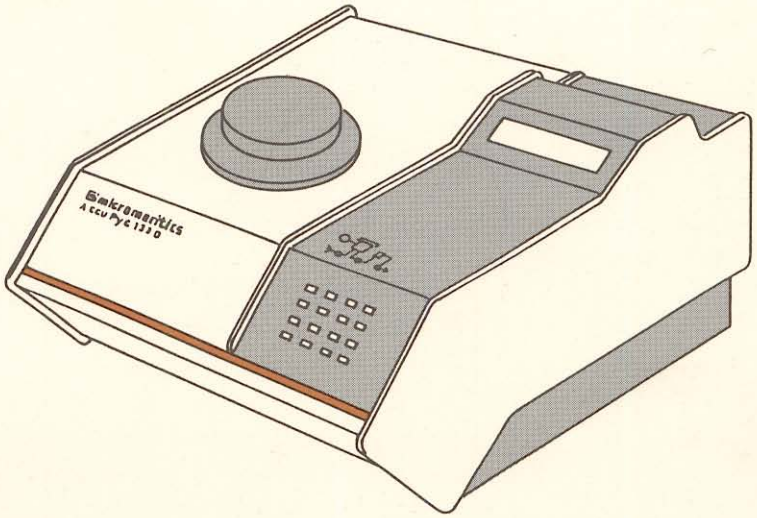
AccuPyc 1330



Quick Reference Guide

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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 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.

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].

ACCUPYC 1330

Kurzbedienungsanleitung

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Benutzung der Tastatur

Um die Standard-Belegung zu erhalten, drücken Sie die Taste mit dem entsprechenden Symbol-Aufdruck.
Zum Beispiel: Drücken Sie [6], um die Zahl „6" einzugeben.

Um die zweite Tasten-Belegung zu aktivieren, die über der jeweiligen Taste angegeben ist, drücken Sie die weiße Taste und anschließend die gewünschte Funktionstaste.
Zum Beispiel: Drücken Sie [ ] [6], um ein Meßprotokoll ausdrucken zu lassen.

Standard-Belegung

Taste	Wirkung
[0] – [9]	Eingabe der Ziffern 0 – 9.
[.]	Dezimal-Punkt oder Bindestrich bei der Eingabe der Probenbezeichnung.
[ENTER] (Eingabe)	Beenden der Eingabe oder Starten einer Messung.
[CHOICE] (Auswahl)	Auswahl der nächsten Meldung im Anzeige-Modus, Anzeigen der nächsten Auswahlmöglichkeit im Programmier-Modus.
[CLEAR] (Löschen)	Löschen einer Meldung im Anzeige-Modus oder einer Eingabe im Programmier-Modus.
[SAVE] (Sichern)	Sichern der eingegebenen Angaben und Rückkehr in den Anzeige-Modus.
[ ]	Wechsel der Tastatur-Belegung zwischen Standard- und Kombinationsbelegung. Die Tastatur kann in einem der beiden Modi betrieben werden. Nach dem Drücken der weißen Taste erscheint ein Plus-Zeichen („+") in der oberen rechten Ecke der LCD-Anzeige und die über den Tasten angebrachte Belegung wird wirksam. Durch erneutes Drücken der weißen Taste wird das Plus-Zeichen gelöscht und die Standard-belegung wird wirksam.

Tasten-Kombinationen

Funktion	Tasten	Wirkung
NULL.abgleich	[ ] [0]	Nullabgleich des Druckaufnehmers.
CAL.ibrieren	[ ] [.]	Starten des Kalibrationsprogramms.
MANUELL	[ ] [1]	Manuelle Bedienung der drei Ventile. Nach dem Drücken dieser Tastenkombination können die drei Tasten der ersten Reihe benutzt werden (s. unten).
SET UP (Voreinstellungen)	[ ] [2]	Anzeigen oder Ändern der voreingestellten Analysenbedingungen, der Protokoll-Optionen, der Kalibrations-Volumina, der verwendeten Druckeinheit oder der Bedienungs-Sprache.
SENDEN	[ ] [3]	Übertragen von Analysen- oder Kalibrations-Daten über die serielle Schnittstelle. Wenn eine automatische Messung läuft, wird ein Teilbericht gesendet, ohne die Messung zu unterbrechen.
ESCAPE (Verlassen)	[ ] [CLEAR]	Löschen aller eingegebenen Daten in der gerade gewählten Funktion und Rückkehr in den Anzeige-Modus (Gegenteil von ENTER). Wenn eine automatische Messung läuft, wird sie abgebrochen.
ANALYS.ieren	[ ] [4]	Beginn einer automatischen Messung.
EDIT (Verändern)	[ ] [5]	Editieren einer beendeten Messung oder der Kalibrationsdaten.
DRUCKEN	[ ] [6]	Drucken eines Analysen- oder Kalibrations-Protokolls über die parallele Schnittstelle. Wenn eine automatische Messung läuft, wird ein Teilbericht gedruckt, ohne die Analyse zu unterbrechen.

Tasten der oberen Reihe (Orange-farbene Beschriftung):
Aktiv, wenn die manuelle Betriebsart eingestellt ist ([ ] MANUELL):

FÜLLEN	[7]	Öffnen und Schließen des Füll-Ventils.
EXPAND.ieren	[8]	Öffnen und Schließen des Expansions-Ventils.
BELÜFTEN	[9]	Öffnen und Schließen des Belüftungs-Ventils.

Die Leuchtdiode über der jeweiligen Taste leuchtet auf, wenn das betreffende Ventil geöffnet ist und erlischt nach dem Schließen.

Durchführen einer Analyse

1. Wiegen Sie den leeren, sauberen Probenbecher.
2. Füllen Sie die Probe in den Becher und trocknen Sie sie gründlich.
3. Die vorbereitete, abgekühlte Probe wird mit dem Probenbecher erneut gewogen. Die Differenz zwischen diesem Gewicht und dem leeren Becher ergibt die Einwaage (nur für Dichtebestimmung erforderlich).
4. Öffnen Sie die Probenkammer.
5. Setzen Sie den Probenbecher ein und verschließen Sie die Probenkammer.
6. Drücken Sie die Tastenkombination für ANALYS.ieren ([ ] [4])
7. Wenn die Probenbezeichnung erfragt wird, geben Sie sie über die Tastatur ein und drücken [ENTER]. Wenn nicht, weiter bei Punkt 8.
8. Wenn das Probengewicht erfragt wird, geben Sie es über die Tastatur ein und drücken [ENTER]. Wenn nicht, weiter bei Punkt 9.
9. Drücken Sie [ENTER] zum Starten der Analyse. Mit ESCAPE ([ ] [CLEAR]) kann abgebrochen werden.
10. Während der Analyse werden auf der LCD-Anzeige Statusmeldungen angezeigt. Das Ende der Messung wird durch drei helle Töne signalisiert. Entfernen Sie die Probe aus der Probenkammer.
11. Die Meldung „NEULADEN" wird angezeigt. Drücken Sie mehrmals [CHOICE] (Auswahl), um eventuell vorhandene Fehlermeldungen sowie die Meßdaten auf der LCD-Anzeige anzuzeigen.

