AccuPyc 1330 Pycnometer

Operator's Manual
V3.03

Part No. 133-42808-01
March 2001

AccuPyc is a trademark of Micromeritics Instrument Corporation.

WARRANTY

MICROMERITICS INSTRUMENT CORPORATION warrants for one year from the date of shipment each instrument manufactured by it to be free from defects in material and workmanship impairing its usefulness under normal use and service conditions except as noted herein.

Our liability under this warranty is limited to repair, servicing and adjustment, free of charge at our plant, of any instrument or defective parts, when returned prepaid to us, and which our examination discloses to have been defective. The purchaser is responsible for all transportation charges involving the shipment of materials for warranty repairs. Failure of any instrument or product due to operator error, improper installation, unauthorized repair or alteration, failure of utilities, or environmental contamination will not constitute a warranty claim. The materials of construction used in MICROMERITICS instruments and other products were chosen after extensive testing and experience for their reliability and durability. However, these materials cannot be totally guaranteed against wear and/or decomposition by chemical action (corrosion) as a result of normal use.

Repair parts are warranted to be free from defects in material and workmanship for 90 days from the date of shipment.

No instrument or product shall be returned to MICROMERITICS prior to notification of alleged defect and authorization to return the instrument or product. All repairs or replacements are made subject to factory inspection of returned parts.

MICROMERITICS shall be released from all obligations under its warranty in the event repairs or modifications are made by persons other than its own authorized service personnel unless such work is authorized in writing by MICROMERITICS.

The obligations of this warranty will be limited under the following conditions:

1. Certain products sold by MICROMERITICS are the products of reputable manufacturers, sold under their respective brand names or trade names. We, therefore, make no express or implied warranty as to such products. We shall use our best efforts to obtain from the manufacturer, in accordance with his customary practice, the repair or replacement of such of his products that may prove defective in workmanship or materials. Service charges made by such manufacturer are the responsibility of the ultimate purchaser. This states our entire liability in respect to such products, except as an authorized person of MICROMERITICS may otherwise agree to in writing.

2. If an instrument or product is found defective during the warranty period, replacement parts may, at the discretion of MICROMERITICS, be sent to be installed by the purchaser, e.g., printed circuit boards, check valves, seals, etc.

3. Expendable items, e.g., sample tubes, detector source lamps, indicator lamps, fuses, valve plugs (rotor) and stems, seals and O-rings, ferrules, etc., are excluded from this warranty except for manufacturing defects. Such items which perform satisfactorily during the first 45 days after the date of shipment are assumed to be free of manufacturing defects.

Purchaser agrees to hold MICROMERITICS harmless from any patent infringement action brought against MICROMERITICS if, at the request of the purchaser, MICROMERITICS modifies a standard product or manufactures a special product to the purchaser's specifications.

MICROMERITICS shall not be liable for consequential or other type damages resulting from the use of any of its products other than the liability stated above. This warranty is in lieu of all other warranties, express or implied, including, but not limited to the implied warranties of merchantability or fitness for use.

One Micromeritics Drive • Norcross, GA 30093-1877 • Fax: (770) 662-3696

Domestic Sales — (770) 662-3633
International Sales — (770) 662-3660
Domestic Repair Service — (770) 662-3666
Customer Service — (770) 662-3636

Form No. 008-42104-00
Rev. 12/95

TABLE OF CONTENTS

1. GENERAL DESCRIPTION

2. INSTALLATION

3. PERFORMING AN ANALYSIS

4. GENERAL OPERATING INSTRUCTIONS

5. TROUBLESHOOTING AND MAINTENANCE

6. ORDERING INFORMATION

APPENDICES

INDEX

CHAPTER 1

GENERAL INFORMATION

- Organization of the Manual
- Conventions
- Equipment Description

GENERAL DESCRIPTION

Organization of the Manual

This manual describes how to install, operate, and maintain the AccuPyc 1330 Pycnometer. The manual is divided into six chapters.

Chapter 1 Provides a general description and specifications of the pycnometer.

Chapter 2 Provides unpacking and inspection information, and installation instructions.

Chapter 3 Provides instructions for performing an analysis.

Chapter 4 Provides detailed operating instructions.

Chapter 5 Provides troubleshooting and maintenance procedures and error messages.

Chapter 6 Provides information on ordering parts and accessories for the pycnometer.

Several appendices are also included.

Conventions

This manual uses the icons shown below to identify notes of importance, cautions, and warnings.

Notes contain information to help you operate the instrument more effectively.

Cautions contain information to help you prevent actions which could damage the instrument.

Warnings contain information to help you prevent actions which could cause personal injury.

Equipment Description

The AccuPyc 1330 Pycnometer is an easy-to-use, fully-automatic gas displacement pycnometer. Analyses are initiated with a few keystrokes. Once an analysis is initiated, data are collected, calculations performed, and results displayed without further operator intervention. The pycnometer may be operated in any of five languages: English, French, German, Italian, or Spanish.

The AccuPyc 1330 Pycnometer's unique run precision feature increases the precision of analysis results by reporting data from five consecutive runs that are within a user-specified tolerance. This feature allows early termination of analyses, thereby decreasing the number of runs needed for accurate results.

The AccuPyc 1330 Pycnometer, shown in the following illustration, contains a keypad, a display area, and an analysis chamber (referred to as the cell chamber). The pycnometer is controlled by commands entered through the keypad. The operational status of the pycnometer can be continually monitored on the display. The optional printer prints out complete analysis and calibration results. A serial port is provided for electronic data transmission.

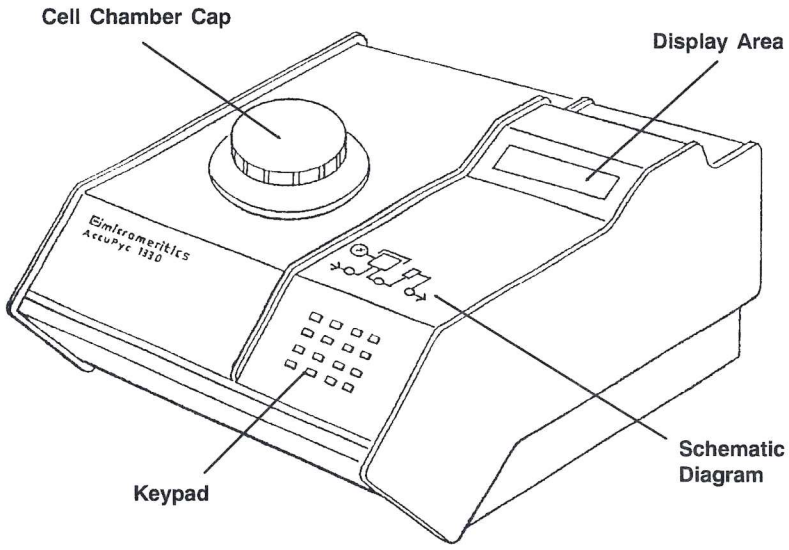


Figure 1-1. AccuPyc 1330 Pycnometer

The AccuPyc 1330 Pycnometer determines density and volume by measuring the pressure change of helium in a calibrated volume. It also reports the chamber temperature at the end of the requested runs. The schematic diagram, which is included above the keypad, indicates system status. The three indicators show the current state of the fill, expansion, and vent valves. The indicator is lit when a valve is open.

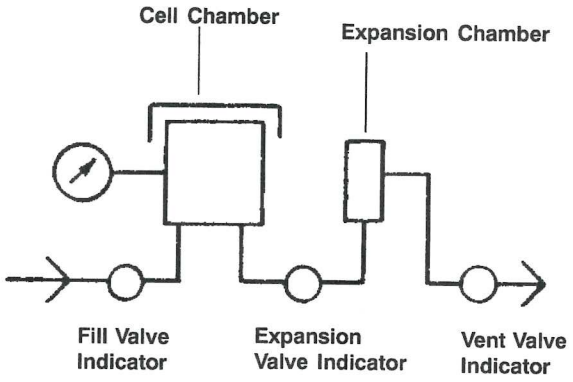


Figure 1-2. Schematic Diagram

In addition to analysis, there are two other automatic operations performed by the pycnometer: calibration and transducer zero reset.

Analysis measures sample volume, from which density can be derived automatically if sample weight has been entered. The unit comes pre-programmed with default conditions and ready to perform analyses. It can be easily reprogrammed to meet your specific needs. You can modify your own analyses by entering the following parameters: number of purges, purge fill pressure, number of runs, run fill pressure, equilibration rate, and run precision. The run precision option allows early termination of the analysis if the last five runs are all within a user-specified tolerance. By modifying these parameters, you retain control over the two main sections of the analysis: purge and run.

A purge is used strictly for sample clean up and air and moisture removal from the chamber's inside. It is accomplished by closing off the pycnometer block and filling the cell chamber to the designated purge fill pressure. The chambers are then vented to atmosphere, resulting in elimination of water vapor or other contaminants. A run is used for collecting the precise, accurate data used in report calculations.

Calibration is used to determine the size of the cell and expansion chambers within the instrument. After calibration, the cell and expansion chamber volumes are automatically stored in the set-up parameters.

The zero function is used to calculate a new zero pressure offset. It is measured when the system is at atmospheric pressure and thermal equilibrium and is subtracted from all subsequent pressure readings in order to obtain a true gauge pressure.

Pycnometer Keypad

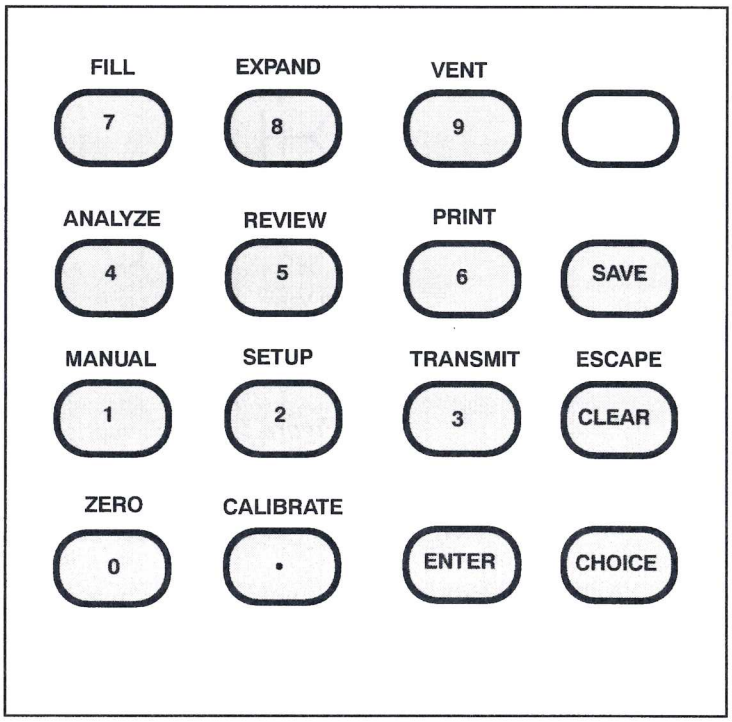
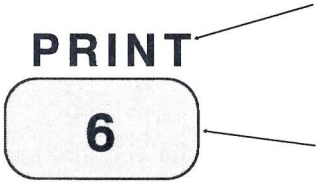


Figure 1-3. Pycnometer Keypad

Most keys on the keypad perform one primary and one alternate function. The primary function of any key is indicated by the number or command on the face of the key. The alternate function is indicated by the command above the key.



To select the alternate function printed above the key, first press [ ], then press the key. For example: press [ ] [6] to print. Refer to Table 1-2.

To select the primary function on the face of the key, press only that key. For example: press [6] to enter the number 6. Refer to Table 1-1.

Tables 1-1 and 1-2 describe how the keys are used to control and monitor the pycnometer.

Table 1-1. Standard Keys

Key	Used To
0 - 9	Enter the numbers 0 through 9.
.	Enter a decimal point, a dash for sample ID, a slash for date, or a colon for time.
ENTER	Complete an entry or begin an action.
CHOICE	Display the next message when in display mode. Display the next multiple choice item when in a command mode.
CLEAR	Clear a message when in display mode. Clear an entry when in a command mode.
SAVE	Save the information you entered and return to display mode.
	Change the keypad mode. The keypad may be used in either primary function mode or alternate function mode. When you press the white key to enter alternate function mode, a plus sign (+) appears in the upper right corner of the display and the commands written above the keys become available. To exit alternate function mode, press the white key again. The plus sign (+) will be removed from the display.

Table 1-2. Key Combinations

Function	Keys	Used To
ZERO	[ ] [0]	Zero the pressure transducer.
CALIBRATE	[ ] [•]	Calibrate the pycnometer.
MANUAL	[ ] [1]	Manually control the valves. After pressing the Manual key, you may use the FILL, EXPAND, and VENT commands to open and close the valves.
SET UP	[ ] [2]	Display or edit analysis parameters, report options, calibration data, data transmission parameters, unit types, operating language, and date and time.
TRANSMIT	[ ] [3]	Transmit analysis or calibration data over the serial line. If an automatic operation is in progress, transmit a partial report.
ESCAPE	[ ] [CLEAR]	Delete all data entered in the current mode and return to display mode. If an automatic operation is in progress, cancel the operation.
ANALYZE	[ ] [4]	Perform an analysis.
REVIEW	[ ] [5]	Review or edit completed analysis or calibration data.
PRINT	[ ] [6]	Print analysis or calibration report. If an automatic operation is in progress, print a partial report.
FILL	[7] (when in manual mode)	Open and close the fill valve. The indicator above the FILL key is on when the valve is open and off when the valve is closed.
EXPAND	[8] (when in manual mode)	Open and close the expansion valve. The indicator above the EXPAND key is on when the valve is open and off when the valve is closed.
VENT	[9] (when in manual mode)	Open and close the vent valve. The indicator above the VENT key is on when the valve is open and off when the valve is closed.

Specifications

Table 1-3. AccuPyc 1330 Pycnometer Specifications

Characteristic	Specification
Temperature	Stable between 15 and 35 °C (59 to 96 °F)
Humidity	20 to 80% relative, non-condensing
Voltage	90 to 264 VAC
Power	30 VA
Frequency	50/60 Hz
Sample Volume	0.5 - 100 cm^3
Standard Sample Holder	19 mm OD x 39.8 mm long x 0.254 mm wall (0.750 in. OD x 1.570 in. long ±0.005 in. x 0.010 in. wall) for standard (10 $cm^{3)}$ holder.
Optional Sample Modules	Provide ranges of 1 to 100 cm^3
Precision	Reproducibility typically to within ±0.01% of the nominal full-scale cell chamber volume.* Reproducibility guaranteed 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 cell chamber volume.*
Cabinet	3ll mm W x 173 mm H x 357 mm D (12 in. W x 7 in. H x 14 in. D)
Gases	Research grade helium is recommended. If unavailable, use helium with a dewpoint of -67 °C (-88 °F) or lower.
*Nominal full-scale cell chamber volume is the sample capacity.	

CHAPTER 2

INSTALLATION

- Unpacking and Inspection
- Selecting the Location
- Gas Requirements
- Equipment Setup
- Verifying Operation

INSTALLATION

This chapter describes how to unpack, inspect, and install the AccuPyc 1330 Pycnometer.

Unpacking and Inspection

Lifting the Accupyc 1330

The Accupyc 1330 can be lifted by one person using two hands placed on opposite sides of the instrument. As always, proper lifting techniques should be used to prevent injury.

Unpacking the Cartons

When you receive the shipping cartons, carefully compare the Packing List with the equipment actually received and check the equipment for any damage during shipment. Be sure to sift through all packing materials before declaring equipment missing.

If you need to declare equipment as damaged or lost, save the shipping cartons. The claims investigator must examine the cartons in order to complete the inspection report.

Equipment Damage or Loss During Shipment

If equipment is damaged or lost in transit, you are required to make note of the damage or loss on the freight bill. The freight carrier, not Micromeritics, is responsible for all damage or loss occurring during shipment. If you discover damage or loss of equipment during shipment, report the condition to the carrier immediately.

Equipment Return

Micromeritics strives to ensure that all items arrive safely and in working order. Occasionally, due to circumstances beyond our control, a customer may receive equipment which is not in working order. When equipment has been damaged (either during shipment or in use) and you wish to return the equipment to Micromeritics for repair or replacement, please follow the steps below:

1. Tag or otherwise identify the defective equipment, noting the defect and, if possible, the circumstances under which the defect occurs.

2. Make reference to the sales order or purchase order for the equipment, and provide the date the equipment was received.

3. Notify a Micromeritics Service Representative of the defect and request shipping instructions. The Service Department will assign a Return Material Authorization (RMA) number to your return and provide shipping information.

