

MicroMeritics AccuPyc 1330 Pycnometer User Guide

Joe Doyle 12/12/01
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Introduction

This MicroMeritics AccuPyc 1330 Pycnometer has been optimized for non-destructively measuring the volume and calculating the density of small samples. It has two options for sample size. The standard sample cup has an inside volume of about 1 cm³. There is an alternate second sample cup with an inside volume of 0.1 cm³. Because volume is actually measured, the best measurements are made when the sample cup is near full.

Samples may be solids or powders. The MRL has other hardware to measure liquid density. The sample cell is aluminum, so no sample which could outgas anything corrosive may be run.

Typically, the users starts by logging in with their name and a short description of the sample to be run. Then the Pycnometer is calibrated with the 3/8" OD silicon carbide sphere. Calibration typically takes an hour and a half. Then the sample cup is removed, weighed, the sample is loaded into the sample cup, the sample may be dried in an oven and then cooled in a desiccator, and the loaded cup is then weighed again. The sample cup is then carefully placed into the Pycnometer and an Analysis is started. An arbitrary sample ID number is assigned and entered by the user, then the sample mass are entered, and the run is started. The machine will then perform the measurement, typically five times, unattended. Tests usually take about an hour. When done it will print a report showing the measured sample volume, the calculated sample density, and the sample temperature.

Users must make sure that the inside of the sample chamber and the outside of the sample cup are completely free of any sample particles. Particles can cause the sample cup to become stuck in the instrument.

Users must remove their sample when they pick up their report. Completed samples may not be left in the pycnometer or abandoned on the lab bench.

Because sample masses and volumes are very small, it is critical that fabric gloves be worn EVERY TIME the sample cups are handled. Rubber and plastic gloves often have a film on their exterior that will come off onto equipment, so they are not to be used on the pycnometer. Finger prints will compromise the measurements. Likewise changes in the barometric pressure during the measurement or calibration will cause a loss of accuracy.

Principal of Operation

This pycnometer is a device that provides the density of small samples. It actually measures the sample volume and incorporates the sample mass, from an external measurement, into its software to give the sample density.

Per MicroMeritics

The AccuPyc works by measuring the amount of displaced gas [helium]. The pressures observed upon filling the sample chamber and then discharging it into a second empty chamber allow computation of the sample solid phase volume. Gas molecules rapidly fill the tiniest pores of the sample; only the truly solid phase of the sample displaces the gas.

A sample chamber of 0.1 or 1 cc can be selected to provide the best fit with your samples. The run precision mode allows you to achieve high repeatability. The instrument automatically purges water and volatiles from the sample

User Notes

- 1) Because the instrument requires two hours for full warm up and stabilization it should be left on all of the time. Likewise the He supply should be left on.
- 2) Users must ALWAYS FABRIC WEAR GLOVES when handling any internal component of the Pycnometer including the sample cups or calibration standard.
- 3) There is a special tool for handling the sample cup, be sure to use it.
- 4) The calibration standard is a 3/8" silicon carbide sphere with a volume of 0.452254 cm^3 . Load it into the machine using soft tip tweezers. Remove it with a plastic covered magnet probe or use the special tool to remove both the sample cup and the sphere. Always have its box underneath it when moving so that should it drop, it will only fall a short distance and will fall onto soft foam. The calibration standard would be ruined by a three foot fall onto a hard floor.
- 5) The button labeled as a period, may appear as a period, a dash, or as a slash depending on the screen in use.

- 6) Setting a large number of purge cycles will facilitate sample drying at the cost of more time. Five purges is enough for dry samples.
- 7) The volume of closed internal pores will be measured as part of the sample volume. The volume of any exposed pores, which are available to helium, will not be measured as part of the volume.
- 8) The instrument must be calibrated with each use or if the barometric pressure changes. The calibration is specific to the individual sample cell to be used.
- 9) The calibration procedure is different for the two sample cell sizes. For the 1.0 cm³ cell see page 4-15 of the manual, and for the 0.1 cm³ cell, page 3-2.
- 10) System precision and accuracy are enhanced by using enough sample to nearly fill the sample cup.
- 11) Error messages are described on pages 5-4 to 5-12 of the manual.

