Pycnometer Standard Operating Procedure

1) Record your name, date, and other relevant information in the Pycnometery Log Book.
2) Plug in the pycnometer to a power outlet and attach the gas hose to a gas cylinder, either helium or nitrogen.
3) Power "ON" and allow 10-15 minutes for the pressure transducer to warm up and stabilize.
4) Adjust the tank pressure regulator to slightly above 20 PSIG. Pressures above 25 PSIG can damage the pressure transducer.
5) Select the correct REFERENCE VOLUME for the sample cell to be used. See the front panel of the pycnometer for reference volume selection.

Note: Select the reference volume where the sample cell can be filled ¾ full of sample material. The three sizes from smallest to largest are micro, small, and large.

Note: The toggle valves are CLOSED when their handles are parallel to the cabinet face and OPEN when perpendicular to the cabinet.

6) Fill the sample cup, insert it into the cell holder, and replace the cover.
7) Turn the selector valve to "CELL" if it is not already.
8) Open the "GAS OUT" toggle valve and turn the "GAS OUT RATE" control completely counter-clockwise. Wait for a stable reading.
9) Close the "GAS OUT" toggle valve, turn the "GAS OUT RATE" needle valve clockwise until resistance is felt (not completely closed), and set the meter to zero using the knob next to the digital readout.
10) Turn the selector valve to "REF".
11) Rotate the "GAS IN RATE" needle valve clockwise until it is just slightly open.
12) Open the "GAS IN" toggle valve, and pressurize as close to 17 PSIG as possible using the "GAS IN RATE" needle valve to control the rate of pressurization. Stop the flow by closing the "GAS IN" toggle valve.
13) Record the display reading after it has stabilized. This value is P_1.

14) Slowly turn the selector valve to “CELL”.
15) Record the display reading after it has stabilized. This value is P_2.
16) Divide P_1 by P_2 and record this number.
17) Vent the pressure slowly to prevent blowing powder out of the cell by opening the “GAS OUT” toggle valve with the “GAS OUT RATE” control slightly open.
18) Repeat steps 9-16 until the ratio in step 15 is within ±0.005 for three consecutive measurements.
19) Use the following equation to calculate the true powder volume.

$$V_P = V_C - V_R\left[\left(\frac{P_1}{P_2}\right) - 1\right]$$

Where V_P is the volume of the powder, V_C is the cell volume, and V_R is the reference volume. V_C and V_P depend on the reference volume used are found under ”Pycnometer Calibration Values”.

20) Remove the sample cup from the pycnometer and use an analytic balance to measure the mass of the sample cup with powder.
21) Remove powder from the sample cup and measure the mass of the sample cup. The difference in masses is the mass of the powder
22) Divide the mass by the volumes found in step 18 to find the density of the powder. The average of these measurements should be recorded as the density of the powder.

Pycnometer Calibration Values

LARGE CALIBRATION SPHERE	V_{CAL} large= 56.559 cm^3
SMALL CALIBRATION SPHERES (2)	V_{CAL} small=2.145 cm^3
LARGE SAMPLE CELL VOLUME	V_c large=151.7178 cm^3
LARGE REFERENCE VOLUME	V_{ref} large=90.2884 cm^3
SMALL SAMPLE CELL VOLUME	V_c small=29.8415 cm^3
SMALL REFERENCE VOLUME	V_{ref} small=13.0142 cm^3
MICRO CELL VOLUME	V_c micro=12.4670 cm^3
MICRO REFERENCE VOLUME	V_{ref} micro=7.0537 cm^3

Calibration values measured on 07/12/2007 by Quantachrome Corporation.

Contact

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