Equipment Setup

The pycnometer should be checked to make sure it is operating properly before actual analyses are attempted. The remainder of this chapter describes how to install the pycnometer and verify operation.

Selecting the Location

When selecting the location of the pycnometer, keep the following in mind:

- The pycnometer performs best in a constant temperature environment.
- It should be installed on a workbench about 36 in. high in a location free of drafts from either a forced-air heating or cooling system.
- The slots in the baseplate, which provide ventilation, should not be blocked.
- The pycnometer should not be placed near a window; exposure to sunlight may cause the temperature to vary.

Gas Requirements

The pycnometer uses helium (99.995% pure or better) to provide rapid, accurate analyses. The cylinder containing helium must be fitted with a gas regulator and the regulator set for 15 to 23 psig (we recommend 20-22 psig). In no instance should the pressure input to the pycnometer be greater than 23 psig. Excessive pressures waste gas due to a protection device contained in the pycnometer that vents the pycnometer to atmospheric pressure if pressure exceeds 25 psig.

Minimum recommended helium tank pressure is 200 psig.

Installing the Power Cord

Locate the female end of the power cord and insert it into the power entrance on the rear panel of the instrument.

The power cord supplied with your instrument is a shielded cord (as are the optional RS-232 and printer cords). If you alter or replace any of these cords with non-shielded cords, your instrument may no longer conform to the European Union Council Directives (CE Mark).

Rear Panel Connections

1. Attach an appropriate regulator to the gas supply cylinder. Leave the gas cylinder shut-off valves closed until instructed otherwise.

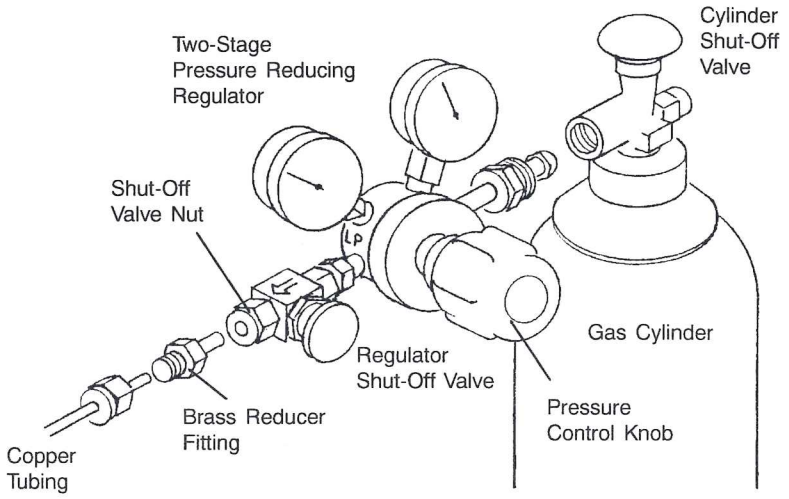


Figure 2-7. Connecting Gas Supply Cylinder to the Regulator

2. Attach the supplied brass reducer fitting to the outlet of the regulator shut-off valve.

3. Turn the shut-off valve nut clockwise until it is finger tight. Then use a 9/16-in. open-ended wrench (or an adjustable wrench) to tighten the nut an additional 1/4 turn.

Do not overtighten the fittings. Doing so can collapse the brass fitting and cause a leak.

4. Attach the copper tubing to the brass reducer fitting.

5. Turn the nut on the tubing clockwise until it is finger-tight. Then use a 7/16-in. open-ended wrench (or an adjustable wrench) to tighten the nut an additional 1/4 turn.

6. Attach the other end of the copper tubing to the fitting on the INLET connector on the back of the pycnometer.

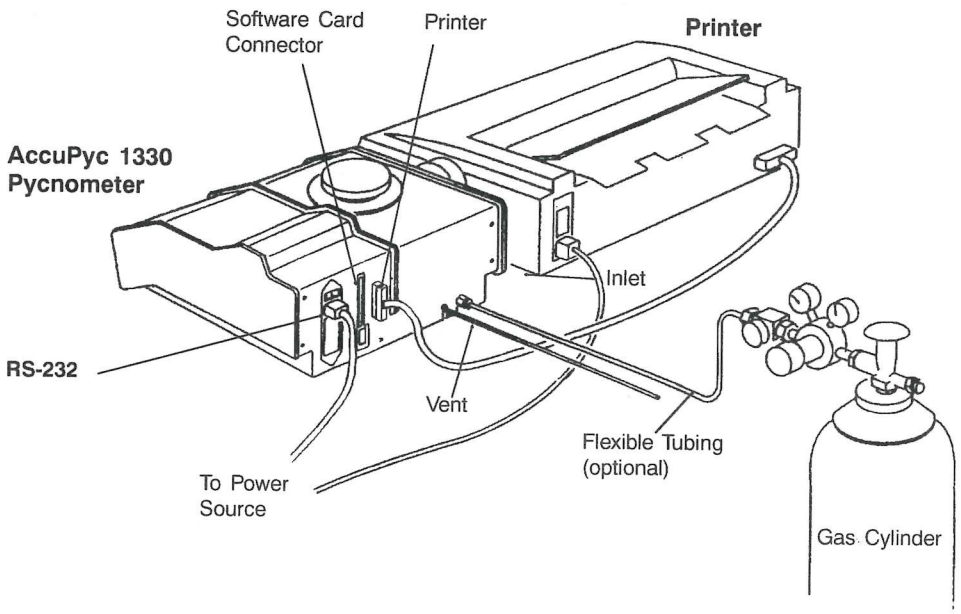


Figure 2-8. Rear Panel Connections

7. Turn the nut on the tubing clockwise until it is finger-tight. Then use a 7/16-in. open-ended wrench (or an adjustable wrench) to tighten the nut an additional 1/4 turn.

8. A port labeled VENT is provided on the rear panel. If you are using a gas other than helium and wish to vent the system, use this port. Connect one end of 1/8-in. ID flexible tubing to the rigid tubing labeled VENT and the other end to a venting system.

9. If a serial line is to be used for data transmission, connect one end of the RS-232 cable to the connector labeled RS-232 on the back of the pycnometer. Connect the other end to the output device.

10. Connect one end of the printer cable to the connector labeled with the printer icon on the back of the pycnometer. Connect the other end to the input connector on the printer.

Refer to *Appendix E: Printer Character Set* for the print characters supported by the software.

Before inserting the software card, make sure the instrument is turned OFF (O). Do NOT install or remove the software card while the power is turned ON.

11. Hold the AccuPyc 1330 Software Card with the label facing up. Insert the card (notched end first) into the connector on the rear panel of the pycnometer and press firmly to lock into place.

The software card must be properly inserted in the pycnometer connector during operation. It is best to keep it installed at all times, even when power to the unit is turned off.

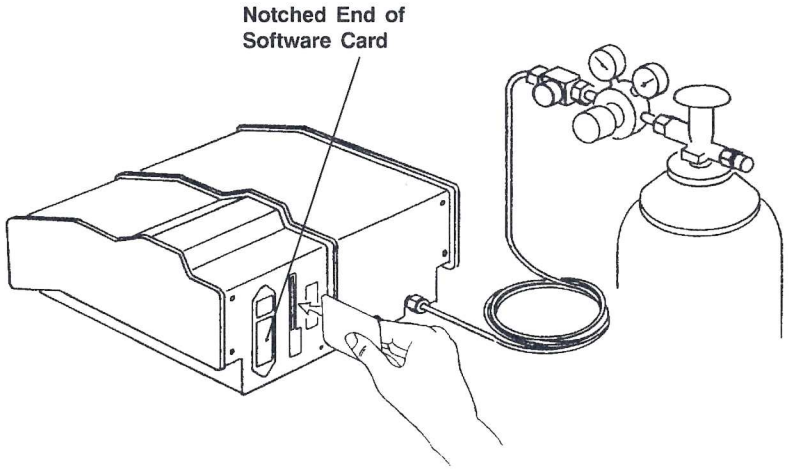
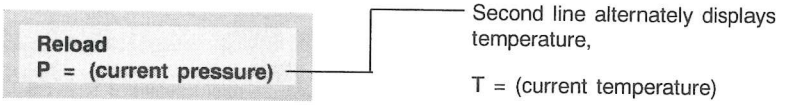


Figure 2-9. Inserting Software Card

12. Plug in the printer power cord and the pycnometer power cord.

Turning On the Pycnometer

To turn on the pycnometer, place the power ON/OFF switch on the rear panel in the ON (|) position. The system is automatically vented and the following prompt is displayed:



Reload is displayed on the first line, indicating that the system is idle. The current pressure and current temperature are shown alternately on the second line. There may be messages containing the results of a previous analysis or the status of the pycnometer. If there are any error messages, an asterisk appears in the right corner of the first line. When this occurs, press CHOICE to display the first message. Continue pressing CHOICE to cycle through all messages and return to the **Reload** prompt.

When the system is turned on the first time or after a period of non-use, it should be allowed to warm up for at least 30 minutes before analyses are performed. For analyses that require very precise results, it should warm up for two hours.

It is important that a constant temperature be maintained inside the unit because a change in temperature could alter analysis results. We recommend that the pycnometer remain turned on at all times to maintain thermal stability. Power consumption is small and the cost of electricity will be minimal.

Entering Report and Analysis Parameters

The pycnometer is shipped with the following default values for analysis parameters:

Number of purges	3
Purge fill pressure	19.5 psig
Number of runs	3
Run fill pressure	19.5 psig
Equilibration rate	0.005 psig/min
Run precision	No
Percent full scale	0.05

If you wish to use these values, no further action is necessary. If you wish to change the values, refer to **Analysis or Calibration Parameters** in Chapter 4.

The pycnometer is shipped with the following default values for report options:

Request sample ID	no
Analysis display mode	volume
Report destination	display
Transmission format	Single column

If you wish to use these values, no further action is necessary. If you wish to change the values, refer to **Report Options** in Chapter 4.

Selecting Units of Pressure Measurement and Operating Language

The units of measurement for pressure may be displayed in pounds-per-square-inch gauge (psig) or kilopascal gauge (kPag). In this manual, psig is used in the examples shown. The pycnometer may be operated in any of five languages: English, French, German, Italian, or Spanish. The default for units is **psig** and the default language is **English**. If you wish to use these defaults, no further action is necessary; proceed to **Greasing the Chamber Cap O-Ring**. If you wish to change the defaults:

1. Press [ ] [2], then press [CHOICE] until **Unit types** is displayed. Press [ENTER].

2. Press [CHOICE] then [ENTER] to change the value to kPag.

3. The **Language** prompt is displayed. Press [CHOICE] until the desired language is displayed, then press [SAVE].

If there are error messages in the message queue (indicated by an asterisk next to the Reload prompt), delete the messages before changing languages. Refer to *Error, Report, and System Status Messages*, Chapter 4. Make sure you press [SAVE] after changing languages.

4. Place the appropriate template (included in the accessory kit) over the pycnometer keypad.

Greasing the Chamber Cap O-Ring

A greasing disk is included in the accessory kit shipped with the pycnometer. Use the disk to grease the O-ring as described below.

Rubber gloves should be worn to prevent contaminating the grease with oil from your fingertips. Carefully apply a thin layer of grease; both too much and too little grease can cause problems. Too much grease may alter cell volume. Too little grease results in an imperfect seal and leaks.

1. Remove the chamber cap by turning it counterclockwise then lifting off.
2. Using your fingertip, apply a thin layer of Dow Corning high vacuum grease (or equivalent) to the greasing disk.
3. Insert the greasing disk into the chamber cap and turn slightly to apply a very light coating of grease to the O-ring.

Apply a VERY LIGHT coating of grease to the O-ring. There should be no visible ridges of grease; excess grease may alter analysis results.

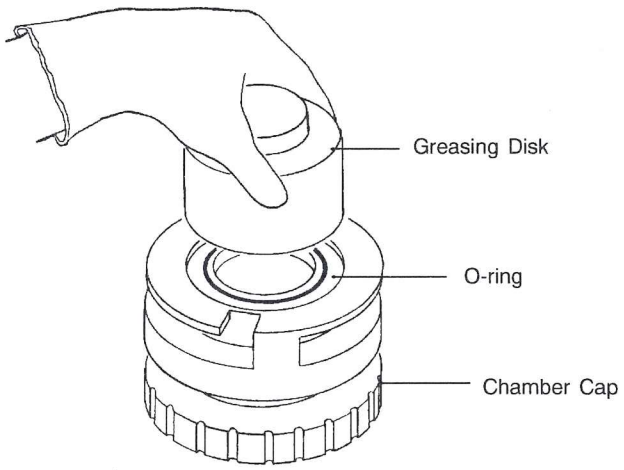


Figure 2-10. Greasing the O-Ring in Chamber Cap

4. Replace the chamber cap.

Verifying Operation

Before performing an analysis, verify that the pycnometer is operating properly by performing the following steps.

Numbers are displayed on the pycnometer as shown in the following example:

10000.00

Note that a period is used to denote a decimal fraction.

1. Press [ ] [0] to zero the transducer.
2. Once the zeroing process is complete (the pressure display shows approximately zero), press [ ] [1] on the keypad to enter manual mode.
3. Use the [FILL], [EXPAND], and [VENT] keys to close all valves. A valve opens and closes when you press the appropriate key. The valve is open when the indicator above the key is on and closed when the indicator is off.
4. Open the fill valve. Wait until the pressure reaches 15 to 19 psig.

Do not exceed 19.9 psig. If the pressure exceeds 20 psig the transducer may overrange and an error message will be displayed. If this occurs, open the expansion and vent valves and allow the transducer to drift back down, then start the procedure over.

5. Close the fill valve.
6. Observe the pressure display. After an equilibration period, the pressure should not vary more than 0.005 psig/min.
7. Open the expansion valve.
8. Observe the pressure display. After equilibration, the pressure should not vary more than 0.005 psig/min. Open the vent valve. After a period of time, the pressure should again stabilize.

If the pycnometer responds as described above, it is ready to operate. If it does not, check installation procedures, then repeat the verification procedures. If it still does not respond as described above, service to the system or operational assistance may be required. Contact a Micromeritics Service Representative.

CHAPTER 3

PERFORMING AN ANALYSIS

- Setting Regulator Pressure
- Preparing and Loading a Sample
- Starting the Analysis
- Viewing or Printing Analysis Results

PERFORMING AN ANALYSIS

This chapter briefly describes how to perform an analysis. Chapter 4 describes in detail how to use all the functions of the AccuPyc 1330 Pycnometer.

The cell chamber and cap must be kept clean at all times. Particles on the cap seating surface, in the sample cup, under the sample, or clinging to the sample chamber wall may cause inaccurate results. Inspect the cell and cap before each use. Use a lint-free cloth to remove any dust or particles.

Setting Regulator Pressure

Before setting regulator pressure, make sure the *tank* pressure for the gas regulator is at least 200 psig. Pressures less than 200 psig may cause the sample to be inadequately saturated, resulting in inaccurate data or termination of analysis.

Before beginning an analysis, check the regulator pressure. Fill pressure is user-specified for purge and run using the AccuPyc 1330 **Set Up** function (refer to **Analysis or Calibration Parameters** in Chapter 4. Set the regulator pressure at the higher of the two (plus about 2.0 psig) as follows:

1. Press [ ] [1] to enter manual mode.
2. Press [8] (EXPAND) and [9] (VENT) to open the expansion and vent valves. When the valves are open, the indicators above the keys are turned on.
3. Press [7] (FILL) to open the fill valve.
4. Set the regulator pressure control knob on the cylinder to the desired pressure and then increase it about 2.0 psig.
5. Press [7] (FILL) to close the fill valve.
6. Press [SAVE] to return to display mode.

Preparing and Loading a Sample

Preparing the sample is the first step in obtaining accurate results from the pycnometer. Samples must be free of moisture in order to obtain true sample weight and to avoid the distorting effect of water vapor on the volume measurement. The following procedures are recommended; however, modifications may be necessary for some materials. Heat sensitive materials may have to be dried by long-time exposure to silica gel, freeze drying, etc. Materials having a low melting point may be dried using the purge process. In this case, do not weigh the sample and cup until after the purge and analysis have been completed.

The important point to keep in mind is that each step should be conducted to avoid exposure of the dried sample to atmospheric moisture. This means weighing as rapidly as possible and installing in the instrument without unnecessary delay.

Keep the cap on the cell chamber except when actually inserting or removing a sample. If the chamber remains uncapped, temperature instability will occur which could affect analysis results.

1. Weigh the empty sample cup. (If volume only is to be measured, you may skip this step).

2. Place a quantity of sample in the cup. Use as large a quantity of sample as possible; we recommend that the cup be at least two-thirds full. Pack powders and fluffy materials (if permissible) to obtain maximum sample weight in the cup.