Mettler AG245 Balance

There is a Mettler balance in the same room as the pycnometer. It shares a balance table with the TGA. Do not use the balance when the TGA is measuring because the vibration will show up on the TGA plot.

Before use the balance should be tared, with the bar at the bottom of the instrument; then the scale should be switched to 5 digit with by hitting the 1/10 d tab. After it comes to rest at 0.00000 grams, press and hold the 1/10 d tab until the Auto Calibration sequence begins.

The balance is now ready to use.

Operation

All page numbers refer to the MicroMeritics AccuPyc 1330 Manual.

Calibrate

Make sure the sample chamber is clean by blowing it out with an air bulb. Load a clean and empty 1 cm³ sample cup into the sample chamber, hit the WHITE button then the CALIBRATE button, then accept the calibration standard volume of 0.452254 cm³ by hitting the ENTER button, then hit the ENTER button again at the prompt. The instrument will do a series of measurements and eventually prompt the user to load the calibration standard, do so carefully and hit the ENTER button. When done, remove the sample cell and the calibration standard. A report will be printed.

See also the main Manual pages 4-15, or page 3-2 for 0.1 cm³ cell.

Prep Sample page 3-5, a drying oven is available in room 1013.

Start Analysis Be sure that there are no sample particles on the exterior of the sample cup. See following page, and pages 3-8 & 4-6 in the full manual

To Cancel Run WHITE button then CLEAR button

More Information and Technical Notes

http://www.micromeritics.com/d_accupyc.html

AccuPyc 1330 Accuracy & Precision Specifications from MicroMeritics

Precision:

Typical reproducibility to within (0.01% of the nominal full-scale sample chamber volume. Guaranteed reproducibility to within 0.02% of the nominal full-scale volume on clean, dry, thermally equilibrated samples.

Accuracy:

Accurate to within 0.03% of reading plus 0.03% of nominal full-scale sample chamber volume.

See the MicroMeritics Quick Reference Guide

12/14/2000 Questions to David Gilley of MicroMeritics on initial set up of AccuPyc 1330

Hi Joe, I will respond in BOLD below to each of your questions so that you can distinguish my comments from your questions. Feel free to contact me again if any of my answers need further clarification.

Dave

1) When we use the 0.1 cm³ insert, that is only inside the 1.0 cm³ sample cup, correct?

NO. THE 0.1 CC INSERT IS USED IN PLACE OF THE 1 CC CUP.

2) Is there a calibration standard for the 0.1 cm³ sample insert, or do we calibrate with the one for the 1.0 cm³ cell and assume that the lower volume is also calibrated.

YES. YOU CALIBRATE WITH THE 1 CC CUP AND THEN MEASURE THE VOLUME OF THE 0.1 CC INSERT AND CUP WITH THE INSTRUMENT AND THEN SUBTRACT THE VOLUME OF THE SMALL INSERT FROM THE CALIBRATION OF THE SAMPLE CELL. THIS CORRECTED NUMBER IS INSERTED INTO THE CALIBRATION MENU. IF THE PROCEDURE ISN'T CLEAR IN THE MANUAL (OR MY MESSAGE) WE CAN DISCUSS IT ON THE PHONE.

3) In the manual Page 3-2, gloved hands are shown holding the sample cup handling tool, but the manual does not say to use gloves to handle this tool. How should we approach the issue of when to wear gloves?

DUE TO THE NATURE OF THE SMALL MASS OF SAMPLE USED BY THIS INSTRUMENT, IT IS CRITICAL TO PREVENT FINGERPRINTS FROM ADHERING TO ANY COMPONENT BEING WEIGHED. THE SAMPLE CELL MUST BE HANDLED WITH GLOVES OR TWEEZERS OR THE TOOL. SINCE THE TOOL IS NOT WEIGHED IT DOESN'T MATTER IF FINGER PRINTS ADHERE TO IT.