Ändern der Bedienungssprache (Englisch → Deutsch)

1. Warten Sie das Ende einer laufenden automatischen Messung ab.
2. Stellen Sie mit ESCAPE ([ ] [CLEAR]) den Anzeige-Modus ein.
3. Drücken Sie SET UP ([ ] [2]).
4. Drücken Sie CHOICE, bis die Anzeige „Unit Type" erscheint.
5. Drücken Sie ENTER, bis „Language?" erscheint.
6. Drücken Sie CHOICE, bis die gewünschte Sprache angezeigt wird.
7. Drücken Sie SAVE; die LCD-Anzeige zeigt die Aufforderung „Neuladen" an.

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AccuPyc 1330 Pycnometer

Operator's Manual

Part Number: 133-42801-01
22 August 1990

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.

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International Sales — (404) 662-3660
Domestic Repair Service — (404) 662-3666
Customer Service — (404) 662-3636

Form No. 008-42104-00
28 Oct 1988

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2. INSTALLATION

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4. GENERAL OPERATING INSTRUCTIONS

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6. ORDERING INFORMATION

APPENDICES

CHAPTER 1

GENERAL DESCRIPTION

- Organization of the Manual
- Equipment Description
- Pycnometer Keypad
- Specifications

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.

EQUIPMENT DESCRIPTION

The AccuPyc 1330 Pycnometer is an easy-to-use, fully-automatic gas displacement pycnometer. Analyses are initiated with a keystroke. 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 four languages: English, German, Spanish, or French. The language used may be changed by a simple keystroke sequence.

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. In addition, a serial port is provided for data transmission to other devices.

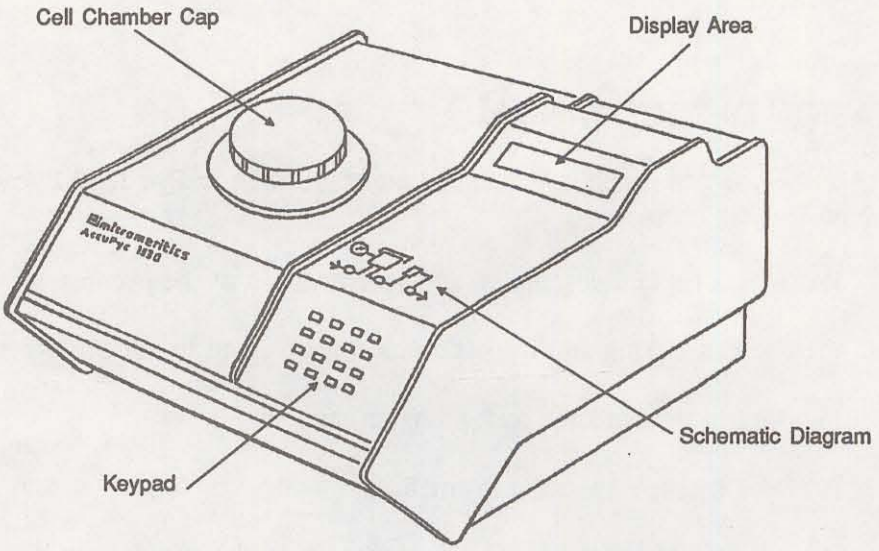


Figure 1-1. AccuPyc 1330 Pycnometer

The AccuPyc 1330 Pycnometer determines density and volume by measuring the pressure change of helium in a calibrated volume. 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.

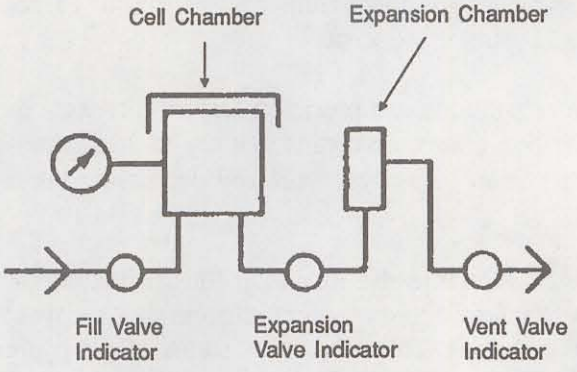


Figure 1-2. Schematic Diagram

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

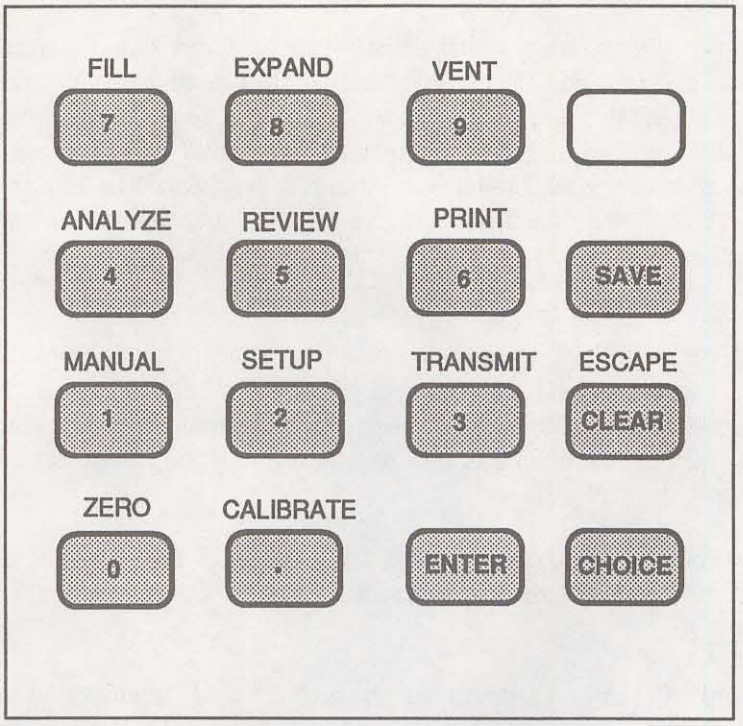
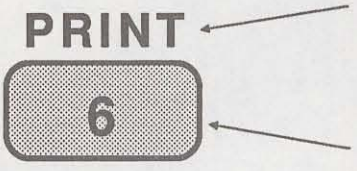


Figure 1-3. Pycnometer Keypad

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 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 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 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 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	100/120 or 220/240 VAC ±10%
Current	0.5A (100/120 VAC); 0.25A (220/240 VAC)
Frequency	50/60 Hz
Sample Volume	0.5 - 100 mL
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 mL) holder
Optional Sample Modules	Provide ranges of 1 to 100 mL
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

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.

NOTE

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.

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.

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 INPUT POWER

All instruments leave the factory set for 120 VAC and with the line fuse removed. The correct setting of the universal power entrance must be checked and the appropriate fuse installed before the pycnometer can be operated. The pycnometer is designed to operate with either 100, 120, 200 or 240 VAC at 50 or 60 Hz. Voltage selection and fusing are made at the power connector, which is located on the rear panel of the unit.

WARNING

The power cord should be disconnected from the pycnometer before removing the cover from the input power connector. Failure to disconnect the power cord could result in electrical shock.

1. Make sure the power cord is disconnected from the pycnometer.

2. Using a pointed object, remove the fuse block and cover assembly from the power connector at the rear of the pycnometer.

