c = 1 - \frac{V_{meas}}{V_{sam}}$$

The same samples are then crushed (grain size of about 20-30 μm) and the volume again determined using the pycnometer. This then gives the mean density of the solid phases in each sample (ρ_s). The total vesicularity (X_t) is then given by:

$$X_t = 1 - \frac{m_{mas}}{\rho_s V_{sam}}$$

where m_{sam} is the mass of the sample. The fraction of isolated vesicle is given by $X_t - X_c$.

Note: For detailed instructions regarding the installation of the AccuPyc II 1340 pycnometer, in addition to detailed notes on the user interface, operating instructions and troubleshooting, please refer to the AccuPyc II 1340 Operators Manual V1.05 (English only). A PDF version of this manual is also available

Equipment
AccuPyc II 1340 Helium Gas Pycnometer
Calibrations standards
Rubber Gloves
Calibrated digital scales
Glass Beads (600-800microns)
300+ml Beaker
Measuring recipient of known volume
Standard of known volume
Ruler
Samples (>8mm-<30mm clast fractions)
Sample should be cleaned and dried for a period of 24 hours, at temperatures of 100 degrees Celsius, prior to analysis (8hrs at 200 degrees Celsius is also suitable).

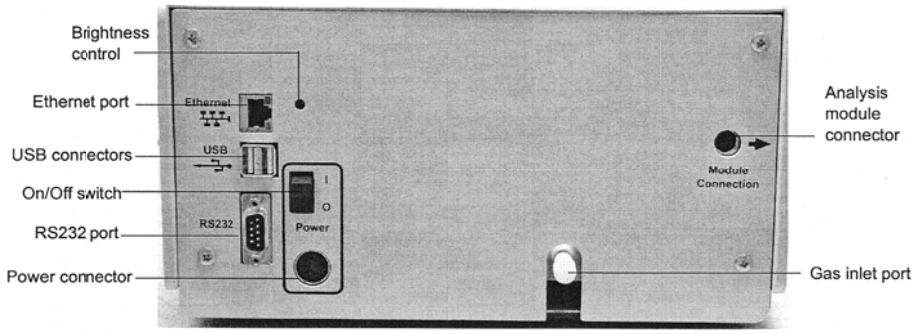
Sample Mass
Sample mass should be quantified using a set of Metler Toledo DB602 electric scales or equivalent. The standard unit of measurement is grams (g).

1. Ensure the scale sits flat and balanced on the operating surface. If a balance is attached to the rear of the scale, this should be used as a reference. If angled or unbalanced, adjusts the length of the supporting legs by rotating a knob at the base of each leg.
2. Ensure the scale is plugged into a power socket.
3. Tare (Zero) the scales (this should be conducted following each measurement).
4. Place your sample on the scale and record the mass (repeat five times and average the results).
5. Record the calculated mass.

Volume minus connected vesicularity (He-Pycnometer)