4) For 1 out of 3 tries to print, we get an error, " Printer Port Not Responding". The printer is a new Epson 440.

THIS PROBLEM HAS BEEN REPORTED BY SOME CUSTOMERS RECENTLY WITH NEW PRINTERS. THIS ERROR NEEDS TO BE REPORTED TO SERVICE (770)662-3666. THEY WILL KNOW HOW TO CORRECT IT.

5) Calibration of the 1.0 cm³ cell is described starting on page 4-15 of the manual. One step in the process is to insert the calibration standard into the cell chamber.

-Is there a good or bad way to handle the steel ball that is the calibration standard? Gloved hands? Plastic tweezers? I would think that greasy hands or metal pliers would be bad.

YES. BOTH GLOVED HANDS AND PLASTIC TWEEZERS ARE NECESSARY WHEN HANDLING THE CALIBRATION STANDARD. THIS STANDARD SHOULD ALSO BE STORED IN THE WOODEN BOX TO PREVENT CORROSION- ESPECIALLY IN YOUR CORROSIVE ENVIRONMENT BY THE WATER.

-Does the calibration ball go inside one of the 1.0 cm³ sample cups or just inside the well without a cup?

THE CUP VOLUME (METAL VOLUME NOT INTERIOR CUP VOLUME) IS ALWAYS PART OF THE CALIBRATION. THEREFORE, ALL MEASUREMENTS ARE MADE WITH THE CUP - STANDARDS AND SAMPLES.

6) On page 4-15 of the manual it says to check the Pycnometer calibration 4 times per day, but calibrating it took about an hour and a half for 5 repetitions and the manual recommends 10 repetitions. Doing 4 calibrations a day does not leave much time to actually measure anything. Am I missing something?

I AGREE THAT THIS RECOMMENDATION IS EXCESSIVE. HOWEVER IT DOES ILLUSTRATE THE IMPORTANCE OF CALIBRATION TO THE MEASUREMENTS WITH ULTRA SMALL VOLUMES.

7) How often does the pressure transducer need to be zeroed? Before each calibration?

NEVER. THE INSTRUMENT AUTOMATICALLY RE-ZEROES THE TRANSDUCER BEFORE EVERY CALIBRATION CYCLE AND EVERY SAMPLE RUN. NO NEED TO EVER RE-ZERO UNLESS YOU ARE TROUBLE SHOOTING.

8) As I understand it, the calibration procedure actually measures the real cell volume and enters that into the tables for subsequent measurements. Correct?

TWO NUMBERS ARE AUTOMATICALLY SAVED IN THE CALIBRATION MENU AFTER THE INSTRUMENT IS CALIBRATED: THE CELL VOLUME AND THE EXPANSION VOLUME. THESE NUMBERS ARE AUTOMATICALLY APPLIED

TO EVERY MEASUREMENT THE INSTRUMENT TAKES UNTIL THE CALIBRATION IS RUN AGAIN.

9) The only way I could get the standard ball out of the sample cup inside the Pycnometer was with a magnet. Is there a better way?

YES. USE THE SAMPLE CUP HANDLING TOOL TO LIFT THE CUP OUT OF THE INSTRUMENT AND TO PLACE THE POWDER FILLED CUP BACK INTO THE INSTRUMENT.

10) What about porous samples? It seems like large accessible pores would not be counted as part of the measured sample volume. But that as the pores get smaller, at some point the pore volume would be measured as part of the sample volume. Could you please explain this and if possible say at what pore size the cut off is?

YOU ARE CORRECT. ANY PORE THAT IS ACCESSIBLE TO THE HELIUM WILL NOT BE INCLUDED IN THE VOLUME OF THE MEASUREMENT. THIS INCLUDES THE INTRA-PARTICLE VOLUMES OF A POWDERED SAMPLE. THIS IS WHY THE TECHNIQUE WORKS SO WELL WITH POWDERS. HELIUM WILL GET THROUGH CRACKS AND CREVICES THAT ARE MUCH SMALLER THAN THE HELIUM MOLECULE DIAMETER.