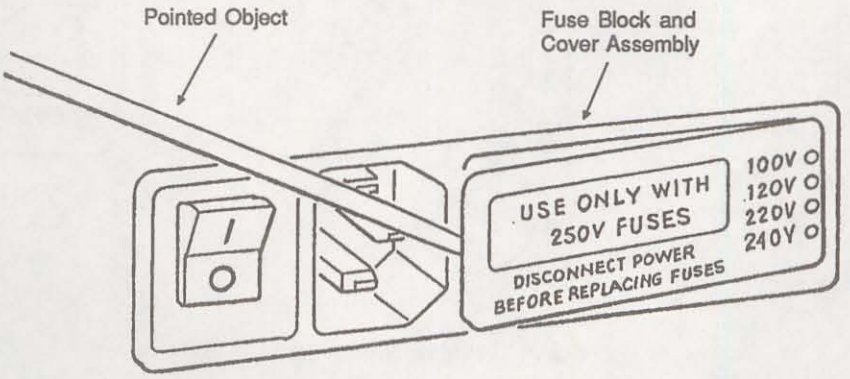


Figure 2-1. Removing Fuse Block and Cover Assembly

3. Pull the voltage selector card straight out of the power connector housing.

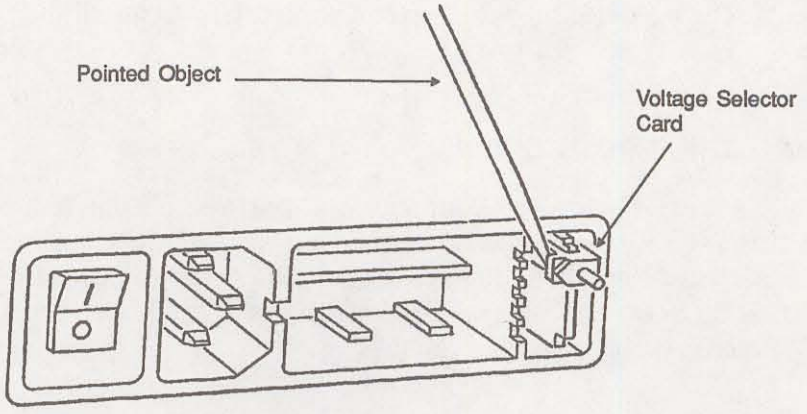


Figure 2-2. Removing Voltage Selector Card

4. Orient the voltage selector card so that the desired voltage is indicated at the bottom. Orient the indicator pin so that it points upward as shown in the following illustration.

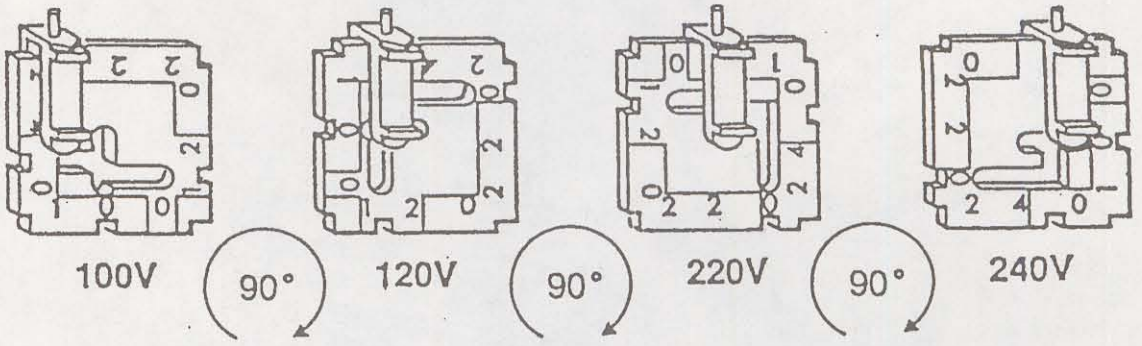


Figure 2-3. Voltage Selection

5. Insert the voltage selector card into the power connector housing with the edge containing the desired voltage first and with the printed side facing the POWER ON/OFF switch.

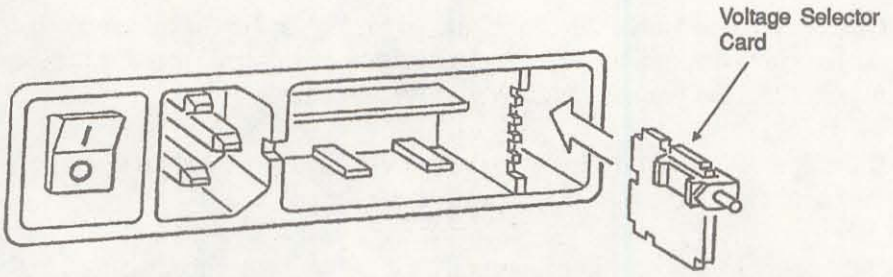


Figure 2-4. Inserting Voltage Selector Card

6. Fuse the input power line according to local safety practices. The input power connector can be used with either a single-fuse arrangement or a double-fuse arrangement, as shown in the following illustration.

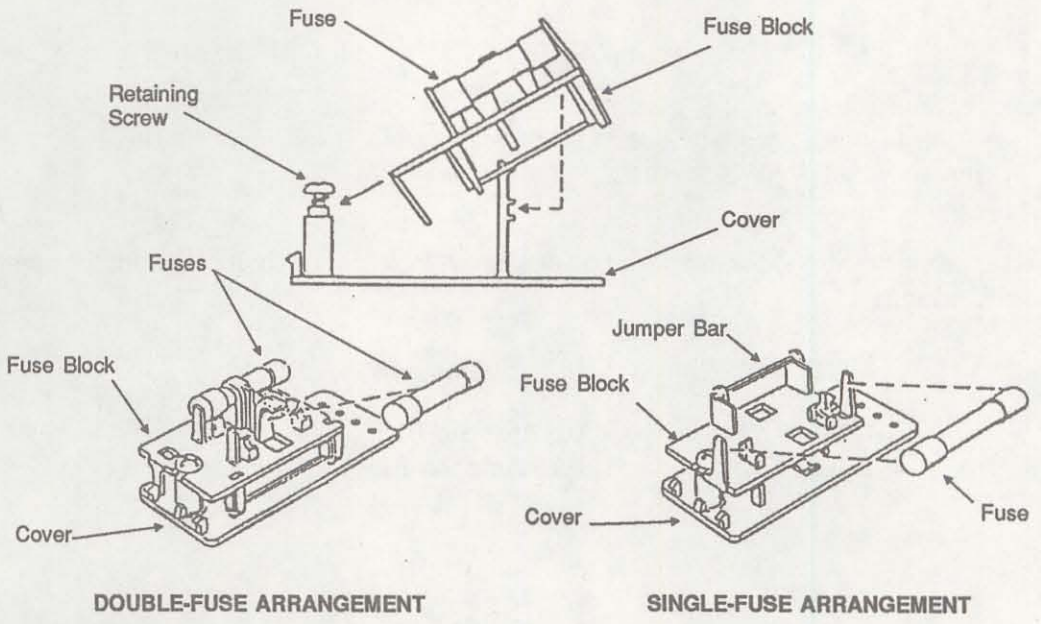


Figure 2-5. Fusing Arrangements

WARNING

The power cord should be disconnected from the pycnometer when installing or replacing fuses. Failure to do so could result in electrical shock.

a. Observe the position of the fuse block, using Figure 2-5 for reference. If the single-fuse arrangement is desired, the fuse block should be positioned such that the side with the single fuse slot and the jumper bar is away from the cover.

   If the double-fuse arrangement is desired, the fuse block should be positioned such that the side with the double fuse slots is away from the cover.

b. If the fuse block is positioned properly for the fusing desired, proceed to Step c.

   If the fuse block is not positioned properly for the fusing desired:

   1. Remove the fuse block retaining screw.
   2. Lift the fuse block from the cover.
   3. Rotate the fuse block.
   4. Mount the fuse block to the cover.
   5. Replace the retaining screw.