3. Place the sample cup with sample in a drying oven. The amount of time the sample must be heated depends on the material and the temperature it will tolerate; this may have to be established by other tests.

4. Remove the sample cup from the oven and transfer it to a desiccator provided with active desiccant. Allow it to cool until near room temperature. In the steps which follow, minimize air exposure of the sample.

5. Weigh the cup and sample and record the weight. Subtract the empty sample cup weight from the sample cup plus sample weight to determine the sample weight. (If volume only is to be measured, you may skip this step.)

6. Remove the cell chamber cap.

When you remove the chamber cap, place it on a clean, dry surface with the greased side down so that particles will not accumulate on the greased surface. If the cap is placed on a dirty surface, analysis errors may result.

7. Insert the sample cup with sample into the cell chamber.

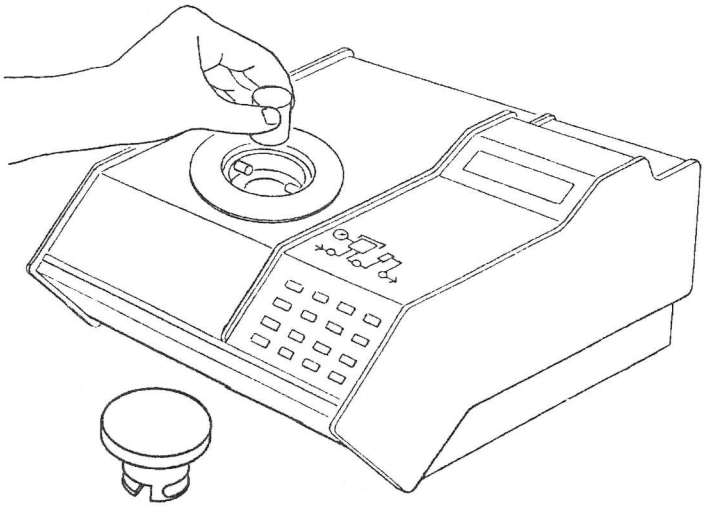


Figure 3-1. Inserting Sample Cup Into Cell Chamber

8. Replace the cell chamber cap.

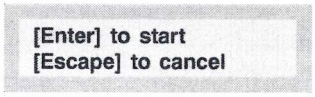
Do not remove the cell chamber cap when the pycnometer is pressurized. Sample may be discharged from the chamber.

Starting an Analysis

Using the Default Parameters

To start an analysis using the analysis and report parameters shipped with the pycnometer:

1. Press [ ] [4].
2. The following prompt is displayed:

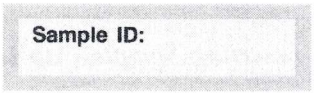


Press [ENTER] to begin the analysis.

Using Modified Analysis Parameters

To start an analysis using analysis parameters that have been modified:

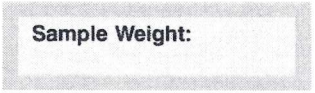
1. Press [ ] [4].
2. The following prompt is displayed if Sample ID was enabled in the Report Options function.



Using sample IDs can help you keep track of data from various analyses. You may, for example, use the sample ID as a date and time stamp. The sample ID can contain from 1 to 20 numbers and dashes.

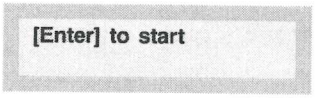
Enter the sample ID and press [ENTER].

3. The following prompt is displayed if density was selected for Analysis display mode in the Report Options function.



Enter the sample weight and press [ENTER]. The range of valid entries is 000.0000 to 999.9999 g.

4. The following prompt is displayed.



Press ENTER to begin the analysis.

Viewing or Printing Analysis Results

1. As the analysis is performed, operational status messages are displayed. (Refer to **Monitoring the System** in Chapter 4 for a description of the messages.)

2. When the analysis is complete, the pycnometer beeps three times. Remove the sample from the cell chamber. Press CHOICE to cycle through the error messages.

3. After the error messages are displayed, the average volume or density and the deviation from the mean are displayed. Press ENTER.

4. A report is automatically printed if report destination was set to *printer*. If not, you may print a report by pressing [ ] 6. You may transmit a report by pressing [ ] 3.

5. When the **Reload** prompt is displayed, you may begin another operation.

CHAPTER 4

GENERAL OPERATING INSTRUCTIONS

- Monitoring the System
- Canceling an Automatic Operation
- Entering Commands
- Printing Reports
- Transmitting Reports
- Calibrating the Pycnometer
- Zeroing the Pressure Transducer
- Manually Controlling the Valves
- Editing the Setup Parameters

GENERAL OPERATING INSTRUCTIONS

The pycnometer remains in the display mode until you enter a command by pressing the appropriate keys. This chapter describes how to read the display and enter commands.

Keep the cap on the cell chamber except when actually inserting or removing a sample. If the chamber remains uncapped, temperature instability will occur which could affect analysis results.

Monitoring the System

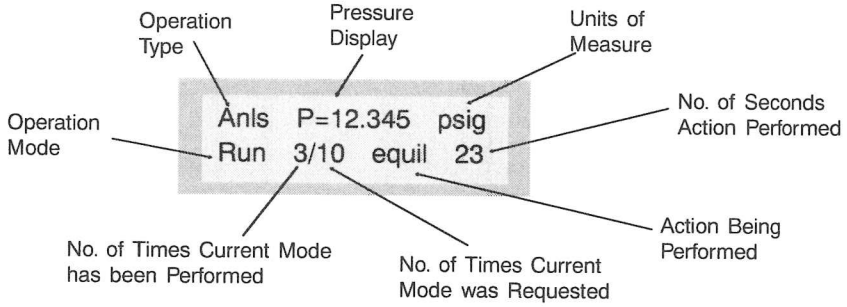
Status Messages Displayed During an Automatic Operation

When an automatic operation is in progress, operational status messages are continually displayed. You may **not** enter commands, other than the following, until the operation is finished.

- Press [ ] [CLEAR] to cancel the automatic operation.
- Press [ ] [6] to print a partial report of analysis or calibration results.
- Press [ ] [3] to transmit a partial report of analysis or calibration results.

Analysis is suspended until printing or transmission is complete.

The following example shows a typical status message for an automatic operation. Refer to Table 4-1 for a detailed description.



When the operation is finished, the pycnometer beeps three times.

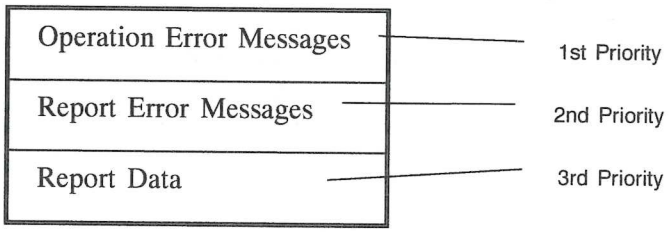
Table 4-1. Automatic Operation Status Messages

Item	Abbreviation Displayed	Description
Operation Type	Anls	Analysis
	Cal 1	Calibration Pass 1, which is the first pass during calibration with the cell chamber empty.
	Cal 2	Calibration Pass 2, which is the second pass during calibration with the calibration standard in the cell chamber.
	Zero	Zero offset
Pressure Display	P	Current pressure in system
	P1	Pressure for data point P1†
	P2	Pressure for data point P2†
Operation Mode	Purg	Purge in progress
	Zero	Transducer zero reset in progress
	Run	Run in progress
Action	fill	Filling the cell chamber or expansion chamber
	equil	Equilibrating
	wait	Wait the displayed number of seconds

† Refer to Appendices B and C.

Error and Report Messages

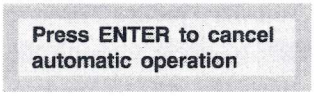
Error and report messages generated during the operation are placed in a queue in chronological order, with error messages having the highest priority (refer to Figure 4-1). When the automatic operation is complete, the **Reload** prompt is displayed. You may press CHOICE to display the first message and continue pressing CHOICE to cycle through the messages. You may press CLEAR while a message is displayed to delete the messsage.



After the operation error messages, report error messages, then report data (average density or volume and deviation from the mean) are displayed. The messages remain in the message queue until you delete them or until another automatic operation is begun, at which time they are automatically deleted.

Canceling an Automatic Operation

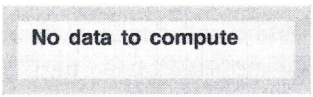
You may cancel an automatic operation that is in progress by pressing [ ] [CLEAR]. The following message is displayed:



Press [ENTER] within five seconds to cancel the operation.

When you cancel an operation, messages are displayed indicating that termination is in progress. The termination process, which vents the system, takes about 30 seconds. You may cancel the termination process, by pressing [ ] [CLEAR] again, but if you do, you must manually vent the system.

If no runs were completed and there are not enough data to compute, the following message is displayed:



If there are enough data to compute and reports were requested, they are printed.

Entering Commands

The pycnometer remains in display mode until you enter a command by pressing the appropriate keys on the keypad. Commands start an analysis or other automatic operations and allow you to modify operating parameters. The commands available are:

Perform an Analysis
Review and Edit Analysis or Calibration Data
Print or Transmit Reports
Calibrate the Pycnometer
Zero the Pressure Transducer
Edit Set-Up Parameters
Manually Control the Valves

You may not enter a command (other than Print or Transmit) while an automatic operation is in progress.

When you enter a command, most functions will display **prompts**. A prompt is a request for you to enter information. A prompt generally contains two lines. The first line contains a description of the requested information and the second line displays a default value (when applicable). To use the default value, just press the ENTER key. For example, when you press [ ] 2 the following prompt is displayed:

Setup type? — Description of Requested Information
Analysis parameters — Default Value

To select **analysis parameters**, which is the default value, press ENTER.

If you do not wish to use the default value, you can use the keypad to enter other responses. There are two types of prompts: data entry and multiple choice. Prompts that require you to enter data are followed by a colon (:). Use the keypad to enter the desired value, then press ENTER. If you enter an invalid value, you will hear a beep. Try again.

Multiple choice prompts contain a fixed set of responses and are followed by a question mark (?). To select a multiple choice response, press CHOICE until the desired value is displayed, then press ENTER.

At any time while entering information you may:

Press SAVE to save the information you entered and return to display mode.

Press [ ] CLEAR to delete the information you entered and return to display mode.

Performing an Analysis

Press [ ] [4] to perform an analysis.

Display	Entry
Sample ID:	This prompt is displayed only when **Sample ID** was enabled in the Set-Up function (refer to **Report Options** later in this chapter). The sample ID can contain from 1 to 20 numbers and dashes. Enter the sample ID and press [ENTER] Press [•] to insert a dash.
Sample Weight: **1.0000g**	This prompt is displayed only if density was selected for Analysis display mode (refer to **Report Options** later in this chapter.) Enter the sample weight and press [ENTER]. *Range: 000.0000 to 999.9999 g*
[Enter] to start **[Escape] to cancel**	Choose one of the following: ■ Press [ENTER] to start the analysis. The analysis begins and operational status messages are continually displayed. ■ Press [SAVE] to store the data you entered and return to display mode. ■ Press [ ] [CLEAR] to cancel the analysis.

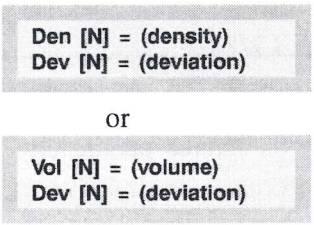
Reviewing and Editing Data

The Review function enables you to review and edit the results of the last analysis or calibration along with its entered parameters.

Reviewing and Editing Analysis Results and Parameters

Press [ ] [5] to review or edit analysis results.

Display	Entry
Sample ID: (sample ID)	The sample ID is displayed if it is enabled in the Set-Up function (refer to **Report Options** later in this chapter). Press [ENTER].
Sample Weight: (weight)	This prompt is displayed only if **density** is selected for Analysis display mode (refer to **Report Options**). The sample weight entered in the **Start Analysis** function is displayed. Press [ENTER].



Either density or volume is displayed depending on the selection made for display mode (refer to **Report Options**). [N] is replaced with the number of the run. The deviation from the mean is also displayed.

You may choose to exclude the displayed density or volume from the calculated average by pressing CHOICE. When a density or volume is excluded from the average, an asterisk appears next to it.

Similarly, a density or volume that was previously excluded from the calculated average may be included by pressing CHOICE. When you press CHOICE, a new deviation is calculated and displayed.

Press ENTER to display values for the next run.

Press SAVE when you wish to exit review and edit mode and return to display mode. When you press SAVE, all collected data are automatically recalculated and all data reduction messages are added back into the queue.

Reviewing and Editing Calibration Results and Parameters

Press [ ] [5] to review or edit calibration results.

Display	Entry
Cal std volume: **(volume)**	The volume of the calibration standard is displayed.
Which chamber? **Cell**	Select either cell chamber or expansion chamber, then press [ENTER].
Cell [N] = (volume) **Dev [N] = (deviation)** or **Exp [N] = (volume)** **Dev [N] = (deviation)**	Either cell or expansion volume is displayed depending on the selection made at the previous prompt. [N] is replaced with the number of the run. The deviation from the mean is also displayed. You may choose to exclude the displayed cell or expansion volume from the calculated average by pressing [CHOICE]. When a volume is excluded from the average, an asterisk appears next to it. Likewise, a cell or expansion volume that was previously excluded from the calculated average may be included by pressing [CHOICE]. When you press [CHOICE], a new deviation is calculated and displayed. Press [ENTER] to display values for the next run. Press [SAVE] when you wish to exit review and edit mode and return to display mode. When you press [SAVE], all collected data are automatically recalculated and all data reduction messages are added back into the queue.

Printing Reports

Reports are generated after analysis and calibration and remain available for viewing or printing until another automatic operation (other than zero) is performed. When you perform an automatic operation, reports from the previous operation are deleted.

The Report Options function enables you to select the destination of the report that is automatically generated after an analysis or calibration. The destination may be: *display*, *printer*, or *transmission line*. A display report is always calculated regardless of the specified destination. The display report for analyses contains average density or volume, depending on the option selected (refer to **Report Options**), and the deviation from the mean. The printed report contains volume, density, and deviation for each run, as well as an average of all runs. Note that if you do not enter a sample weight, the value shown for density will be incorrect. When sample weight is not entered, 1.0000 g is used. The report also contains the date and time the analysis was started and completed and the temperature of the cell chamber.

The display report for calibration contains average cell or expansion volume, depending on the option selected (refer to **Report Options**), and the deviation from the mean. The printed report contains cell and expansion volumes for each run, as well as an average of all runs.

A run with an asterisk next to the run number is not included in the calculations.

Figure 4-2 shows a calibration report. Figure 4-3 shows a report for an analysis performed using the same calibration standard. Figure 4-4 shows a report for an analysis performed with run precision enabled.

To display reports, press CHOICE to cycle through messages until the report messages are displayed.

Press [ ] [6] to print reports. Reports are immediately sent to the printer. If you press [ ] [6] during an automatic operation, a partial report is printed.

You may cancel reports sent to the printer or serial line by pressing [ ] [CLEAR]. You may cancel partial reports that were requested during an automatic operation when the following message is displayed:

```
Print in progress
[Escape] to cancel
```

The following is a calibration report. The calibration standard used must be at least 10% of nominal cell volume. We recommend 60 to 70% for the best results.

Page 1

AccuPyc 1330 VX.XX
Serial Number: XXXXX
Calibration Report

Started: 01/09/92 10:00:00 Completed: 01/09/92 11:15:22
Temperature: 24.5 C
Volume of Calibration Standard: 5.5775 cm3
Number of Purges: 10 Equilibration Rate: 0.0100 psig/min

Run Pair#	Cell Volume cm3	Deviation cm3	Expansion Volume cm3	Deviation cm3
1	12.3844	0.0008	7.9988	0.0003
2	12.3841	0.0004	7.9988	0.0003
3	12.3841	0.0004	7.9993	0.0008
4	12.3834	-0.0003	7.9984	-0.0001
5	12.3844	0.0007	7.9991	0.0006
6	12.3839	0.0002	7.9987	0.0002
7	12.3835	-0.0001	7.9984	-0.0002
8	12.3831	-0.0006	7.9979	-0.0006
9	12.3830	-0.0006	7.9980	-0.0005
10	12.3828	-0.0009	7.9977	-0.0008

Average Cell Volume: 12.3837 cm3 Standard Deviation: 0.0006 cm3
Average Expansion Volume: 7.9985 g/cm3 Standard Deviation: 0.0005 g/cm3

Figure 4-1. Calibration Report

The following report represents an analysis that was performed on the calibration standard used for the previous report.