1. Turn on the AccuPyc II 1340 Micrometrics Gas Pycnometer. (black interrupter behind the machine)

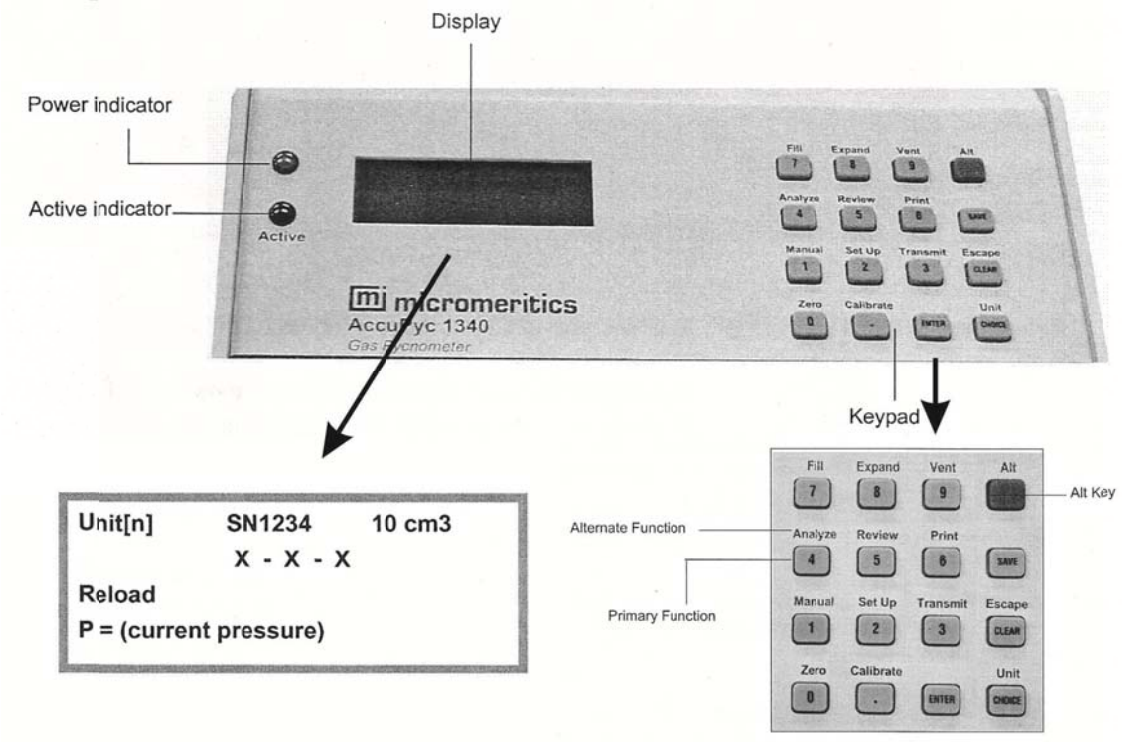
Rear panel



2. Leave for 24 hours to ensure pressure and temperature stabilization inside the pycnometer.

3. Organize the required materials.
   Sample Cups (10 cm^3, 35 cm^3, 100cm^3 350cm^3)
   Calibration standards
   Rubber Gloves
   Samples

Front panel



Display

- **First line**: unit number, serial number and nominal cell volume of the selected unit
- **Second line:** first character = **Fill** valve, second character = **Expand** valve, third character = **Vent** valvX = closed O = open
- **Third line:** display status of the current operation or **Reload** when in an idle state This line may contains an (*) indicating there is a message
- **Fourth line**: at the Reload prompt it show P and T (pressure and temperature), this line is also used to choose options, enter information or provide information
- **Data entry** prompts are followed by a colon (:). Use the keypad to enter the value. If it is out of range you will hear a beep and a message showing the displayed range; enter a value in the range
- **Multiple choice** prompts are followed by a colon (?). to select one press **CHOICE** until the response is displayed **ENTER**
- Press **SAVE** to save information
- Press **Alt + clear** to discard the information you entered

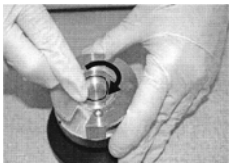
Keyboard

Most keys on the keyboardperform one primary and one alternate function

- Alternate fuction Alt + the key
- Primary function press only the key

4. The chamber cap contains an O-ring that requires routine maintenance. The chamber cap O-ring should be greased at the beginning of each period of use

Greasing the chamber cap O-ring



- Wear gloves

- Turn the chamber cap counterclockwise and lift it from the chamber
- Place the chamber cap on a clean surface, with the O-ring side exposed
- Place a small amount of Dow Corning High vacuum grease on your index finger
- Run your finger around the O-ring groove
- replace the chamber cap; turn the cap clockwise to close it
- Open the gas valve to the pycnometer by lifting the red lever on the gas tank. The gas pressure for the following sample cup holders should be:

$10\ cm^3$ – 1.3bar
$35\ cm^3$ – 1.3bar
$100cm^3$ – 1.5bar
$350cm^3$ – 1.5bar

- Adjust the pressure if necessary.