WARNING

The fuses used in the pycnometer must be identical in type and rating to that specified. Use of other fuses could result in electrical shock and/or damage to the pycnometer.

c. Insert appropriate fuse(s) for the input power source. Refer to the chart below for the appropriate fuse rating.

Power Source	Fuse
100-120 VAC	0.5 Amp Slo-Blo
220-240 VAC	0.25 Amp Slo-Blo

7. Insert fuse block and cover assembly into input power connector and snap it into place. Once the fuse block and cover assembly are in place, the position of the indicator pin shows the input power selected.

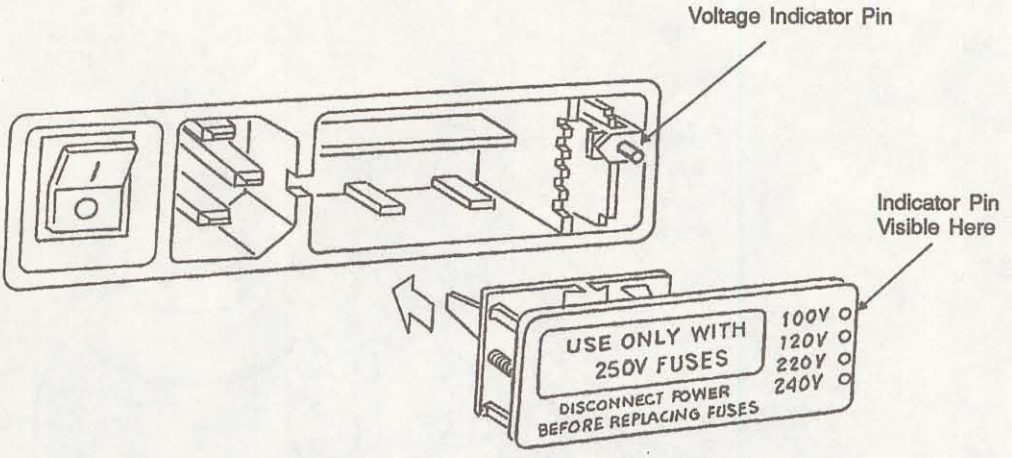


Figure 2-6. Inserting Fuse Block and Cover Assembly

REAR PANEL CONNECTIONS

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

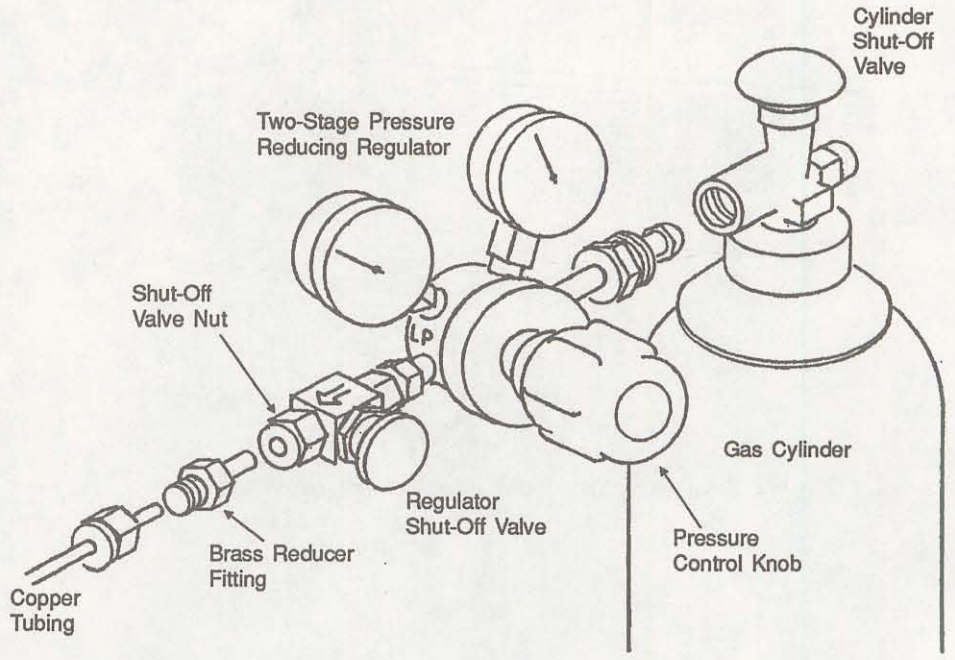


Figure 2-7. Connecting Gas Supply Cylinder to the Regulator

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.

CAUTION

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.

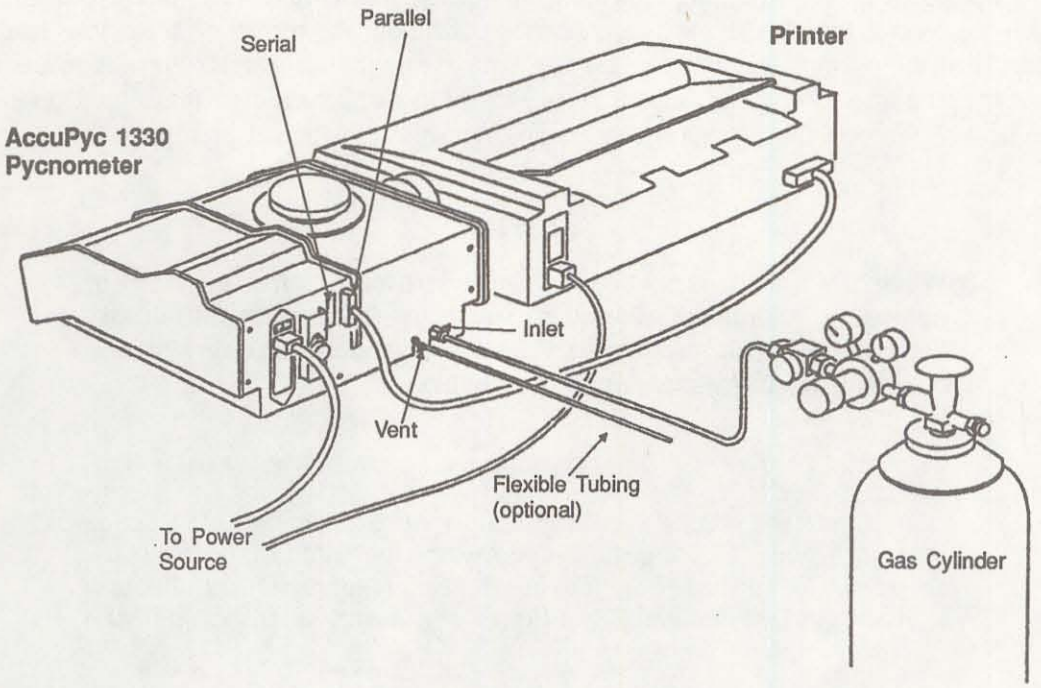


Figure 2-8. Rear Panel Connections

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 SERIAL 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 PARALLEL on the back of the pycnometer. Connect the other end to the input connector on the printer.

11. 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 (I) position. The system is automatically vented and the following prompt is displayed:

```
Reload
P = (current pressure)
```

Reload is displayed on the first line, indicating that the system is idle. The current pressure is shown 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.

NOTE

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.

CAUTION

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 ANALYSIS AND REPORT 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

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

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 four languages: English, German, Spanish, and French. 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].

NOTE

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.

CAUTION

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.

CAUTION

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.