Page 1

AccuPyc 1330 VX.XX
Serial Number: XXXXX
Density and Volume Report

Sample ID: 11-6-89--12-55
Sample Weight: 42.6966 g
Temperature: 24.5 C
Number of Purges: 20
Cell Volume: 12.3837 cm3

Started: 01/09/92 10:00:00
Completed: 01/09/92 11:18:02

Equilibration Rate: 0.0050 psig/min
Expansion Volume: 7.9985 cm3

Run#	Volume cm3	Deviation cm3	Density g/cm3	Deviation g/cm3	Elapsed Time (h:m:s)
1	5.5767	-0.0005	7.6562	0.0007	0:15:43
2	5.5772	-0.0001	7.6556	0.0001	0:18:38
3	5.5771	-0.0001	7.6557	0.0002	0:21:53
4	5.5771	-0.0001	7.6557	0.0002	0:24:53
5	5.5777	0.0004	7.6549	-0.0006	0:28:12
6	5.5775	0.0003	7.6551	-0.0004	0:31:10
7	5.5776	0.0003	7.6550	-0.0005	0:34:12
8	5.5779	0.0006	7.6546	-0.0009	0:37:15
9	5.5774	0.0001	7.6553	-0.0002	0:40:17
10	5.5775	0.0002	7.6552	-0.0003	0:43:44
11	5.5773	0.0001	7.6554	-0.0001	0:47:16
12	5.5770	-0.0002	7.6558	0.0003	0:50:45
13	5.5776	0.0003	7.6550	-0.0005	0:54:19
14	5.5776	0.0004	7.6549	-0.0006	0:57:54
15	5.5770	-0.0002	7.6558	0.0003	1:01:05
16	5.5776	0.0004	7.6550	-0.0005	1:04:38
17	5.5766	-0.0007	7.6564	0.0009	1:07:49
18	5.5765	-0.0007	7.6565	0.0010	1:10:55
19	5.5766	-0.0006	7.6563	0.0008	1:14:30
20	5.5762	-0.0010	7.6569	0.0014	1:18:02

Average Volume: 5.5772 cm3
Average Density: 7.6555 g/cm3

Standard Deviation: 0.0004 cm3
Standard Deviation: 0.0006 g/cm3

Figure 4-2. Analysis Report

The following report represents an analysis that was performed on an untreated (not dried) sample with run precision enabled. The run precision feature eliminates data from all but five runs that are within a specified tolerance. An asterisk next to a run indicates that the data from the run were eliminated from the calculations.

Page 1

AccuPyc 1330 VX.XX
Serial Number: XXXXX
Density and Volume Report

Sample ID: 11-6-89--14-45
Sample Weight: 5.8293 g
Temperature: 24.5 C
Number of Purges: 20
Cell Volume: 12.3837 cm3

Started: 01/09/92 12:00:00
Completed: 01/09/92 13:24:45

Equilibration Rate: 0.0050 psig/min
Expansion Volume: 7.9985 cm3

Run#	Volume cm3	Deviation cm3	Density g/cm3	Deviation g/cm3	Elapsed Time (h:m:s)
1*	1.4081	0.0000	4.1397	-0.0000	0:15:43
2*	1.4072	-0.0010	4.1425	0.0028	0:18:38
3*	1.4072	-0.0009	4.1425	0.0028	0:21:53
4*	1.4072	-0.0010	4.1426	0.0029	0:24:53
5*	1.4111	0.0030	4.1310	-0.0087	0:28:12
6*	1.4083	0.0001	4.1393	-0.0004	0:31:10
7*	1.4074	-0.0007	4.1418	0.0021	0:34:12
8*	1.4076	-0.0005	4.1412	0.0015	0:37:15
9*	1.4059	-0.0011	4.1431	0.0033	0:40:17
10*	1.4059	-0.0023	4.1464	0.0067	0:43:44
11*	1.4070	-0.0011	4.1429	0.0032	0:47:16
12*	1.4076	-0.0006	4.1414	0.0017	0:50:45
13*	1.4074	-0.0007	4.1419	0.0022	0:54:19
14*	1.4107	0.0025	4.1323	-0.0075	0:57:54
15*	1.4068	-0.0013	4.1436	0.0039	1:01:05
16*	1.4062	-0.0020	4.1455	0.0058	1:04:38
17*	1.4082	0.0000	4.1396	-0.0001	1:07:49
18	1.4083	0.0002	4.1393	-0.0005	1:10:55
19	1.4084	0.0003	4.1389	-0.0009	1:14:30
20	1.4085	0.0004	4.1387	-0.0011	1:18:02
21	1.4073	-0.0008	4.1421	0.0024	1:21:35
22	1.4081	-0.0000	4.1397	0.0000	1:24:45

Average Volume: 1.4081 cm3
Average Density: 4.1397 g/cm3

Standard Deviation: 0.0005 cm3
Standard Deviation: 0.0014 g/cm3

Figure 4-4. Analysis with Run Precision Report

Transmitting Reports

The AccuPyc RS-232 interface transmits report data to a computer using the standard ASCII file format. Once captured with an asynchronous serial communications program such as COTERM, which is available from Micromeritics (part no. 003-20632-00), the report data can be used in popular spreadsheet and data manipulation programs.

Press [ ] [3] to transmit report data over the serial line. If you press [ ] [3] during an automatic operation, a partial report is transmitted.

You may cancel report data sent to the serial line by pressing [ ] [CLEAR]. You may cancel partial reports that were requested during an automatic operation when the following message is displayed:

```
Sending line (line no.)
[Escape] to cancel
```

Reports may be transmitted in one of two formats: *single column* or *spreadsheet*. Refer to Appendix D, **Format of Transmitted Data** for a description of the formats.

Calibrating the Pycnometer

You should check or calibrate the pycnometer anytime you restart it. For a quick check, run an analysis with an empty cup to see how close the average volume is to 0. It should be ±0.05% of full-scale. If it is not within ±0.05% of full-scale, calibrate according to the instructions given below. When you calibrate the pycnometer, **cell volume** and **expansion volume** in the set-up parameters are updated automatically.

When calibrating the pycnometer, you should set up your calibration parameters so that 10 purges and 10 runs are performed. See the section on *Analysis or Calibration Parameters* later in this chapter.

1. Place an empty cup in the cell chamber.
2. Replace the cell chamber cap.
3. Press [ ] [•] to begin the calibration procedure.

Display	Entry
Volume of cal std: 1.0000 cm^3	Enter the volume of the calibration standard and press ENTER. *Range: 0.1 to 999.0000 cm^3*
[Enter] to start [Escape] to cancel	Press ENTER to begin the calibration or [ ] CLEAR to cancel the calibration. Status messages are displayed during the first phase of calibration. When the first phase ends, the pycnometer beeps three times.
Insert cal std [Enter] to start	Place the calibration standard* in the cup in the cell chamber.

In most cases, use one ball for the 100-cm^3 pycnometer; two balls for the 10-cm^3 pycnometer. For best results, sample volume should be approximately equal to the volume of the calibration standard. Therefore, if you are calibrating the 10-cm^3 pycnometer for use with smaller sample volumes, you may wish to use only one ball.

Replace the cell chamber cap; then press ENTER. When you press ENTER, calibration continues and operational status messages are continually displayed.

Zeroing the Pressure Transducer

The transducer offset may be reset to zero in order to perform diagnostics or to operate the pycnometer manually.

The transducer is automatically set to zero for each run in an analysis or calibration.

Press [ ] [0] to zero the pressure transducer.

Display	Entry
[Enter] to start **[Escape] to cancel**	Press [ENTER] to zero the transducer or [ ] [CLEAR] to cancel the operation. The transducer is zeroed and the pycnometer returns to display mode.

Manually Controlling the Valves

This function enables you to perform a manual analysis by controlling the valves.

Press [ ] [1] to enter manual mode.

Display	Entry
Manual **p = (pressure)**	When **Manual** is shown in the display, you can manually open and close the fill, expansion, and vent valves by pressing the appropriate keys. 7 - Opens and closes the fill valve 8 - Opens and closes the expansion valve 9 - Opens and closes the vent valve Observe the indicators above the [FILL], [EXPAND], and [VENT] keys. When a valve is open, the indicator is on. When a valve is closed, the indicator is off. Press [SAVE] when you wish to exit manual mode and return to display mode.

Prolonged opening of all three valves at one time may cause excessive loss of helium.

Editing the Setup Parameters

The set-up function enables you to enter parameters to be used for analysis, calibration, reporting, and data transmission. There are five sets of parameters:

- Analysis or calibration parameters
- Report options
- Calibration volumes
- Data transmission
- Unit types and Operating Language

The pycnometer is shipped with default values entered for analysis parameters; it is not necessary to edit these values in order to perform an analysis. However, you may modify an analysis to meet your own particular needs by changing the parameters, which include number of purges and runs (0 to 99), fill pressures (0 to 19.850 psig), and equilibration rate (0.0001 to 9.0000 psig/min).

Default values are also entered for calibration parameters. These may be edited in the same manner as analysis parameters. The only difference is that for calibration the maximum number of purges and runs that will be performed by the system is ten.

Report options specify the report mode (density or volume for analysis) and the report destination.

Calibration volumes specify the sample volume and the expansion volume to be used for calibration.

Data transmission specifies transmission parameters such as baud rate, parity, etc.

Unit types specify either pounds-per-square-inch gauge (psig) or kilopascals gauge (kPag) to be used as the pressure measurement unit type. It also specifies the language to be used.

The parameters you enter are stored and used to control the pycnometer until new parameters are entered. Instructions follow for each set of parameters.

Analysis or Calibration Parameters

Press [ ] [2] to display or edit analysis or calibration parameters.

Display	Entry
Set-up type? **Analysis parameters**	Press [CHOICE] until **Analysis parameters** is displayed. Then press [ENTER].
Number of purges: **3**	Enter the number of purges to be performed and press [ENTER]. *Range:* *0 to 99* Purging cleans the cell and expansion chambers before an analysis begins. The greater the number of purges, the cleaner the sample will be when analyzed.
Purge fill pressure: **19.500 psig**	Enter the fill pressure and press [ENTER]. *Range:* *0 to 19.850 psig* *0 to 136.86 kPag* Generally, the greater the fill pressure, the easier it is to measure the volume precisely. However, a lower pressure may be desirable for some samples.
Number of runs: **3**	Enter the number of runs to be performed and press [ENTER]. *Range:* *1 to 99*
Run fill pressure: **19.500 psig**	Enter the fill pressure and press [ENTER]. *Range:* *0 to 19.850 psig* *0 to 136.86 kPag*

Display	Entry
Equilibration rate: **0.005 psig/min**	Enter the equilibration rate and press ENTER . *Range:* *0.0001 to 9.0000 psig/min* *0.0007 to 62.05 kPag/min* A high rate will produce faster results, but results may not be as precise as desired. The lowest rates may cause errors when some materials (such as materials with appreciable vapor pressures, closed cell foams, or organics) are analyzed.
Use run precision? **No**	The run precision feature enables early termination of the analysis when certain criteria are met. When you select run precision, the analysis will be terminated after five consecutive runs are within the specified tolerance. If you select run precision, you should request a large number (50 to 99) of runs. If you select a small number of runs, the analysis will stop when the number you entered is reached even though the specified tolerance has not been met. Select yes or no, then press ENTER .

This prompt applies for analysis only.

Display	Entry
Percent full-scale: **0.05%**	This prompt is displayed only if the number of runs is greater than five. Enter the run precision volume tolerance which is expressed as a percent of nominal cell volume, then press ENTER. *Range:* *0.01% to 50%*

Nominal cell volume is the sample capacity.

Set-up type? **Analysis parameters**	Choose one of the following options: ■ Press SAVE to save the information you entered and return to display mode. ■ Enter another set-up type. ■ Press CLEAR to discard the information you entered and return to display mode.

Report Options

Press [ ] [2] to display or edit report options.

Display	Entry
Set-up type? **Report options**	Press [CHOICE] until **Report options** is displayed. Then press [ENTER].
Anls display mode? **Density**	Select the mode, either density or volume, in which the analysis report will be displayed, then press [ENTER]

Both density and volume will be shown on the printed report, but density will be correct only if sample weight was entered.

Request sample ID? **Yes**	A sample ID is a unique identifier of the sample. It can contain from 1 to 20 numbers or dashes. Using sample IDs can help you keep track of data from various analyses. For example, you can use the sample ID as a date and time stamp. Select either yes or no, then press [ENTER].
Transmission format? **Single column**	Select the format of data to be used when transmitting reports. Refer to Appendix D for a description of the formats. *Choices:* *Single column, Spreadsheet*
Report destination? **Display**	Select the destination of the report that is automatically generated after an analysis or calibration, then press [ENTER]. *Choices:* *display, printer, transmission line.*

Display	Entry

A display report is always calculated regardless of the destination.

Display	Entry
Set-up type? **Report options**	Choose one of the following options: ■ Press SAVE to save the information you entered and return to display mode. ■ Enter another set-up type. ■ Press CLEAR to discard the information you entered and return to display mode.

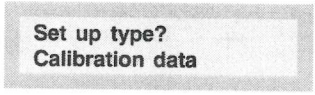
Calibration Data

Press [ ] [2] to display or edit calibration data.

Display	Entry
Set up type? **Calibration data**	Press [CHOICE] until **Calibration data** is displayed. Then press [ENTER].

When the pycnometer is calibrated, cell volume and expansion volume are updated automatically.

Cell volume: **(value from calibration)**	Enter the cell volume and press [ENTER]. *Range:* *0.01 to 999.0000 cm^3*
Expansion volume: **(value from calibration)**	Enter the expansion volume and press [ENTER]. *Range:* *0.5 to 999.0000 cm^3*
Temperature offset: **(pre-set value)**	The temperature offset is pre-set at the factory and is entered on the Calibration Sheet shipped with the instrument. It should not be changed unless the battery-backed RAM is reinitialized. If battery-backed RAM is reinitialized, enter the value from the calibration sheet and press [ENTER]. *Range:* *0 to 100.00°C*



Choose one of the following options:

- Press SAVE to save the information you entered andreturn to display mode.
- Enter another set-up type.
- Press [ ] CLEAR to discard the information you entered and return to display mode.

Data Transmission

Press [ ] [2] to display or edit data transmission parameters.

Display	Entry
Set-up type? **Data transmission**	Press [CHOICE] until **Data transmission** is displayed. Then press [ENTER].
Baud rate? **9600 baud**	Baud rate specifies the rate of data transmission. Select the baud rate, then press [ENTER]. *Choices:* *9600* *600* *110* *1200* *150* *2400* *300* *4800*
Number of data bits? **8**	Select either 8 or 7, then press [ENTER].
Number of stop bits? **1**	Select either 1 or 2, then press [ENTER].
Parity? **None**	Select the parity, then press [ENTER]. *Choices:* *none, even, odd.*
Xon/Xoff? **Disabled**	Select either disabled or enabled, then press [ENTER].
Set-up type? **Data transmission**	Choose one of the following options: ■ Press [SAVE] to save the information you entered and return to display mode. ■ Enter another set-up type. ■ Press [ ] [CLEAR] to discard the information you entered and return to display mode.

Unit Types, Operating Language, Date, and Time

Press [ ] [2] to display or edit unit types, operating language, date, or time.

Display	Entry
Set up type? **Unit types**	Press [CHOICE] until **Unit types** is displayed. Then press [ENTER].
Pressure Units? **psig**	Select either psig (pounds-per-square-inch gauge) or kPag (kilopascal gauge), then press [ENTER].
Language? **English**	Select the desired language, then press [ENTER]. *Choices:English, Deutsch, Español, Francais, or Italiano*

If there are error messages in the message queue (indicated by an asterisk next to the Reload prompt, delete the messages before changing languages. Refer to Error, Report, and System Status Messages in Chapter 4. Make sure you press SAVE after changing languages.

Date (DD/MM/YY) **(pre-set date)**	The date is included in all printed and transmitted reports. If the date shown is incorrect, you may enter the correct date as follows: day/month/year. Note that each entry must contain two digits. For example, to enter September 1, 1992, enter **01/09/92** and press [ENTER]. The [•] key is used to enter a slash (/).

Display	Entry
Time (HH:MM:SS) **(pre-set time)**	The time (in 24-hour format) at which an analysis was started and completed is shown on all printed and transmitted reports. If the time shown is incorrect, you may enter the correct time as follows: hours/minutes/seconds. Note that each entry must contain two digits. For example, to enter 1:05 P.M., enter **13:05:00** and press [ENTER]. The [•] key is used to enter a colon (:).
Set up type? **Unit types**	Choose one of the following options: ■ Press [SAVE] to save the information you entered and return to display mode. ■ Enter another set-up type. ■ Press [ ] [CLEAR] to discard the information you entered and return to display mode.