Note: The pycnometer fill valve should be open when adjusting gas pressure. If the pressure is too high or low, the pycnometer will not function.

Setting regulator pressure

- Be sure the tank pressure for the gas regulator is at least 200 psig. Analyses are terminated automatically if gas is depleted.
- Press **Alt+1** to enter manual mode
- Press **8** (EXPAND) and **9** (VENT) to open the expansion and vent valves;
  second line of the display: **X - 0 - 0**
- Press **7** (FILL) to open the fill valve
  second line of the display: **0 -0 - 0**
- Adjust the regulator pressure control knob (see values above) until the pressure is shown on the regulator display
- Press 7 to close the fill valve, then increase the regulator valve by 2.0 psig;
  second line of the display: **X - 0 - 0**
- Allow the pressure in the pyc to drop below 2.0 psig, then press **8** and **9** to close the other two valves
  second line of the display: **X - X - X**
- Press **SAVE** to return to the **Reload** prompt

Note

- If you press the Alt key accidentally, press it a second time to cancel its function
- You must pres **ALT+CLEAR** to exit Manual mode

5. Choose the appropriate sample holder and gently place a standard into the sample holder/cup using gloves. The standard/sample should represent at least 10 vol. % of the sample holder to minimize uncertainties. The pycnometer will alert you if the sample volume represents <10% of the sample chamber. However, specific standards are provided for each sample holder.

6. Turn the chamber cap anticlockwise/counter clockwise and place on a clean surface (the underside facing up/o-ring at the surface).

IMPORTANT NOTICE:

- always wear gloves
- always touch the black holder, never the side
- keep the cap on the sample chamber except when inserting or removing the sample
- if performing multiple analysis never interchange sample chamber caps

7. Carefully place the sample holder into the fill chamber.
8. Seal the chamber by turning the chamber cap clockwise.

If making measurements directly from the machine

Performing the calibration

Analysis of the standard should be conducted each morning to ensure the pycnometer is calibrated prior to conducting analyses on your samples.

Before starting an analysis, we need to specify the analysis conditions:

1. **Alt+Set-up** => (to scroll the last line use **CHOISE,** and chose**:**
   Analysis parameters, Report options, Calibration data, Communications, Unit types)
   Analysis parameters **ENTER**
2. **Number of purge:* 10 (for the standard) **ENTER**

(for sample cleaning up and removing air and moisture from the inside of the chamber)

3. *Purge fill pressure:* 19.500 psig **ENTER**
4. **Number of cycles:* 10 (for the standard) **ENTER**

(for collecting the precise, accurate data)

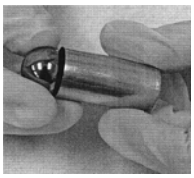
5. *Cycle fill pressure* 19.500 psig **ENTER**
6. *Equilibrate* accepted **ENTER**
7. *Equilibrate rate* 0.005 psig/min **ENTER**
8. *Use run precision* No **ENTER**
9. Press **SAVE** to save the values
10. **Alt+Set-up**
    Report options **ENTER**
11. *Anls display mode*
12. Press **CHOICE** to select **Density** or **Volume** **ENTER**
13. For the other choices we usually press NO
14. Press **SAVE** to save the values and return to the **Reload** prompt
    Calibration data **ENTER**

To view current calibration data

15. *Chamber insert*
16. Press **CHOICE** to select the chamber **ENTER**
17. Cell volume (from calibration) **ENTER**
18. Expansion volume(from calibration) **ENTER**
19.