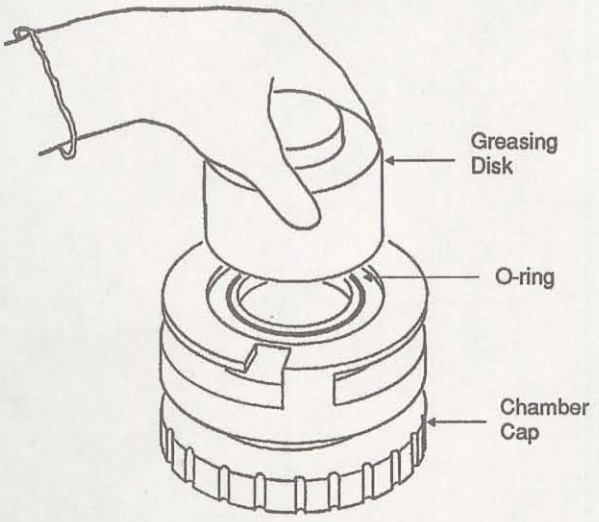


Figure 2-9. Greasing the O-Ring in Chamber Cap

4. Replace the chamber cap.

VERIFYING OPERATION

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

NOTE

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. In some languages, commas are used instead.

1. Zero the transducer by pressing [ ] [0]. The pressure display will show approximately zero.

2. 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.

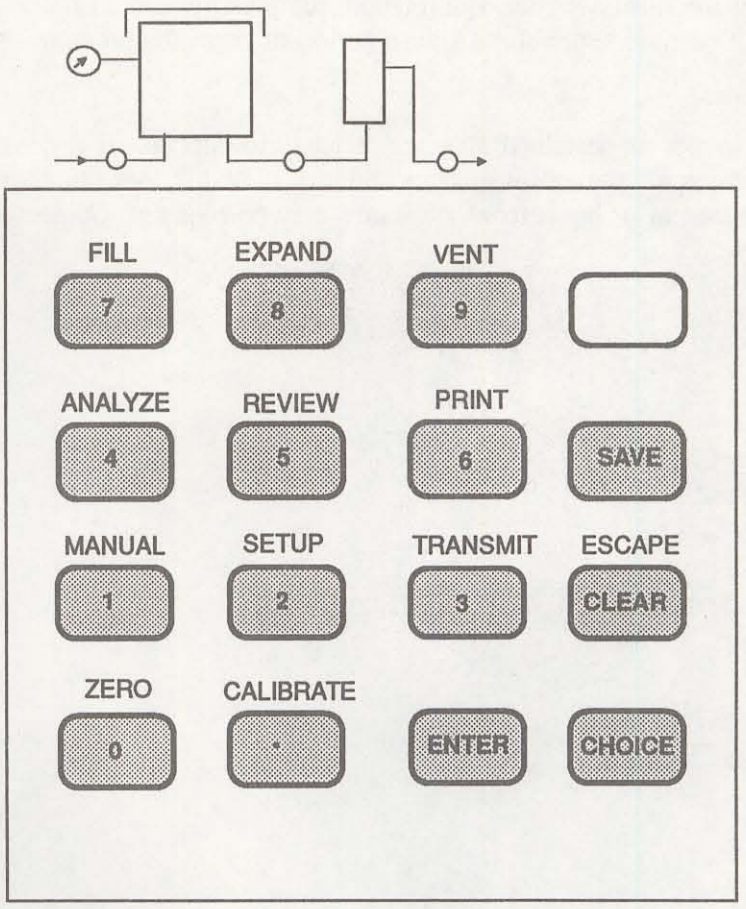


Figure 2-10. Front Panel

4. Open the fill valve. Wait until the pressure reaches 15 to 19 psig.

NOTE

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.

CAUTION

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 beginning an analysis, check the regulator pressure. Fill pressure is separately specified for purge and run; 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.

CAUTION

Keep the cap on the cell chamber except when actually inserting or removing a sample. The cell chamber must be free of particles and as dry as possible for accurate 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.

CAUTION

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.

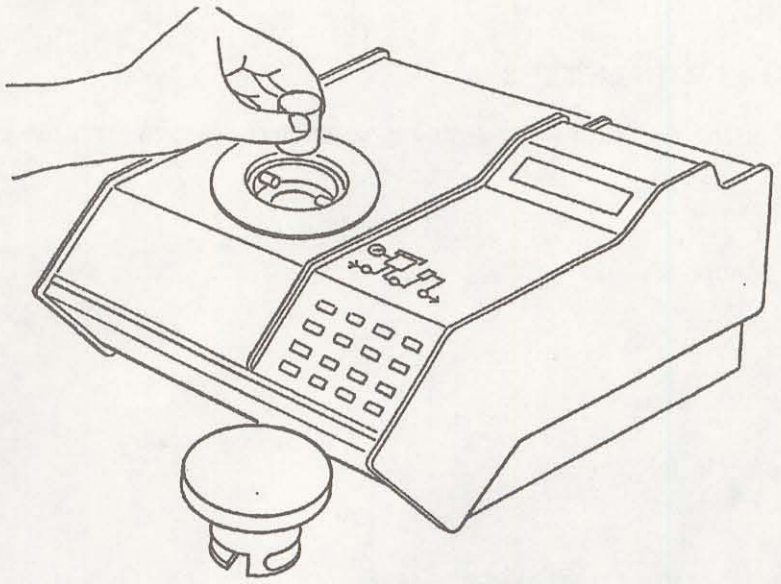


Figure 3-1. Inserting Sample Cup Into Cell Chamber

8. Replace the cell chamber cap.

CAUTION

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

STARTING THE 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

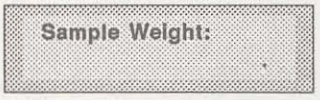
To start an analysis using analysis parameters that have been modified:

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. If there were any errors, the first error message is displayed. 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

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.

CAUTION

Keep the cap on the cell chamber except when actually inserting or removing a sample. The cell chamber must be free of particles and as dry as possible for accurate analysis results.

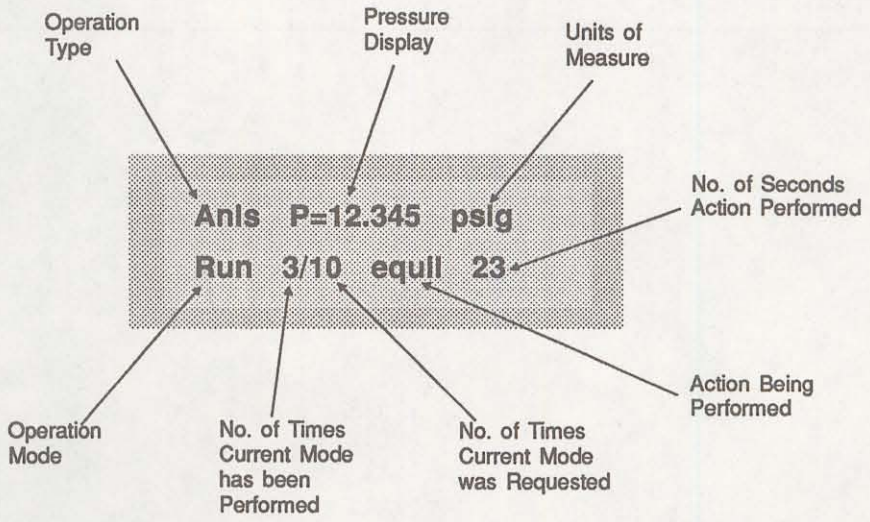
MONITORING THE SYSTEM

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 until the operation is finished. You may:

- 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.