CHAPTER 5

TROUBLESHOOTING AND MAINTENANCE

- Troubleshooting
- Error and Status Messages
- Maintenance

TROUBLESHOOTING AND MAINTENANCE

This chapter describes common operational problems and their solutions, error and status messages, and maintenance procedures. If further assistance is needed after following the procedures in this chapter, contact a Micromeritics Service Representative.

Troubleshooting

Operating problems encountered with the pycnometer are usually easily corrected. Typical problems and the steps required to correct them are described in the following table.

Table 5-1. Common Operational Problems

What Happened	Why	What To Do
Nothing happens when power switch is turned on (ie: no lights on schematic display, nothing on LCD screen).	Power cord not fully inserted at one end or the other.	Insert power plug firmly into outlet socket; insert unit connector firmly into power connector opening.
	No power at outlet.	Plug in lamp or small appliance to test outlet. If there is no power, contact electrician.
	Plug prongs bent so that contact not made at outlet.	Wiggle power plug at outlet while watching display. If display comes on, have electrician adjust prongs or replace outlet or plug.
	Power cord damaged.	Have electrician check cord using test meter. Replace if defective.

What Happened	Why	What To Do
Nothing happens when power switch is turned on (ie: no lights on schematic display, nothing on LCD screen). (Continued)	Loose internal connection, broken wire, or failure of internal power supply.	Contact a Micromeritics Service Respresentative for repair or replacement information.
Unexpected characters on LCD screen (ie: squares, nothing but a cursor).	Software card not properly installed or defective.	Check to be sure that the card is properly installed. (NEVER insert or remove the card while the power is turned on.)
Specified pressure not reached or maintained.	Chamber cap not properly closed.	Close chamber cap by turning clockwise.
	Chamber cap contains dust or debris or the O-ring is not properly greased.	Using a lint-free tissue, clean the chamber cap and the rim of the cell chamber. Lightly grease the chamber cap O-ring (refer to **Greasing the Chamber Cap O-Ring** later in this chapter).
	O-ring in chamber cap not properly seated.	Check the chamber cap. Ensure that the O-ring is properly seated and that it contains no scratches or cuts.
	O-ring in chamber cap is cut or scratched.	Repace the O-ring in the chamber cap (refer to **Replacing the Chamber Cap O-Ring** later in this chapter).
	Gas leaks in the cell chamber or expansion chamber.	Check the pycnometer for leaks (refer to **Checking the Cell and Expansion Chambers for Leaks** later in this chapter).

What Happened	Why	What To Do
Specified pressure not reached or maintained. (continued)	Helium tank empty.	Check tank. Minimum recommended pressure is 200 psig.
	Cylinder Shut Off Valve closed.	Check that valve is open.
	Pressure regulator defect or pressure set too low.	Check regulator to be sure it is set properly. Call a Micromeritics Service Representative, if necessary.
	Zero offset (of pressure transducer) too low.	Check offset by opening chamber cap. If offset is negative, run new zero offset (**Zeroing the Pressure Transducer**, Chapter 4).
Helium drained from tank.	Leaks in the gas line connection.	Pressurize the system. Close, then open the Cylinder Shut-Off Valve. If the needle on the pressure gauge on the gas cylinder jumps abruptly, a leak in the gas line connections may be indicated. Check all gas line connections. (Refer to **Rear Panel Connections** in Chapter 2 for connection instructions.)
	Pycnometer was left in manual mode with all the valves open or the fill valve open and the chamber cap off.	Close all valves, then attach a new tank of helium.

What Happened	Why	What To Do
Unit will not equilibrate, or reproducibilty results falter.	Unit was left in manual mode with all the valves open or the fill valve open and the chamber cap off.	Close all valves, then attach a new tank of helium.
	Sample outgassing.	Prior to analysis, remove moisture and contaminants from sample. (Refer to **Preparing and Loading a Sample**, Chapter 3.)
	Pycnometer leaks.	Check cap O-ring for defects. Regrease or replace the O-ring, if necessary.
		While in Manual Mode, with the cap installed, open all valves. With helium flowing through the pycnometer, actuate the expansion valve repeatedly for approximately 1 minute. This will remove lint, which could cause a leak, from the valve seat. Repeat the process for the vent valve with the inlet and expansion valves open.

Error and Status Messages

The following error messages may appear in the display area of the pycnometer. Some of the messages contain the statement: "NN/ZZ runs completed." When this appears, NN will be replaced with the number of runs completed when the error occurred and ZZ will be replaced with the number of runs requested.

The messages are organized into two categories: Queued Messages and Messages Displayed During an Automatic Operation.

Queued Messages

Messages placed in a queue are displayed in chronological order when you press CHOICE . When the queue contains error messages, an asterisk is shown next to the **Reload** prompt.

ANLSERR: Eq failure
NN/ZZ runs completed

Cause: The sample being analyzed failed to equilibrate in 1000 seconds.

Action: Check the system carefully for leaks as described in **Checking the Cell and Expansion Chambers for Leaks** later in this chapter. Make sure the sample is properly prepared before performing an analysis.

Cause: Sample (foams, organics, etc.) absorbs helium slowly.

Action: Set the equilibration rate to progressively higher values until reasonable equilibration times (15 sec to 120 sec) are achieved.

ANLSERR: Fill failure
NN/ZZ runs completed

Cause: There was insufficient pressure to allow filling within five minutes during an analysis.

Action: Increase the regulator pressure or, if there is insufficient helium in the tank, obtain a new tank of helium.

ANLSERR: Overrange
NN/ZZ runs completed

Cause: A pressure overrange occurred during an analysis because the regulator pressure is set too high.

Action: Adjust the pressure so that it is equal to the higher of the two fill pressures specified in the analysis parameters plus 2.0 psig.

Cause: A pressure overrange occurred during an analysis because an error occurred in the pressure measurement electronics.

Action: Call a Micromeritics Service Representative.

ANLSERR: Underrange
NN/ZZ runs completed

Cause: A pressure underrange occurred during an analysis because an error occurred in the pressure measurement electronics.

Action: Call a Micromeritics Service Representative.

CAL1ERR: Eq failure
NN/ZZ runs completed

Cause: The empty cell chamber failed to equilibrate in 1000 seconds during the first pass of calibration.

Action: Check the system for leaks as described under **Checking the Cell and Expansion Chambers for Leaks** later in this chapter.

CAL1ERR: Fill failure
NN/ZZ runs completed

Cause: There was insufficient pressure to allow filling within five minutes during the first pass of calibration.

Action: Open the tank valves if shut, increase the regulator pressure or, if there is insufficient helium in the tank, obtain a new tank of helium. (Minimum recommended tank pressure is 200 psig.)

CAL1ERR: Overrange NN/ZZ runs completed

Cause: A pressure overrange occurred during the first pass of calibration because the regulator pressure is set too high.

Action: Adjust the pressure so that it is equal to the higher of the two fill pressures specified in the analysis parameters plus 2.0 psig.

Cause: A pressure overrange occurred during the first pass of calibration because an error occurred in the pressure measurement electronics.

Action: Call a Micromeritics Service Representative.

CAL1ERR: Underrange NN/ZZ runs completed

Cause: A pressure underrange occurred during the first pass of calibration because an error occurred in the pressure measurement electronics.

Action: Call a Micromeritics Service Representative.

CAL2ERR: Eq failure NN/ZZ runs completed

Cause: The calibration standard failed to equilibrate in 1000 seconds during the second pass of calibration.

Action: Check the system for leaks as described under **Checking the Cell and Expansion Chambers for Leaks** later in this chapter.

CAL2ERR: Fill failure NN/ZZ runs completed

Cause: There was insufficient pressure to allow filling within five minutes during the second pass of calibration.

Action: Open the tank valves if shut, increase the regulator pressure or, if there is insufficient helium in the tank, obtain a new tank of helium. (Minimum recommended tank pressure is 200 psig.)

CAL2ERR: Overrange
NN/ZZ runs completed

Cause: A pressure overrange occurred during the second pass of calibration because the regulator pressure is set too high.

Action: Adjust the pressure so that it is equal to the higher of the two fill pressures specified in the analysis parameters plus 2.0 psig.

Cause: A pressure overrange occurred during the second pass of calibration because an error occurred in the pressure measurement electronics.

Action: Call a Micromeritics Service Representative.

CAL2ERR: Underrange
NN/ZZ runs completed

Cause: A pressure underrange occurred during the second pass of calibration because an error occurred in the pressure measurement electronics.

Action: Call a Micromeritics Service Representative.

DATA_ERR:
No data to compute

Cause: An automatic operation was canceled before all necessary data could be collected.

Action: Restart the automatic operation.

Cause: All the runs have been excluded in review mode.

Action: Return to review mode and include some runs.

DTA_WRN: Cal std 10% of full-scale

Cause: The system tried to generate a calibration report, but the calibration standard used was less than 10% of the nominal cell chamber volume. Resulting data may be inaccurate.

Action: Use a calibration standard of sufficient size (calibration standards are available from Micromeritics). The calibration standard should occupy at least 10% of the nominal cell chamber volume and the more nearly filled the cell is, the better the calibration.

DTA_WRN: Volume 10% of full-scale

Cause: The amount of sample placed in the sample cup was so small that the percentage of precision may be inferior.

Action: Rerun the sample using enough material to occupy the maximum amount of the nominal cell chamber volume.

HW_ERR: A/D I/O failure

Cause: An error in the pressure measurement electronics has occurred.

Action: Call a Micromeritics Service Representative.

HW_ERR; BB RAM has been initialized

Cause: Battery-backed RAM has failed.

Action: Call a Micromeritics Service Representative.

HW_ERR: Valve I/O failure

Cause: An error in the valve control electronics has occurred.

Action: Call a Micromeritics Service Representative.

MAN_ERR: Pressure overrange

Cause: The fill valve was left open until the maximum system pressure was exceeded.

Action: Close the fill valve and open the vent and expansion valves. Allow the pressure to stabilize.

PRT_ERR: Timeout failed to respond

Cause: The printer took longer than 30 seconds to acknowledge receipt of data from the pycnometer.

Action: Check to make sure the printer is properly connected to the pycnometer, is turned on, and is on line.

SYS_ERR: Power Fail NN/ZZ Runs Completed

Cause: A power failure occurred and when power resumed, the automatic operation was canceled.

Action: Restart the automatic operation if desired.

TRN_ERR: Timeout failed to respond

Cause: The receiving device took longer than 10 seconds to acknowledge receipt of data from the pycnometer.

Action: Make sure the receiving device is properly connected to the pycnometer RS-232 port and is turned on. Verify that the serial I/O parameters controlling the receiving device correspond with the data transmission parameters in the set up mode.

USR_ERR: Cal std 10% of full-scale

Cause: You tried to enter a calibration standard volume that is less than 10% of the nominal full-scale volume.

Action: Enter a volume that represents at least 10% of the nominal cell chamber volume.

USR_ERR: No data to review

Cause: You tried to review data for an automatic operation when there were no data to review.

Action: Abandon request.

USR_ERR: Number of runs must be = 5

Cause: You tried to enable run precision without increasing the number of runs to at least five.

Action: Increase the number of runs to five or abandon request.

USR_ERR: Out of range

Cause: You tried to enter a value that is out of the valid range.

Action: Enter a value in the specified range (refer to Chapter 4).

USR_ERR: Pressure overrange

Cause: A pressure overrange occurred but was left uncorrected.

Action: Return to manual mode and vent the system.

ZEROERR: Eq failure

Cause: The system failed to equilibrate within 1000 seconds.

Action: Check the pycnometer for leaks. Make sure the pycnometer has been placed in a draft-free environment.

ZEROERR: Overrange

Cause: A pressure overrange occurred while zeroing.

Action: Check for a fill valve leak. If there is none, contact a Micromeritics Service Representative.

ZEROERR: Underrange

Cause: An error occurred in the pressure measurement electronics while zeroing, causing a pressure underrange to occur.

Action: Call a Micromeritics Service Representative.

Messages Displayed During an Automatic Operation

When an automatic operation is in progress, status messages are continually displayed.

Automatic operation has been canceled

Cause: The automatic operation has been canceled by the user.

Action: Wait for the termination process to complete or end the termination process by pressing [ ] [CLEAR] two more times.

Print in progress [ESCAPE] to cancel

Cause: Status message displayed when a report is being printed.

Action: None; this is a status message only.

Printer port not responding

Cause: The printer took longer than 30 seconds to acknowledge receipt of data from the pycnometer.

Action: Check to make sure the printer is properly connected to the pycnometer, is turned on, and is on line.

Sending line (line number) [ESCAPE] to cancel

Cause: Status message displayed when data are being transmitted.

Action: None; this is a status message only.

Transmission port not responding

Cause: The receiving device took longer than five seconds to acknowledge receipt of data from the pycnometer.

Action: Make sure the receiving device is properly connected to the pycnometer RS-232 port and is turned on. Verify that the serial I/O parameters controlling the receiving device correspond with the data transmission parameters in the set up mode.

Transmission port waiting for Xon

Cause: The receiving device stopped transmission by sending an Xoff, and hasn't resumed the transmission by sending an Xon.

Action: None; when the receiving device is ready for more data, it should send the pycnometer an Xon.

The following message is displayed on the printer or on the screen for transmitted data.

No collected data to report, or all runs excluded

Cause: This message is printed or transmitted if you requested a report but there is either no data available or data has been excluded via review mode.

Action: Initiate an automatic operation or return to review mode and include at least one run.

Maintenance

Greasing the Chamber Cap O-Ring

The cell chamber cap contains an O-ring that requires routine maintenance because it is so often exposed. The chamber cap O-ring should be greased at the beginning of each period of use.

Rubber gloves should be worn to prevent contamination of the grease with oil from your fingertips. Carefully apply a thin layer of grease; both too much and too little grease can cause a problem. Too much grease may alter cell volume. Too little grease results in an imperfect seal.

1. Remove the chamber cap by turning it counterclockwise then lifting off.
2. Wipe off the old grease using lint-free tissue.
3. Using your fingertips, apply a thin layer of Dow Corning high vacuum grease (or equivalent) to the greasing disk.
4. Insert the greasing disk into the chamber cap and turn slightly to apply a very light coating of grease to the O-ring.

Apply a VERY LIGHT coating of grease to the O-ring. There should be no visible ridges of grease; excess grease may alter analysis results.

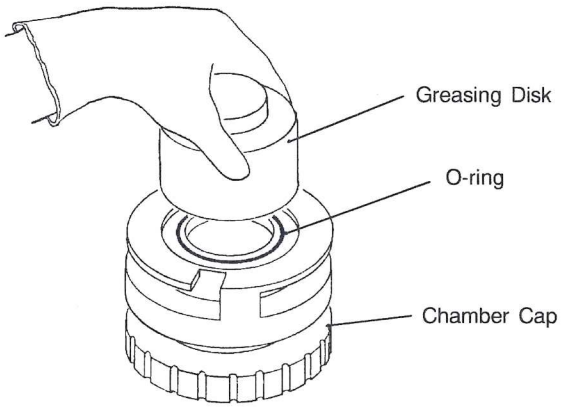


Figure 5-1. Greasing Chamber Cap O-Ring

5. Wipe the mating surface in the cell chamber with lint-free tissue.
6. Replace the chamber cap.

Replacing the Chamber Cap O-Ring

Fine fibers and particles between the O-ring and its sealing surfaces can cause leaks, as can scratches or cuts in the O-ring or in the metal surfaces. When it is necessary to replace the O-ring, follow these steps.

1. Remove the O-ring from its groove by pushing a sharp tool into the ring and prying it out.

Be careful not to scratch the metal surface of the chamber cap. Scratches could result in an imperfect seal.