Performing the calibration

1. Place and empty cup into the sample chamber
2. Press **ALT + .** (decimal)
3. *Calibrate type* **volume** **ENTER**
4. *Volume of Cal Std* value of the standards **ENTER**
5. Press ENTER again to start the calibration
6. The first phase of the calibration, **CAL1**, calibrates the volume offset and when it finishes (after 3 beeps)
7. *Insert Cal* Std place the standards in the cup
8. In doing that tilt the sample cup and allow the standards to roll gently into the cup to prevent denting of the cup. The chamber as to be covered while doing this operation



9. Remove the sample chamber cap, place the sample cup back into the chamber and replace the cap
10. Allow the standards to reach thermal equilibrium with the pycnometer

- Wait 10 min for 1- and 10 cm3 pyc
- Wait 15 min for the 100 cm3 pyc
- Wait 20 min for the 350 cm3 pyc

11. Press **ENTER** to complete the second phase of the calibration, **CAL 2**, the volume scale

After the calibration is complete, the analyzer automatically returns to **Reload** prompt

Proceed to **verifying operation**

12. Press **Alt +4** **ENTER**
13. Press **CHOISE** to view the **Avg Vol** and **Std Dev**

The variance for each sample holder and subsequent standard are listed below. If the analyzed volume is outside the range listed above, recalibrate the pycnometer (see Installation Ch2 of the AccuPyc II 1340 Operators Manual).

Chamber Volume (cm3)	**Standard Volume (cm3)**	**Variance**	**Range**	
10	6.372242	0.004912	6.36733	6.377154
35	16.756283	0.015527	16.74076	16.77181
100	51.076226	0.045323	51.0309	51.12155
350				

Start the analysis

1. To start an analysis:
   Alt+ Analysis
   Chamber insert specify the chamber volume (10, 35, 100, 350 cc) **ENTER**
2. Weigh the sample and insert it in the adequate chamber
3. Introduce the chamber in the pycnometer and close with the cap
4. Press ENTER to start the analysis
5. Press **Alt+Clear** to cancel the analysis
6. Press **CHOICE** to view the average volume and standard calibration

*** NOTE**

Depending on the number of samples to analyze, we recommend distinct approaches.
If there are a small number of samples:
Number of purge: 20
Number of cycles: 10-20 or 30- 40, and take the last value like the real value.
In doing it we consider the bias related to the stabilization of the pycnometer.
If the number of samples is too important and/or the time available is not enough:
Number of purge: 1
Number of cycles: 1
We will apply a correction based on the 40 cycles obtained on a sample-standard, with 20 purge. The mean difference between the first and last value is typically 0,04 so we can add 0,04 to the volume measured with the pycnometer.

If using the computer

1. Open the AccuPyc II 1340 V1.05 Software

2. Select File >Open>Sample Information. Choose a relevant file name and directory to save this file. If working with multiple samples, simple labels (i.e. 1, 2, 3) are best.

3. Hit Enter. A second popup details the sample information, analysis conditions and report options. Choose the desired working conditions. Once chosen, save the conditions and close the pop up window.

4. Select Unit 1>Sample Analysis. Select the appropriate file created for a specific sample (if appropriate) and select OK. Choose the appropriate sample chamber volume and select OK.

5. While waiting for the pycnometer to calculate volume, calculate density of the glass beads (see below) and the bulk density of any sample which has previously been analysed in the pycnometer.

6. Write the result and repeat the above steps with your sample of interest.

Note: Allocate your time appropriately. On average, ten (10) fragments per hour can be analysed, assuming five (5) initial purges and only one (1) analysis cycle. However, for pumicious materials the sample volume increases progressively with each analysis cycle. Subsequently, a series of pumicious calibration analyses should be performed prior to detailed analyses (10 purges and 50 cycles should be sufficient for each pumicious calibration standard - Duration ~1hr per sample).

Precisions: reproducibility to within ± 0.01% of the nominal full-scale cell chamber volume. Reproducibility to within ± 0.02% of the nominal full-scale volume on clean, dry, thermally equilibrated samples, using helium in the 15 to 35 °C range.
Accuracy: accurate to within 0.03% of reading, plus 0.03% of sample capacity