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 message.

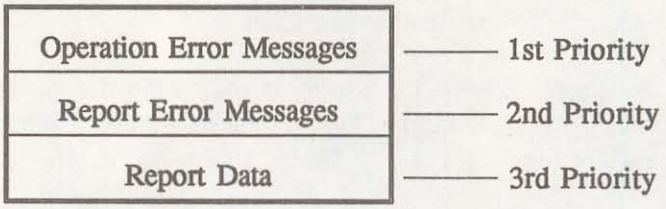


Figure 4-1. Message Queue

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

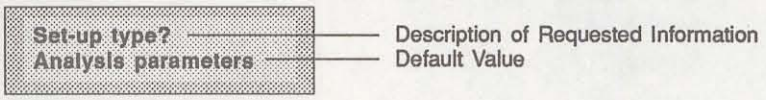
The pycnometer remains in display mode until you enter a command by pressing the appropriate keys on the keypad. Commands start an analysis or other automatic operations and allow you to modify operating parameters. The commands available are:

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

NOTE

You may not enter a command 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:



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.

The remainder of this chapter describes the commands used to begin automatic operations, review, edit, and print results, and edit operating parameters.

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]. **NOTE** **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].
Den [N] = (density) Dev [N] = (deviation) or Vol [N] = (volume) Dev [N] = (deviation)	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. Likewise, 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 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

Volume of Calibration Standard: 5.5775 cc
Number of Purges: 10 Equilibration Rate: 0.0050 psig/min

Run Pair#	Cell Volume cc	Deviation cc	Expansion Volume cc	Deviation cc
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 cc Standard Deviation: 0.0006 cc
Average Expansion Volume: 7.9985 cc Standard Deviation: 0.0005 cc

Figure 4-2. 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
Number of Purges: 20
Cell Volume: 12.3837 cc

Sample Weight: 42.6966 g
Equilibration Rate: 0.0050 psig/min
Expansion Volume: 7.9985 cc

Run#	Volume cc	Deviation cc	Density g/cc	Deviation g/cc
1	5.5767	-0.0005	7.6562	0.0007
2	5.5772	-0.0001	7.6556	0.0001
3	5.5771	-0.0001	7.6557	0.0002
4	5.5771	-0.0001	7.6557	0.0002
5	5.5777	0.0004	7.6549	-0.0006
6	5.5775	0.0003	7.6551	-0.0004
7	5.5776	0.0003	7.6550	-0.0005
8	5.5779	0.0006	7.6546	-0.0009
9	5.5774	0.0001	7.6553	-0.0002
10	5.5775	0.0002	7.6552	-0.0003
11	5.5773	0.0001	7.6554	-0.0001
12	5.5770	-0.0002	7.6558	0.0003
13	5.5776	0.0003	7.6550	-0.0005
14	5.5776	0.0004	7.6549	-0.0006
15	5.5770	-0.0002	7.6558	0.0003
16	5.5776	0.0004	7.6550	-0.0005
17	5.5766	-0.0007	7.6564	0.0009
18	5.5765	-0.0007	7.6565	0.0010
19	5.5766	-0.0006	7.6563	0.0008
20*	5.5762	-0.0010	7.6569	0.0014

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

Standard Deviation: 0.0004 cc
Standard Deviation: 0.0006 g/cc

Figure 4-3. 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.

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

Sample ID: 11-06-89--14-45
Number of Purges: 20
Cell Volume: 12.3837 cc
Sample Weight: 5.8293 g
Equilibration Rate: 0.0050 psig/min
Expansion Volume: 7.9985 cc

Run#	Volume cc	Deviation cc	Density g/cc	Deviation g/cc
1*	1.4081	0.0000	4.1397	-0.0000
2*	1.4072	-0.0010	4.1425	0.0028
3*	1.4072	-0.0009	4.1425	0.0028
4*	1.4072	-0.0010	4.1426	0.0029
5*	1.4111	0.0030	4.1310	-0.0087
6*	1.4083	0.0001	4.1393	-0.0004
7*	1.4074	-0.0007	4.1418	0.0021
8*	1.4076	-0.0005	4.1412	0.0015
9*	1.4070	-0.0011	4.1431	0.0033
10*	1.4059	-0.0023	4.1464	0.0067
11*	1.4070	-0.0011	4.1429	0.0032
12*	1.4076	-0.0006	4.1414	0.0017
13*	1.4074	-0.0007	4.1419	0.0022
14*	1.4107	0.0025	4.1323	-0.0075
15*	1.4068	-0.0013	4.1436	0.0039
16*	1.4062	-0.0020	4.1455	0.0058
17*	1.4082	0.0000	4.1396	-0.0001
18	1.4083	0.0002	4.1393	-0.0005
19	1.4084	0.0003	4.1389	-0.0009
20	1.4085	0.0004	4.1387	-0.0011
21	1.4073	-0.0008	4.1421	0.0024
22	1.4081	-0.0000	4.1397	0.0000

Average Volume: 1.4081 cc
Average Density: 4.1397 g/cc
Standard Deviation: 0.0005 cc
Standard Deviation: 0.0014 g/cc