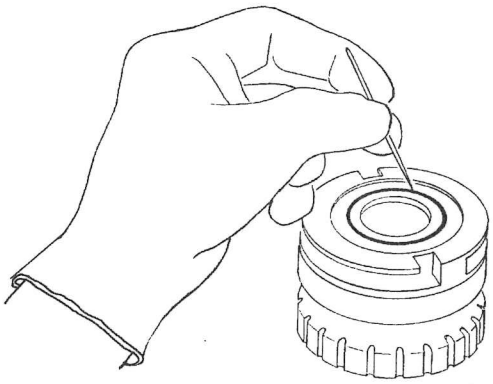


Figure 5-2. Removing the Chamber Cap O-Ring

2. Clean the groove in the chamber cap using a small brush or lint-free tissue and isopropyl alcohol.

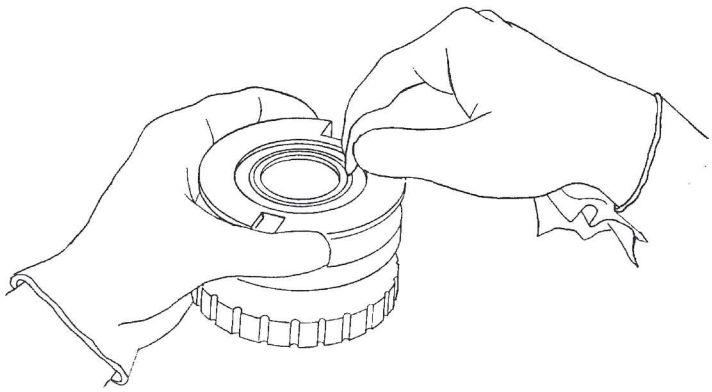


Figure 5-3. Cleaning the Chamber Cap

3. Allow the chamber cap to dry thoroughly.
4. Grease the O-ring using Dow Corning vacuum grease (or equivalent).
5. Replace the O-ring, being careful not to nick or scratch it.

Checking the Cell and Expansion Chambers for Leaks

To check the cell and expansion chambers for leaks perform the following procedure.

This procedure should be performed in a temperature-stable environment after the pycnometer has been warmed up for at least two hours. Before performing this procedure, check the chamber cap to ensure that it is not the source of leaks. It should be free from particles, the O-ring should be properly seated, and it should not contain excessive grease.

1. Allow the pycnometer to equilibrate thermally in a room having a stable temperature.
2. Press [ ] [1] to enter manual mode.

3. If the system has been open, manually purge the system before proceeding as follows:

   a. Press 8 to open the expansion valve and 9 to close the vent valve. (The indicator above the key is turned on when the valve is open.)

   b. Press 7 to open the fill valve and fill the sample chamber to the desired pressure.

   c. Press 7 to close the fill valve and 9 to open the vent valve.

   d. Repeat this procedure two or three times.

   e. Press 8 to close the expansion valve.

4. Press 7 to open the fill valve.

5. Fill the sample chamber to 19.5 psig.

6. Press 7 to close the fill valve.

7. Observe the pressure display. After an equilibration period (about 20 to 30 seconds), the pressure should not vary more than 0.005 psig/min.

   - If the pressure does not vary more than 0.005 psig/min, proceed to step 8.

   - If the pressure varies more than 0.005 psig/min., temperature instability or a leak may be indicated. Vent the system, then repeat steps 4 through 7 several times to verify that a leak is indicated. If a leak is indicated, call a Micromeritics Service Representative.

8. Press 8 to open the expansion valve and 7 to open the fill valve.

9. Fill the chambers to 19.5 psig.

10. Press 7 to close the fill valve.

11. Observe the pressure display. After an equilibration period (about 20 to 30 seconds), the pressure should not vary more than 0.005 psig/min.

    If the pressure varies more than 0.005 psig/min, temperature instability or a leak may be indicated. Vent the system, then repeat steps 8 through 11 several times to verify that a leak is indicated. If a leak is indicated, call a Micromeritics Service Representative.

Replacing the Gas Inlet Filters

The gas inlet contains a filter that may become contaminated after extended periods of use. If you are getting very low or erratic values for volume and density, the filter may be contaminated. Replace it as follows.

1. Turn off the gas supply at the regulator mounted to the cylinder by turning the cylinder shut-off valve fully counterclockwise.

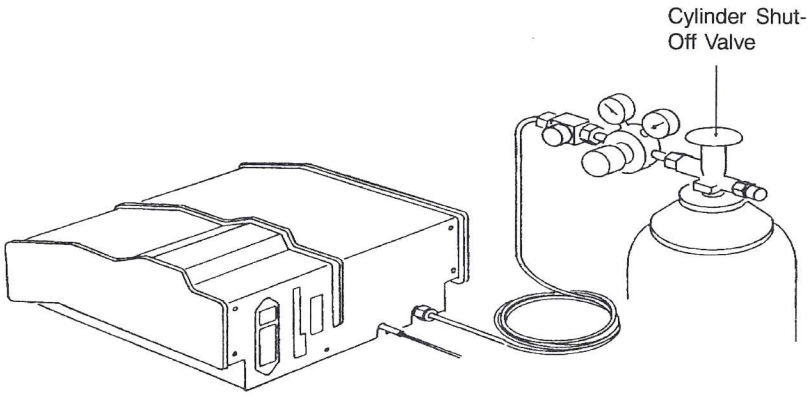


Figure 5-4. Turning Off Gas Supply

2. Press [ ] [1] on the pycnometer keypad to enter manual mode. Then press VENT to open the vent valve, EXPAND to open the expansion valve, and FILL to open the fill valve.

The power cord should be disconnected from the unit before disconnecting the gas supply. Failure to do so could result in electrical shock.

3. Observe the pressure reading. When the pressure drops to less than 1 psig, place the power switch on the back of the pycnometer in the off (0) position. Remove power cord.

4. Disconnect the gas supply line from the rear panel of the pycnometer by turning the nut on the INLET connect counterclockwise with a 7/16-in. open-end wrench.

5. Disconnect the gas vent line (if attached) by gently pulling the flexible tubing from the rigid tubing labeled VENT at the rear of the pycnometer.

6. Remove the printer cable and the serial cable (if used) from the rear panel of the pycnometer.

7. Remove the rear panel by removing the retaining screws that hold the panel in place.

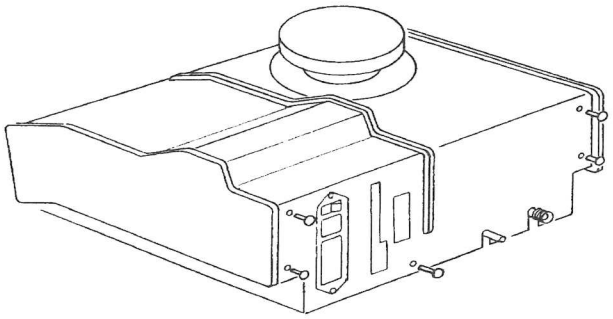


Figure 5-5. Removing Rear Panel

8. Lift the side panels at the rear of the pycnometer and rotate forward. The cover should lock in an upright position as shown below.

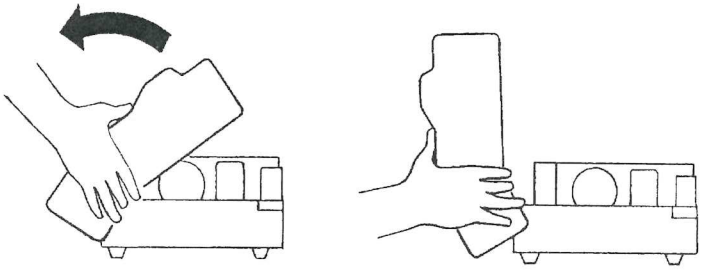


Figure 5-6. Rotating Top Panel

9. Remove the two screws that secure the gas inlet entrance assembly.

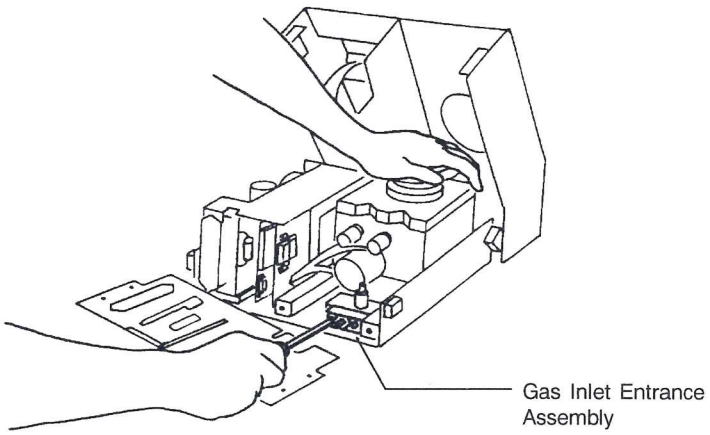


Figure 5-7. Gas Inlet Entrance Assembly

10. Remove the filter and O-ring.

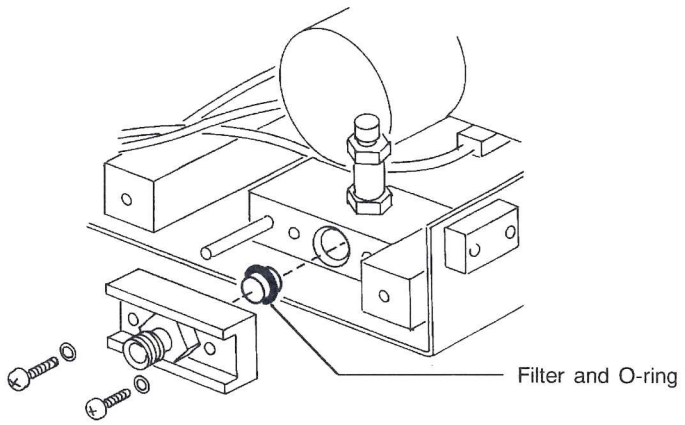


Figure 5-8. Removing Filter and O-ring

11. Wipe the interior of the gas entrance block with lint-free cloth to remove any grease that may still be present.

12. Very lightly grease the new O-ring with high vacuum grease.

Apply only a very light coating of grease. Too much grease can clog the filter or disrupt gas flow.

13. Insert the new filter and O-ring into the gas entrance block.
14. Replace the two screws that secure the gas inlet entrance assembly.
15. Lower the side panels of the pycnometer.
16. Replace the rear panel of the pycnometer and secure with the screws removed in step 7.

Cleaning the Pycnometer

The exterior casing of the pycnometer may be cleaned using a clean cloth, dampened with isopropyl alcohol (IPA), a mild detergent solution, or a 3% hydrogen peroxide solution.

Do not immerse the pycnometer or the power cord in any liquids. Doing so could result in electrical shock to personnel or damage to the unit.

Do not allow liquid to penetrate the casing of the pycnometer. Doing so could result in damage to the unit.

Recovering from a Power Failure

The pycnometer has a battery back-up feature that saves entered data in case of a power failure. Set up parameters and any other data entered will still be present when power is restored. If an automatic operation was in progress when the power failure occurred, it will be canceled when the pycnometer re-starts. Any data collected during the automatic operation will still be present, but the operation should be started again in order to produce complete results.

CHAPTER 6

ORDERING INFORMATION

ORDERING INFORMATION

Part Number	Item and Description
133-00000-00	AccuPyc 1330, fully-automatic gas displacement pycnometer with 10-cm^3 sample capacity.
133-00002-00	AccuPyc 1330, fully-automatic gas displacement pycnometer with 100-cm^3 sample capacity.
133-34802-00	Sample Module, 10 cm^3
133-34803-00	Sample Module, 1 cm^3
133-34804-00	Sample Module, 100 cm^3
133-25805-00	Sample Cup, 10-cm^3 capacity
133-25845-00	Sample Cup, 100-cm^3 capacity
004-25575-00	Chamber Cap O-ring, 10 cm^3
004-25577-00	Chamber Cap O-ring, 100 cm^3
133-34900-00	Calibration Standard Kit, contains two 10-cm^3 calibration standards and one 10-cm^3 sample cup
133-34902-00	Calibration Standard Kit, contains one 100-cm^3 calibration standard and one 100-cm^3 sample cup
004-25633-00	Calibration Standard, 10 cm^3
004-25632-00	Calibration Standard, 100 cm^3
004-25549-00	Reducer, 1/8-in. tube x 1/4-in. tube
004-62230-58	Gas Regulator, 30 psig, CGA 580
230-02001-00	Gas Inlet Line Assembly
004-27042-00	Inlet Filter
004-25469-00	Inlet Transducer Filter O-ring
008-16045-00	High Vacuum Grease, Dow Corning
133-25825-00	Greasing Disk
003-20616-01	RS-232 Cable, F/F 10-ft shielded
133-42701-01	French Template
133-42701-02	German Template
133-42701-03	Spanish Template
133-42701-04	Italian Template

Part Number	Item and Description
133-42801-00	Operator's Manual
	Optional Equipment
003-33009-02	Control Module. Includes 80386SX CPU with 80387SX math coprocessor, 40 Mb hard disk, VGA color monitor, dot-matrix printer, and cables (100/120 V, 50/60 Hz)
003-33009-03	Control Module. Includes 80386SX CPU with 80387SX math coprocessor, 40 Mb hard disk, VGA color monitor, dot-matrix printer, and cables (200/240 V, 50/60 Hz)
133-33602-00	COTERM/220 Software Kit, includes RS-232 cable and diskette.
Call for current part number.	Printer

APPENDIX A

CHANGING THE ANALYSIS MODULE

CHANGING THE ANALYSIS MODULE

Analysis modules, which house the cell chamber, are available in various sizes, enabling you to analyze different size samples with precision. To change the analysis module:

1. Turn off the gas supply at the regulator mounted to the gas cylinder by turning the Cylinder Shut-Off Valve fully clockwise.

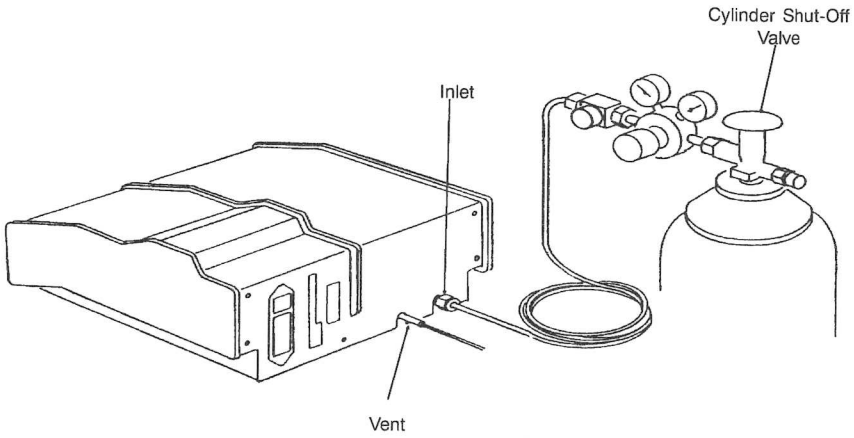


Figure A-1. Turning Off Gas Supply

2. Press [ ] [1] on the pycnometer keypad to enter manual mode. Then press VENT to open the vent valve, EXPAND to open the expansion valve, and FILL to open the fill valve.

The power cord should be disconnected from the unit before disconnecting the gas supply. Failure to do so could result in electrical shock.

3. Observe the pressure reading. When the pressure drops to less than 1 psig, place the power switch on the back of the pycnometer in the off (O) position. Remove the power cord.

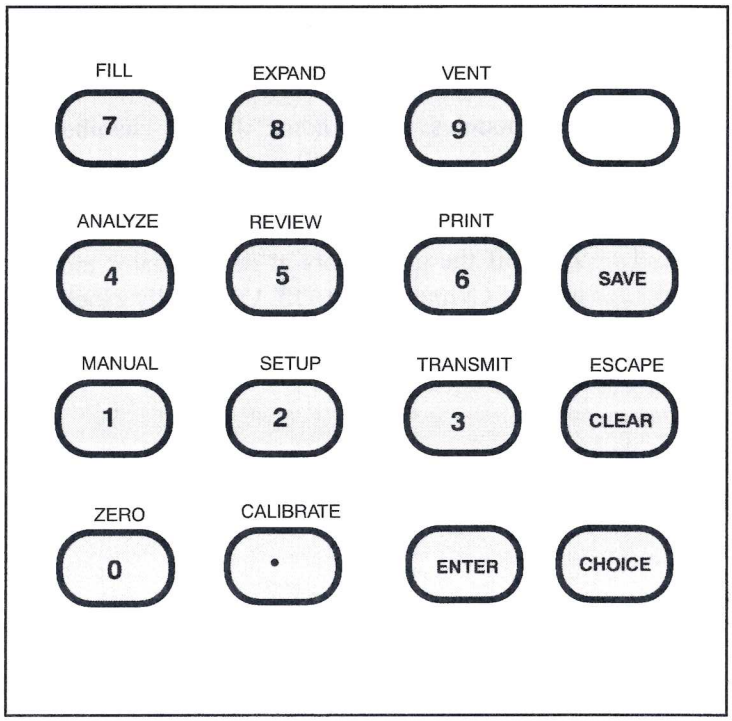


Figure A-2. Pycnometer Keypad

4. Disconnect the gas supply line from the rear panel of the pycnometer by turning the nut on the INLET connector counterclockwise with a 7/16 in. open-end wrench (refer to Figure A-1).

