

Ultraprecise Calibration for the AccuPyc 1330

We do not warrant this technique to improve performance beyond the published specifications for the instrument, but many users will find this technique useful in extending the precision and accuracy of the AccuPyc 1330 pycnometers by several fold. The technique involves two parts:

- Correcting for Residual Zero Offset
- Correcting for Residual Scale Factor Errors

The AccuPyc 1330 pycnometer's automatic calibration routine typically achieves calibration to within $\pm 0.02\%$ of true zero and true scale factor (of the nominal full-scale range) of the pycnometer. For a 10-cc pycnometer this allows an uncertainty of ± 0.002 cc on an empty sample chamber which should read exactly zero. Also, the scale factor could cause a sample of exactly 10.000 cc to read between 9.998 cc and 10.002 cc, and, if the zero error is included, the reading could be between 9.996 cc and 10.004 cc.

Some users may wish to run a sample having a small fraction of the pycnometer full-scale range, for example 0.100 cc, and may consider an error of ± 0.002 cc due to zero error unacceptable. The following procedure allows you to tune the zero error out manually to a level approaching the noise level of the instrument, sometimes as low as ± 0.0002 cc. When a known larger volume sample (for example a 10.0000-cc standard) is being analyzed, you can force it to read to much closer limits with this technique, possibly to a range of 9.9996 cc to 10.0004 cc.



A reasonably constant-temperature environment must be maintained in the laboratory. An unstable temperature may affect the accuracy of results.

Perform all the steps in each procedure each time you wish to perform ultraprecise calibration.

Correcting for Residual Zero Offset

1. Press **Blank** key + **2** to display cell and expansion volumes.
2. Select *calibration parameters* at the **Setup type** prompt. Record the cell volume below:

Original cell volume: _____

3. Press **Enter** to display the expansion volume. Record the value below:

Original expansion volume: _____

4. Press **Save**, then press **Blank** key + **2** to display analysis parameters.

5. Enter the following analysis parameters:

Number of purges: 20
Purge fill pressure: 19.50 psig
Number of runs: 20
Run fill pressure: 19.50 psig
Equilibration rate: 0.005 psig/min.
Use run precision? yes
Percent full-scale: 0.01%

6. Press **Save** to retain the information entered.

7. Place an empty sample cup in the AccuPyc's sample chamber.

8. Press **Blank** key + **4** to perform the analysis.

9. Enter 1.000 gram for sample weight, then press **Enter** to start the analysis.

10. View the analysis results. Record the **Cell volume** below as the average cell volume #1:

Average cell volume #1: _____

11. Perform the following calculation:

New cell volume #1 = Original cell volume - Average cell volume #1

Enter the result of the calculation below:

New cell volume #1: _____

12. Press **Blank** key + **2** to edit calibration volumes.

13. Select *calibration parameters* at the **Setup type** prompt.

14. Enter the **New cell volume #1** calculated in Step 11 at the **Cell volume** prompt.

Correcting for Residual Scale Factor Errors

15. Place a calibration standard in the sample cup and insert the cup in the sample chamber.
16. Use the same analysis parameters entered above.
17. Press **Blank** key + **4** to perform the analysis.
18. Enter 1.000 gram for sample weight, then press **Enter** to start the analysis.
19. View the analysis results and record the **Cell volume** displayed as the average calibration volume #2:

Average calibration volume #2: _____

20. Calculate the **New cell volume #2** and the **New expansion volume** as follows:

$$\text{New cell volume \#2} = \text{New cell volume \#1} \times \frac{\text{Calibration standard volume}}{\text{Average calibration volume \#2}}$$

New cell volume #2: _____

$$\text{New expansion volume} = \text{Original expansion} \times \frac{\text{Calibration standard volume}}{\text{Average calibration volume \#2}}$$

New expansion volume: _____

21. Press **Blank** key + **2** to edit calibration volumes.
22. Select *calibration parameters* at the **Setup type** prompt.
23. Enter the **New cell volume #2** calculated in Step 20 at the **Cell volume** prompt.
24. Enter the **New expansion volume** calculated in Step 20 at the **Expansion volume** prompt.

It should be noted that those users requiring ultraprecision in sample volume measurements should perform these procedures alternately with analyzing samples. As a minimum, the empty chamber volume error should be checked between samples to ensure that it remains insignificant relative to the precision required.