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
```

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. To calibrate the pycnometer, follow the instructions below. When you calibrate the pycnometer, **cell volume** and **expansion volume** in the set-up parameters are updated automatically.

Press [ ] [.] to begin the calibration procedure.

Display	Entry
Volume of cal std: 1.0000 cc	Enter the volume of the calibration standard and press [ENTER]. *Range: 0.1 to 999.0000 cc*
[Enter] to start [Escape] to cancel	Press [ENTER] to begin the calibration or [ ] [CLEAR] to cancel the calibration. Status messages are displayed as the first phase of calibration progresses. When the first phase is finished, you will hear three beeps and the following prompt is displayed.
Insert cal std [Enter] to start	Insert the calibration standard in the cell chamber. 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.

NOTE

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

EDITING THE SET-UP 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. **NOTE** **This prompt applies for analysis only.**
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%* **NOTE** **Nominal cell volume is the sample capacity.**

Display	Entry
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? Analysis parameters	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. **NOTE** **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.
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.* **NOTE** **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 Volumes

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

Display	Entry
Set-up type? Analysis parameters	Press [CHOICE] until **Calibration volumes** is displayed. Then press [ENTER]. **NOTE** **When the pycnometer is calibrated, cell volume and expansion volume are automatically updated.**
Cell volume: (value from calibration)	Enter the cell volume and press [ENTER]. *Range:* *0.5 to 999.0000 cc*
Expansion volume: (value from calibration)	Enter the expansion volume and press [ENTER]. *Range:* *0.5 to 999.0000 cc*
Set up type? Calibration volumes	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.

Data Transmission

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

Display	Entry
Set-up type? Analysis parameters	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 and Operating Language

Press [ ] [2] to display or edit unit types or the operating language.

Display	Entry
Set-up type? Analysis parameters	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, German, Spanish, or French* **NOTE** **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.**
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
Unit does not work when power switch is turned on.	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.
	Wrong voltage selected.	Unplug unit. Check voltage selection board. If wrong voltage selected, correct the voltage and re-insert board.

What Happened	Why	What To Do
Unit does not work when power switch is turned on. (continued)	Fuse blown.	Unplug unit. Check fuse. Check the voltage selected; it must be appropriate for the fuse: 100-120 VAC, 0.5 Amp Slo-Blo fuse; 220-240 VAC, 0.25 Amp Slo-Blo fuse. Have an electrician check the wall outlet. Replace the fuse if blown.
	Loose internal connection or broken wire.	Contact a Micromeritics Service Representative for repair or replacement information.
Specified pressure not reached or not 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.	Clean both the chamber cap and the rim of the cell chamber by wiping with lint-free tissue. 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
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.

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.

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.

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 10 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.

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 five 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.

CAUTION

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.

CAUTION

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.

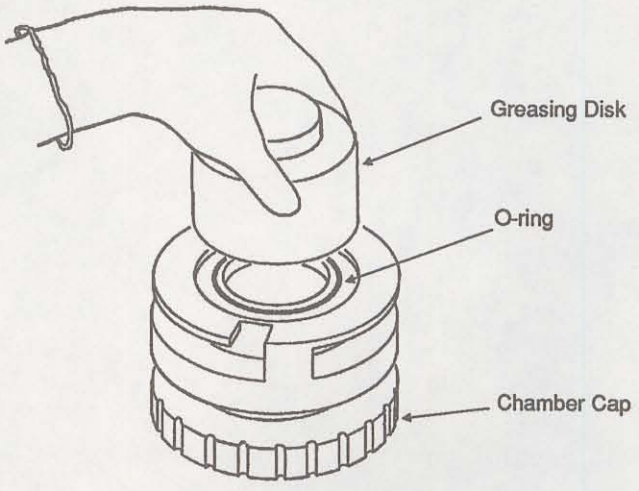


Figure 5-1. Greasing Chamber Cap O-Ring