5. Disconnect the gas vent line (if attached) by gently pulling the flexible tubing from the rigid tubing labeled VENT at the rear of the pycnometer.

6. Remove the printer cable and the serial cable (if used) from the rear panel of the pycnometer.

7. Remove the cap from the cell chamber.

8. Remove the rear panel by removing the retaining screws that hold the panel in place.

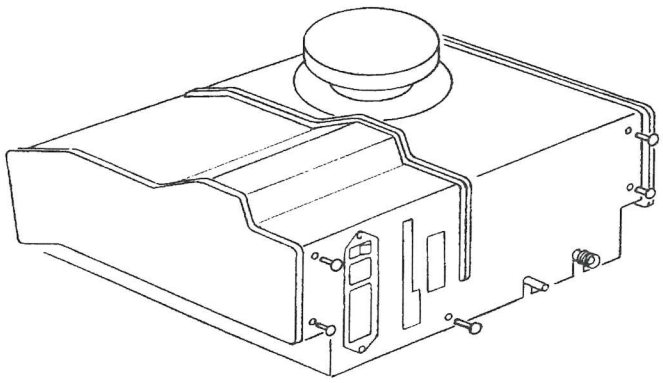


Figure A-3. Removing Rear Panel

9. Lift the side panels at the rear of the pycnometer and rotate forward as shown in Figure A-4A. After about 100 degrees rotation, the assembly should lock in an upright position as shown in Figure A-4B.

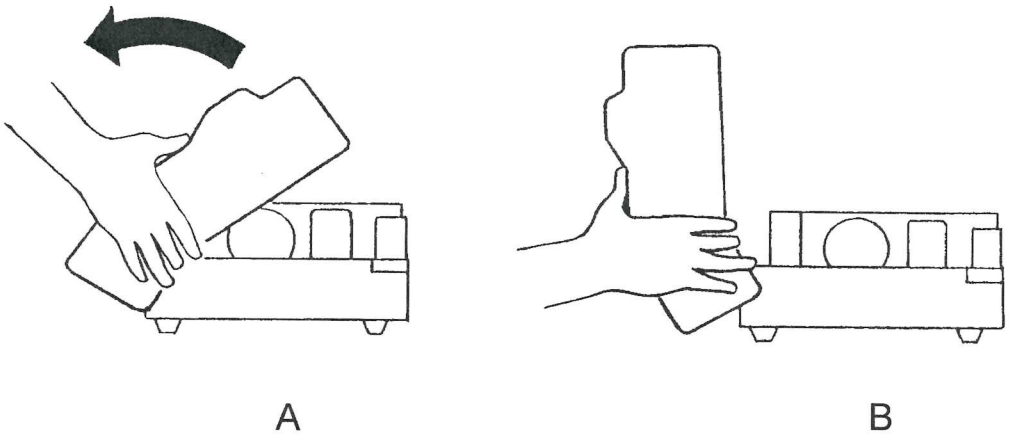


Figure A-4. Rotating Top Panel

10. Remove the screws securing the module mounting rails to the baseplate.

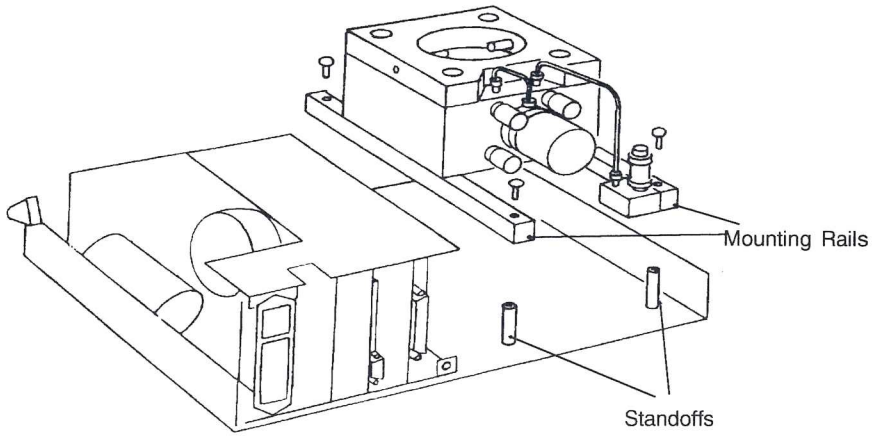


Figure A-5. Module Mounting Rails

11. Grasp the module and pull it straight up. A convenient method of removing the module is to replace the cap completely and use the knob as a handle to pick up the module. Observe the standoffs mounted to the baseplate and the corresponding recesses in the rails.

12. Disconnect the cable assemblies going to the valves mounted to the block.

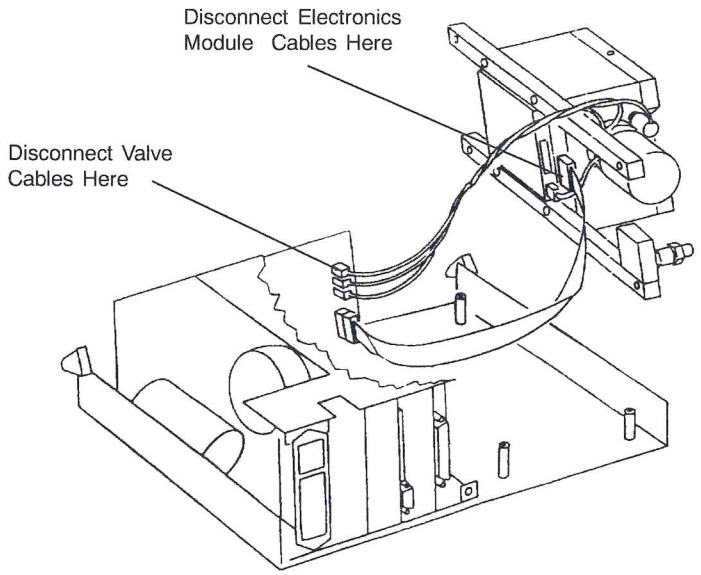


Figure A-6. Disconnecting Cable Assemblies

13. Remove the cable from the electronics module at the point it attaches to the printed circuit
board underneath the module.

14. Attach the cable removed in step 13 to the new module's printed circuit board.

15. Attach cables from the Vent, Expand, and Fill valves to J6, J9, and J11 as shown in Figure A-7.

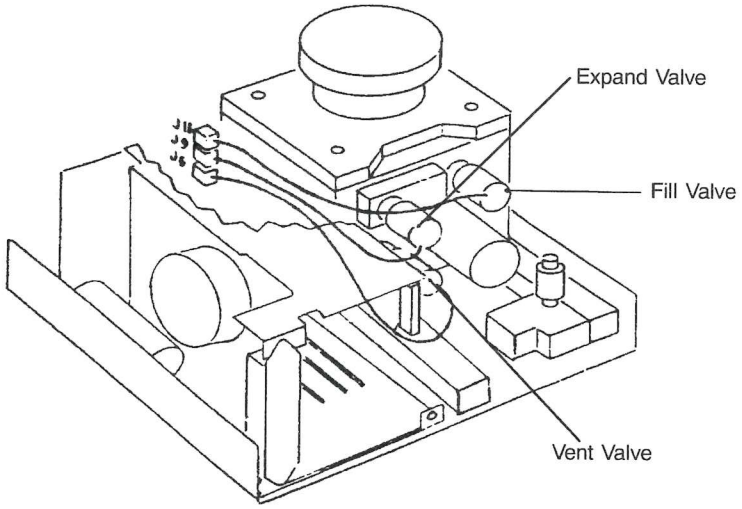


Figure A-7. Connecting Cable Assemblies

16. Place the new module into the pycnometer. Make sure the standoffs fit into the corresponding recesses in the rails. Note that the transducer should be on the rear face of the module.

17. Reassemble the pycnometer by reversing steps 1 through 10. Be sure to remove the cap before closing the cover and to reconnect all cables and the gas supply line. Check the gas connection for leaks.

18. Set the regulator pressure as described in **Setting Regulator Pressure** in Chapter 3.

19. Calibrate the pycnometer as described in **Calibrating the Pycnometer** in Chapter 4.

APPENDIX B

ANALYSIS THEORY

ANALYSIS THEORY

The AccuPyc 1330 Pycnometer is a gas displacement pycnometer, a type of instrument which measures the volume of solid objects of irregular or regular shape whether powdered or in one piece. A greatly simplified diagram of the instrument is shown in Figure B-1.

Assume that both V_{CELL} and V_{EXP} are at ambient pressure P_a, are at ambient temperature T_a, and that the valve is then closed. V_{CELL} is then charged to an elevated pressure P_1. The mass balance equation across the sample cell, V_{CELL}, is

$$P_1 \ (V_{CELL} - V_{SAMP}) = n_c R T_a \qquad (1)$$

where

n_c =the number of moles of gas in the sample cell,
R =the gas constant, and
T_a =the ambient temperature.

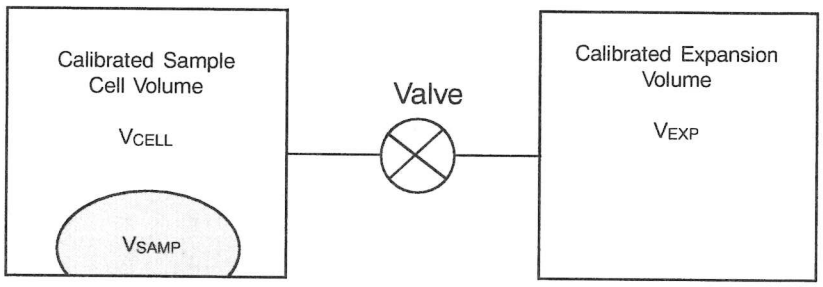


Figure B-1. Simplified Block Diagram

The mass equation for the expansion volume is

$$P_a \ V_{EXP} = n_E R T_a \qquad 2)$$

where

n_E = the number of moles of gas in the expansion volume.

When the valve is opened, the pressure will fall to an intermediate value, P_2, and the mass balance equation becomes

$$P_2 (V_{CELL} - V_{SAMP} + V_{EXP}) = n_c RT_a + n_E RT_a \quad (3)$$

Substituting from equations (1) and (2) into (3):

$$P_2(V_{CELL} - V_{SAMP} + V_{EXP}) = P_1 (V_{CELL} - V_{SAMP}) + P_a V_{EXP} \quad (4)$$

or

$$(P_2 - P_1) (V_{CELL} - V_{SAMP}) = (P_a - P_2) V_{EXP} \quad (5)$$

then

$$V_{CELL} - V_{SAMP} = \frac{P_a - P_2}{P_2 - P_1} V_{EXP} \quad (6)$$

Adding and subtracting P_a in the denominator and rearranging gives

$$-V_{SAMP} = -V_{CELL} + \frac{(P_a - P_2) V_{EXP}}{(P_2 - P_a) - (P_1 - P_a)} \quad (7)$$

Dividing by $(P_a - P_2)$ in both the numerator and denominator

$$V_{SAMP} = V_{CELL} - \frac{V_{EXP}}{-1 - \frac{P_1 - P_a}{P_a - P_2}} \quad (8)$$

or

$$V_{SAMP} = V_{CELL} - \frac{V_{EXP}}{\frac{P_1 - P_a}{P_2 - P_a} - 1} \quad (9)$$

Since P_1, P_2, and P_a are expressed in equations (1) through (9) as absolute pressures and equation (9) is arranged so that P_a is subtracted from both P_1 and P_2 before use, new P_{1g} and P_{2g} may be redefined as gauge pressures

$$P_{1g} = P_1 - P_a \tag{10}$$

$$P_{2g} = P_2 - P_a \tag{11}$$

and equation (9) rewritten as

$$V_{SAMP} = V_{CELL} - \frac{V_{EXP}}{\frac{P_{1g}}{P_{2g}} - 1} \tag{12}$$

Equation (12) then becomes the working equation for the pycnometer. Calibration procedures are provided to determine V_{CELL} and V_{EXP} and the pressures are measured by a gauge pressure transducer. Provisions are made for conveniently charging and discharging gases at controlled rates, for optimizing the relative sizes of the sample chambers and expansion volumes, and for cleansing the samples of vapors which would render equations (1), (2), and (3) inadequate to describe behavior.

APPENDIX C

CALIBRATION THEORY

CALIBRATION THEORY

Prior to running samples on the AccuPyc 1330 Pycnometer, the volume of the sample cell and the expansion volume must be known. The derivation that follows permits these internal volumes to be measured with respect to a removable, accurately known standard volume. A simplified diagram of the instrument is shown in Figure C-1.

Assume that, V_{CALIB} is removed, V_{CELL} is charged to an elevated gauge pressure P_1 and V_{EXP} is at zero gauge (ambient) pressure but sealed and that the valve is closed. Upon opening the valve, the condition established is

$$P_1 V_{CELL} = P_2 (V_{CELL} + V_{EXP}) \quad (1)$$

where P_2 is the resulting intermediate pressure. The use of gauge pressures is permissible because it is equivalent to having subtracted a constant from both sides of the equation.

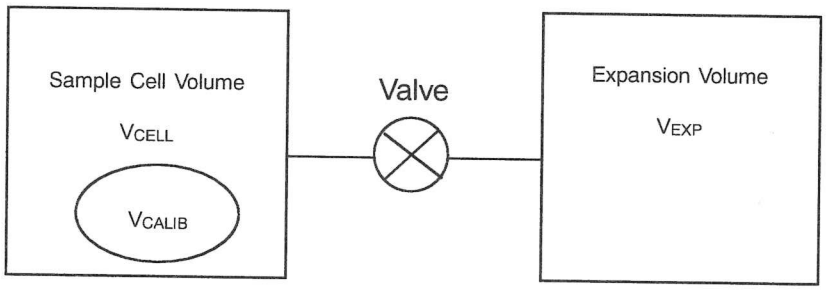


Figure C-1. Simplified Diagram for Calibration

Placement of V_{CALIB} into V_{CELL} and repetition of the charging and expansion yields

$$P_1 *(V_{CELL} - V_{CALIB}) = P_2 * (V_{CELL} - V_{CALIB} + V_{EXP}) \quad (2)$$

Where P_1* and P_2* are the before and after expansion pressures with V_{CALIB} in place.

V_{CALIB}, P_1, P_2, P_1*, and P_2* are assumed to be known or measurable. V_{CELL} and V_{EXP} are to be found. Solving equation (1) for V_{EXP} yields

$$V_{EXP} = V_{CELL} \frac{P_1 - P_2}{P_2} \quad . \tag{3}$$

Substitution of equation (3) into equation (2) yields

$$P_1 * (V_{CELL} - V_{CALIB}) = P_2 * (V_{CELL} - V_{CALIB}) + P_2 * [V_{CELL}] \frac{P_1 - P_2}{P_2} \quad . \tag{4}$$

Gathering terms and solving for V_{CELL} further yields

$$V_{CELL} = \frac{V_{CALIB} (P_1* - P_2*)}{\left[(P_1* - P_2*) - \frac{P_2*}{P_2} (P_1 - P_2) \right]} \quad . \tag{5}$$

Substitution of experimental and known values into equation (5) yields V_{CELL} which when used in equation (3) yields V_{EXP}, the desired result.

APPENDIX D

FORMAT OF TRANSMITTED DATA

FORMAT OF TRANSMITTED DATA

Calibration and analysis data can be transmitted in one of two user-selectable formats: single column or spreadsheet. The data are in ASCII-delimited format. Spreadsheet format is suitable for direct import into many popular spreadsheets (using COTERM or similar serial communication software). The following tables define the formats. Each table is followed by an example of the data defined in the table.

In all tables, units are as follows:

- Date DD/MM/YY
- Time HH:MM:SS
- Pressure psig
- Temperature °C
- Elapsed Time seconds

Table D-1. Calibration Report Format - Single Column

Record Number	Information Conveyed	Form
1	Version number	20 characters
2	Serial number	1 integer
3	Report type = calibration	11 characters
4	Start	
4a	Date	8 characters
4b	Time	8 characters
5	Stop	
5a	Date	8 characters
5b	Time	8 characters
6	Temperature	1 floating point
7	Calibration standard size	1 floating point
8	Number of purges	1 integer
9	Equilibration rate	1 floating point
10	Average cell volume	1 floating point
11	Cell volume standard deviation	1 floating point
12	Average expansion volume	1 floating point
13	Expansion volume standard deviation	1 floating point
14	Number of runs	1 integer
15	P1 (all P1 data)	1 floating point
16	P2 (all P2 data)	1 floating point
17	P1* (all P1* data)	1 floating point
18	P2* (all P2* data)	1 floating point

Record Number	Information Conveyed	Form
19	Included in average calculation (all included data) 0 = Excluded 1 = Included	1 integer

"AccuPyc 1330 VX.XX"
625
"Calibration"
"18/08/92","07:59:43"
"18/08/92","08:22:48"
25.799398
6.371870
3
0.005000
12.247808
0.000417
8.432275
0.000547
3
19.580629
19.564293
19.553852
11.296484
11.587052
11.580896
19.627428
19.618446
19.600245
8.060401
8.056624
8.049247
1
1
1

Figure D-1. Single Column Format

Table D-2. Calibration Report Format - Spreadsheet

Record Number	Information Conveyed	Form
1	Version number	20 characters
2	Serial number	1 integer
3	Report type = calibration	11 characters
4	Start (reported on one line as ASCII comma- delimited data)	
4a	Date	8 characters
4b	Time	8 characters
5	Stop (reported on one line as ASCII comma- delimited data)	
5a	Date	8 characters
5b	Time	8 characters
6	Temperature	1 floating point
7	Calibration standard size	1 floating point
8	Number of purges	1 integer
9	Equilibration rate	1 floating point
10	Average cell volume	1 floating point
11	Cell volume standard deviation	1 floating point
12	Average expansion volume	1 floating point
13	Expansion volume standard deviation	1 floating point
14	Number of runs	1 integer
15	Carriage return/line feed	
16	Carriage return/line feed	
17	Carriage return/line feed	
18	Run number and pressure (reported on one line as ASCII comma-delimited data)	
18a	Run number	1 integer
18b	P1	1 floating point
18c	P2	1 floating point
18d	P1*	1 floating point
18e	P2*	1 floating point
18f	Included in average calculation 0 = Excluded 1 = Included	1 integer
18g	Cell volume	1 floating point
18h	Cell volume deviation	1 floating point
18i	Expansion volume	1 floating point
18J	Expansion volume deviation	1 floating point

"AccuPyc 1330 VX.XX"
625
"Calibration"
"18/08/92","07:59:43"
"18/08/92","08:22:48"
25.799398
6.371870
3
0.005000
12.247808
0.000417
8.432273
0.000547
3

1,19.580629, 11.296484, 19.627428, 8.060401, 1, 12.248280, 0.000472, 8.432904, 0.000631
2, 19.564293, 11.587052, 19.618446, 8.056624,1, 12.247489, -0.000319, 8.431926, -0.000347
3, 19.553852, 11.580896, 19.600245, 8.049247, 1, 12.247654, -0.000154, 8.431990, -0.000283

Figure D-2. Spreadsheet Format for Calibration

Table D-3. Analysis Report Format - Single Column

Record Number	Information Conveyed	Form
1	Version number	20 characters
2	Serial number	1 integer
3	Report type = analysis	8 characters
4	Start	
4a	Date	8 characters
4b	Time	8 characters
5	Stop	
5a	Date	8 characters
5b	Time	8 characters
6	Temperature	1 floating point
7	Sample ID	20 characters
8	Sample Weight	1 floating point
9	Number of purges	1 integer
10	Equilibration rate	1 floating point
11	Cell volume	1 floating point
12	Expansion volume	1 floating point
13	Average volume	1 floating point
14	Volume standard deviation	1 floating point
15	Average density	1 floating point
16	Density standard deviation	1 floating point
17	Number of runs	1 integer
18	Run precision 0 = Disabled 1 = Enabled	1 integer
19	Percent full scale	1 floating point
20	P1 (all P1 data)	1 floating point
21	P2 (all P2 data)	1 floating point
22	Included in average calculation (all included data) 0 = Excluded 1 = Included	1 integer
23	Elapsed time(all elapsed time data)	1 unsigned integer

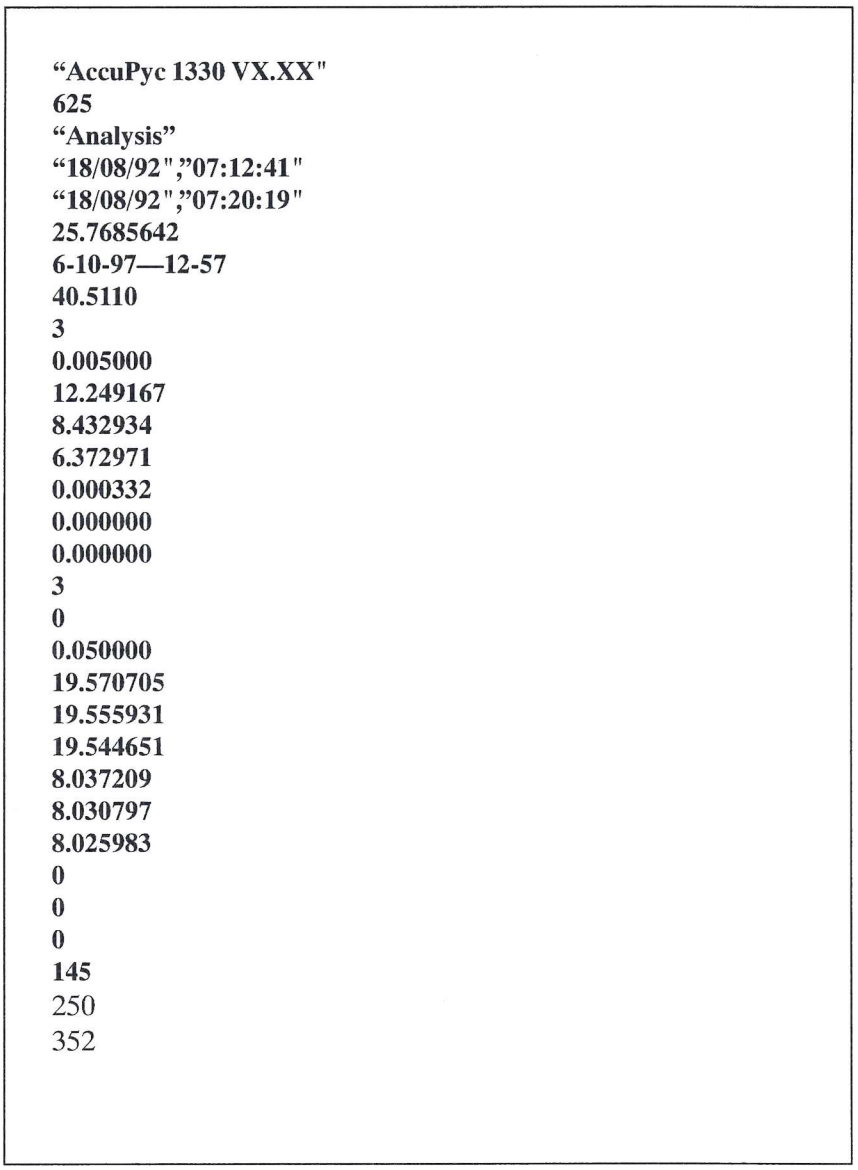


Figure D-3. Single Column Format for Analysis

Table D-4. Analysis Report Format -Spreadsheet

Record Number	Information Conveyed	Form
1	Version number	20 characters
2	Serial number	1 integer
3	Report type = analysis	8 characters
4	Start (reported on one line as ASCII comma- delimited data)	
4a	Date	8 characters
4b	Time	8 characters
5	Stop (reported on one line as ASCII comma- delimited data)	
5a	Date	8 characters
5b	Time	8 characters
6	Temperature	1 floating point
7	Sample ID	20 characters
8	Sample Weight	1 floating point
9	Number of purges	1 integer
10	Equilibration rate	1 floating point
11	Cell volume	1 floating point
12	Expansion volume	1 floating point
13	Average volume	1 floating point
14	Volume standard deviation	1 floating point
15	Average density	1 floating point
16	Density standard deviation	1 floating point
17	Number of runs	1 integer
18	Run precision 0 = Disabled 1 = Enabled	1 integer
19	Percent full scale	1 floating point
20	Carriage return/line feed	
21	Carriage return/line feed	
22	Carriage return/line feed	
23	Run number and pressure (reported on one line as ASCII comma-delimited data)	
23a	Run number	1 integer
23b	P1	1 floating point
23c	P2	1 floating point
23d	Included in average calculation 0 = Excluded 1 = Included	1 integer

Record Number	Information Conveyed	Form
23e	Elapsed time	1 unsigned integer
23f	Volume	1 floating point
23g	Volume deviation	1 floating point
23h	Density	1 floating point
23i	Density deviation	1 floating point

"AccuPyc 1330 VX.XX"
625
"Analysis"
"18/08/92","07:12:41"
"18/08/92","07:20:19"
25.685642
6-10-97—12-57
40.5110
3
0.000000
0.005000
12.249167
8.432934
6.372971
0.000332
0.000000
0.000000
3
0
0.050000

1, 19.570705, 8.037209, 0, 145, 6.372611, -0.000360, 0.000000, 0.000000
2, 19.555931, 8.030797, 0, 250, 6.373038, 0.000067, 0.000000, 0.000000
3, 19.544651, 8.025983, 0, 352, 6.373264, 0.000293, 0.000000, 0.000000

Figure D-4. Spreadsheet Format for Analysis

APPENDIX E

PRINTER CHARACTER SET

PRINTER CHARACTER SET

The following list contains the characters that the AccuPyc 1330 may send to the printer. Check your printer manual to ensure that your printer supports these characters.

Decimal Value	Character	Decimal Value	Character
33	!	59	;
34	"	60	<
35	#	61	=
36	$	62	>
37	%	63	?
38	&	64	@
39	'	65	A
40	(	66	B
41	)	67	C
42	*	68	D
43	+	69	E
44	,	70	F
45	-	71	G
46	.	72	H
47	/	73	I
48	0	74	J
49	1	75	K
50	2	76	L
51	3	77	M
52	4	78	N
53	5	79	O
54	6	80	P
55	7	81	Q
56	8	82	R
57	9	83	S
58	:	84	T

Decimal Value	Character	Decimal Value	Character
85	U	108	l
86	V	109	m
87	W	110	n
88	X	111	o
89	Y	112	p
90	Z	113	q
91	[	114	r
92	\	115	s
93	]	116	t
94	^	117	u
95	_	118	v
96	’	119	w
97	a	120	x
98	b	121	y
99	c	122	z
100	d	123	{
101	e	124	|
102	f	125	}
103	g	126	~
104	h	129	ü
105	i	132	ä
106	j	164	ñ
107	k	225	ß

INDEX

INDEX

G

I

K

L

M

N

O

P

R

S

T

U

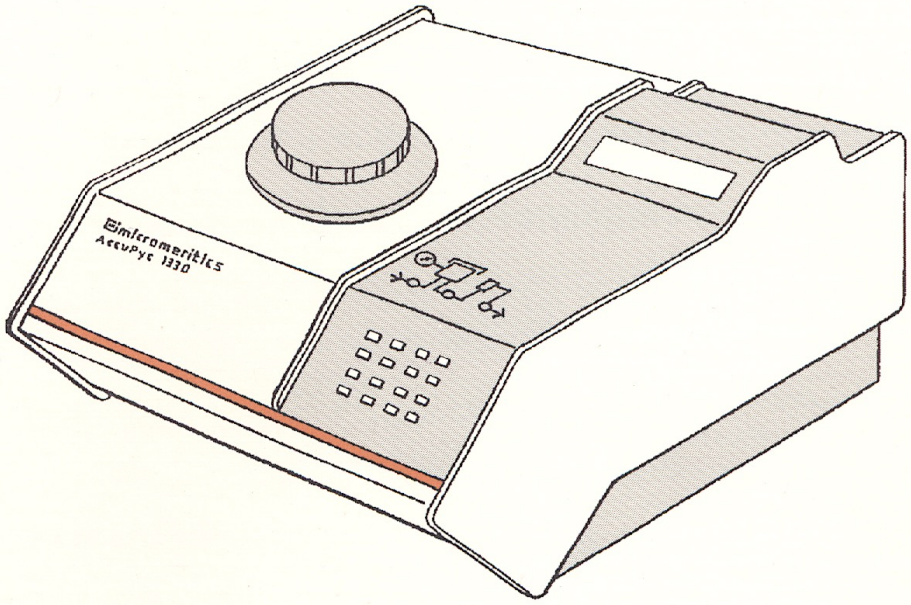
V

W

X

Z

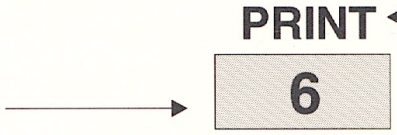
AccuPyc 1330
Quick Reference Guide



micromeritics®

USING THE KEYS

To select the primary function on the face of the key, press only that key. For example: press 6 to enter the number 6.



To select the alternate function printed above the key, first press the white key, then press the key. For example: press [ ] 6 to print a report.

STANDARD KEYS

Key	Used To
0 – 9	Enter the numbers 0 through 9.
.	Enter a decimal point or a dash for sample ID.
ENTER	Complete an entry or begin an action.
CHOICE	Display the next message when in display mode. Display the next multiple choice item when in a command mode.
CLEAR	Clear a message when in display mode. Clear an entry when in a command mode.
SAVE	Save the information you entered and return to display mode.
[ ]	Change the keypad mode. The keypad may be used in primary function mode or alternate function mode. When you press the white key to enter alternate function mode, a plus sign (+) appears in the upper right corner of the display and the commands written above the keys become available. To exit alternate function mode, press the white key again. The plus sign (+) will be removed from the display.

KEY COMBINATIONS

Function	Keys	Used To
ZERO	[ ] 0	Zero the pressure transducer.
CALIBRATE	[ ] .	Calibrate the pycnometer.
MANUAL	[ ] 1	Manually control the valves. After pressing the MANUAL key, you may use the FILL, EXPAND, and VENT keys to open and close the valves.
SET UP	[ ] 2	Display or edit analysis parameters, report options, calibration volumes, data transmission parameters, unit types, and operating language.
TRANSMIT	[ ] 3	Transmit analysis or calibration data over the serial line. If an automatic operation is in progress, transmit a partial report.
ESCAPE	[ ] CLEAR	Delete all data entered in the current mode and return to display mode. If an automatic operation is in progress, cancel it.
ANALYZE	[ ] 4	Perform an analysis.
REVIEW	[ ] 5	Review or edit completed analysis or calibration data.
PRINT	[ ] 6	Print analysis or calibration report. If an automatic operation is in progress, print a partial report.
FILL	7 (when in Manual mode)	Open and close the fill valve. The indicator above the FILL key is on when the valve is open and off when the valve is closed.
EXPAND	8 (when in Manual mode)	Open and close the expansion valve. The indicator above the EXPAND key is on when the valve is open and off when the valve is closed.
VENT	9 (when in Manual mode)	Open and close the vent valve. The indicator above the VENT key is on when the valve is open and off when the valve is closed.

PERFORMING AN ANALYSIS

1. Weigh the empty sample cup and record the weight.
2. Place sample in the cup and dry it thoroughly in an oven.
3. After preparing the sample, weigh the cup and sample and record the weight. Subtract the empty sample cup weight from the sample cup plus sample weight to determine the sample weight. Record the sample weight.
4. Remove the cell chamber cap.
5. Insert the sample cup into the cell chamber and replace the cell chamber cap.
6. Press [ ] [4] on the pycnometer keypad.
7. If **Sample ID** is displayed, enter the ID and press [ENTER]. If not, skip to step 8.
8. If **Sample Weight** is displayed, enter the weight and press [ENTER]. If not, skip to step 9.
9. Press [ENTER] to start the analysis.
10. As the analysis is performed, status messages are displayed. When the analysis is complete, the pycnometer beeps three times. Remove the sample from the cell chamber.
11. The **Reload** prompt is displayed. Press [CHOICE] to cycle through the error messages and report data.

CHANGING THE OPERATING LANGUAGE

1. If an automatic operation is in progress, wait until it is complete.
2. Press [ ] [CLEAR] to return to display mode if you are in another mode of operation.
3. Press [ ] [2].
4. Press [CHOICE] until **Unit type?** is displayed.
5. Press [ENTER] until **Language?** is displayed.
6. Press [CHOICE] until the desired language is displayed; then press [SAVE].

micromeritics®

AccuPyc™ 1330 Pycnometer

Operator's Manual

AccuPyc 1330 Helium Pycnometer

Uses gas displacement technique to determine absolute density

- Measures the absolute density of solids and slurries having volumes from 0.1 to 100 mL
- Completely automatic operation
- Capable of measuring open and closed cell foam materials with optional FoamPyc software
- Multivolume option allows analysis of a large variety of sample sizes
- Temperature control version allows analysis at user-selectable temperatures
- Glove box model allows analysis in controlled environments

Part No. 133-42808-01