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.

CAUTION

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

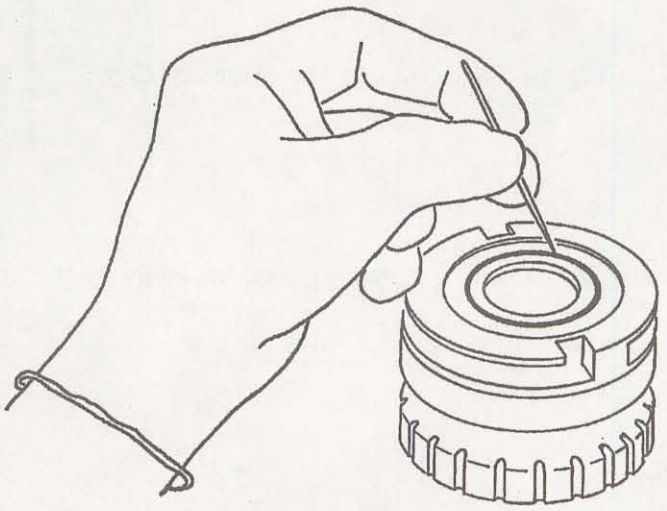


Figure 5-2. Removing the Chamber Cap O-Ring

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

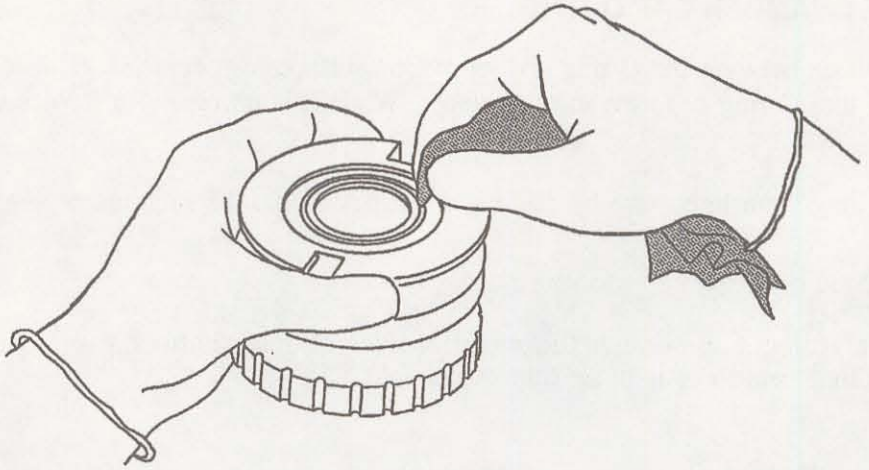


Figure 5-3. Cleaning the Chamber Cap

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.

NOTE

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. Open the EXPAND valve and close the VENT valve. (The indicator above the key is turned on when the valve is open.)
   b. Open the FILL valve and fill the sample chamber to the desired pressure.
   c. Close the FILL valve and open the VENT valve.

   Repeat this procedure two or three times.
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 0.0005 psig/min.
   - If the pressure does not vary more than 0.0005 psig/min, proceed to step 8.
   - If the pressure varies more than 0.0005 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 0.0005 psig/min.

    If the pressure varies more than 0.0005 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.

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.

WARNING

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.

CAUTION

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 restarts. Any data collected during the automatic operation will still be present, but the operation should be started again in order to produce complete results.

REPLACING FUSES

The pycnometer incorporates a fuse in the power connection slot to protect the unit from damage. If the fuse is blown, it must be replaced with another of the same type and the correct rating. Refer to **Selecting the Input Power** in Chapter 2 for fuse requirements and replacement procedures.

CHAPTER 6

ORDERING INFORMATION

ORDERING INFORMATION

Part Number	Item and Description
133-00000-00	AccuPyc 1330, fully-automatic gas displacement pycnometer with 10-mL sample capacity.
133-00001-00	AccuPyc 1330, fully-automatic gas displacement pycnometer with 1-mL sample capacity.
133-00002-00	AccuPyc 1330, fully-automatic gas displacement pycnometer with 100-mL sample capacity.
133-34803-00	Sample Module, 1-mL
133-34802-00	Sample Module, 10-mL
133-34804-00	Sample Module, 100-mL
133-25855-00	Sample Cup, 1-mL capacity
133-25805-00	Sample Cup, 10-mL capacity
133-25845-00	Sample Cup, 100-mL capacity
003-51135-00	Fuse, 3AG Slo-Blo, 0.5 Amp
003-51141-00	Fuse, 3AG, Slo-Blo, 0.25 Amp
004-25076-00	Chamber Cap O-ring, 1-mL
004-25575-00	Chamber Cap O-ring, 10-mL
004-25577-00	Chamber Cap O-ring, 100-mL
004-25633-00	Calibration Standard, 10-mL
004-25632-00	Calibration Standard, 100-mL
004-25549-00	Reducer, 1/8-in. tube x 1/4-in. tube
004-62014-01	Regulator
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

Part Number	Item and Description
133-25825-00	Greasing Disk
130-25643-00	Magnet, 1/4 x 2 inches
003-20603-04	RS-232 Cable
004-54011-00	Tool for removal of sample cup
133-42701-01	French Template
133-42701-02	German Template
133-42701-03	Spanish Template
133-42801-00	Operator's Manual
	Optional Equipment
003-33004-00	Control Module. Includes 80386SX CPU with 80387SX math coprocessor, 40 Mb hard disk, amber monitor, dot-matrix printer and cables (100/120V, 50/60 Hz)
003-33004-01	Same as P/N: 003-33004-00 except 230V, 50/60 Hz.
003-20632-00	COTERM/220 Software Kit, includes RS-232 cable and diskette.
003-20631-00	Printer
003-20615-03	Printer Ribbon

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 counterclockwise.

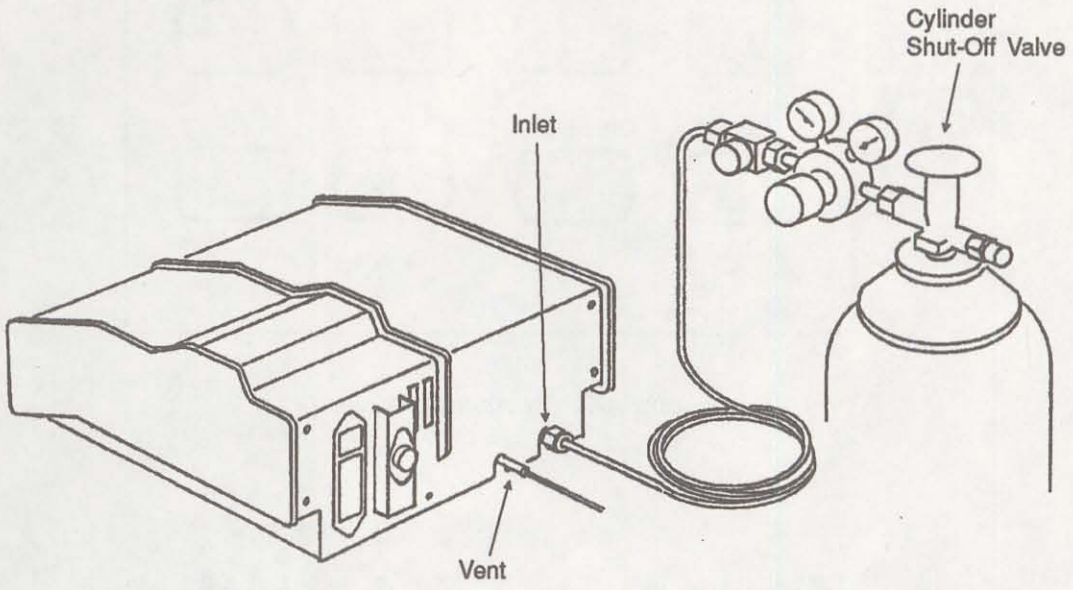


Figure A-1. Turning Off Gas Supply

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.

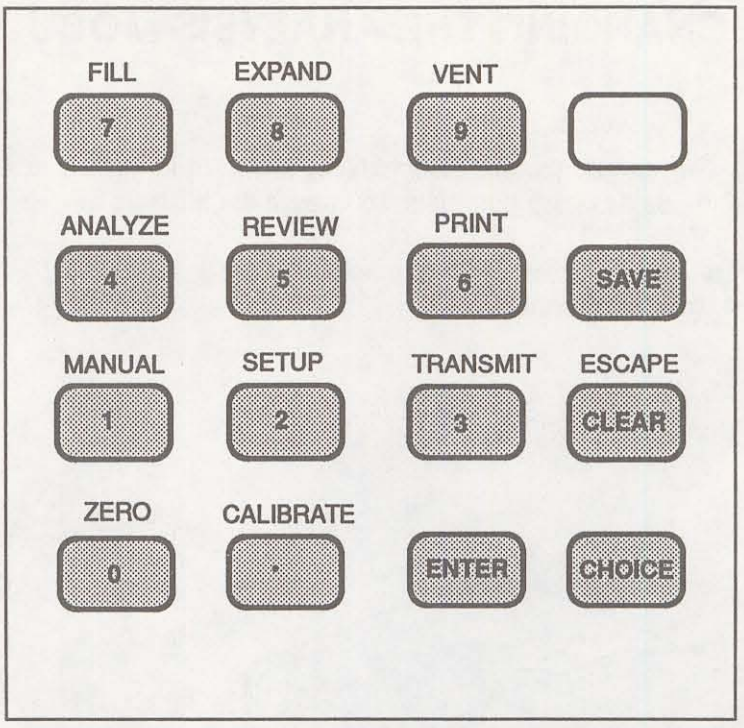


Figure A-2. Pycnometer Keypad

WARNING

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.

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 five retaining screws that hold the panel in place.

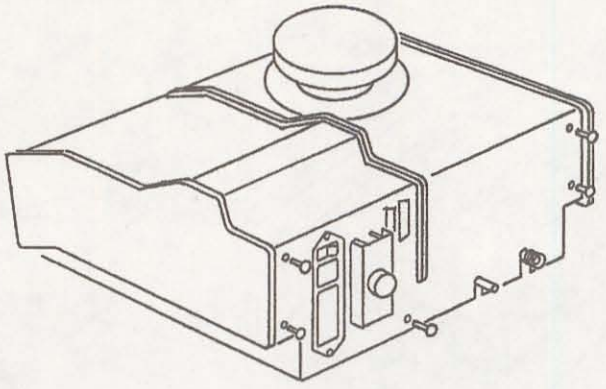


Figure A-3. Removing Rear Panel

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.

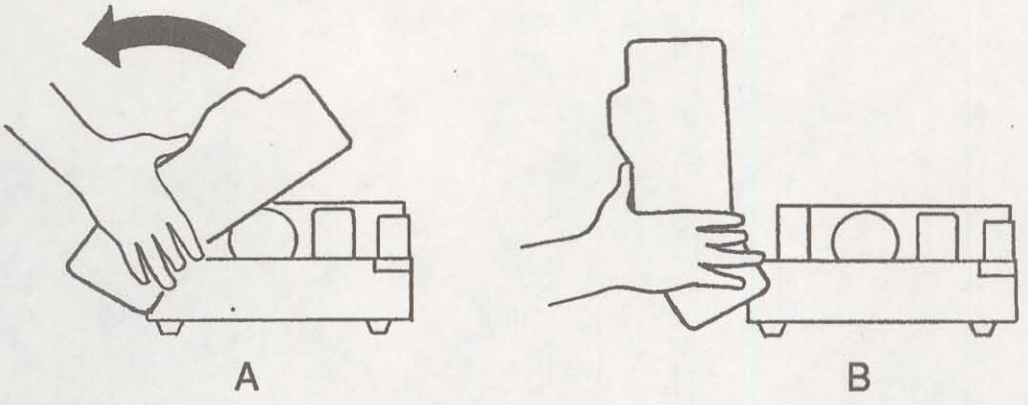


Figure A-4. Rotating Top Panel

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

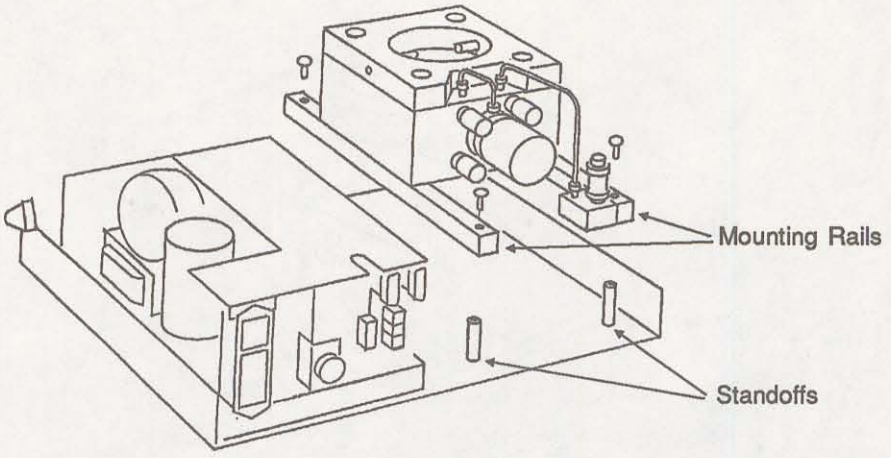


Figure A-5. Module Mounting Rails

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.

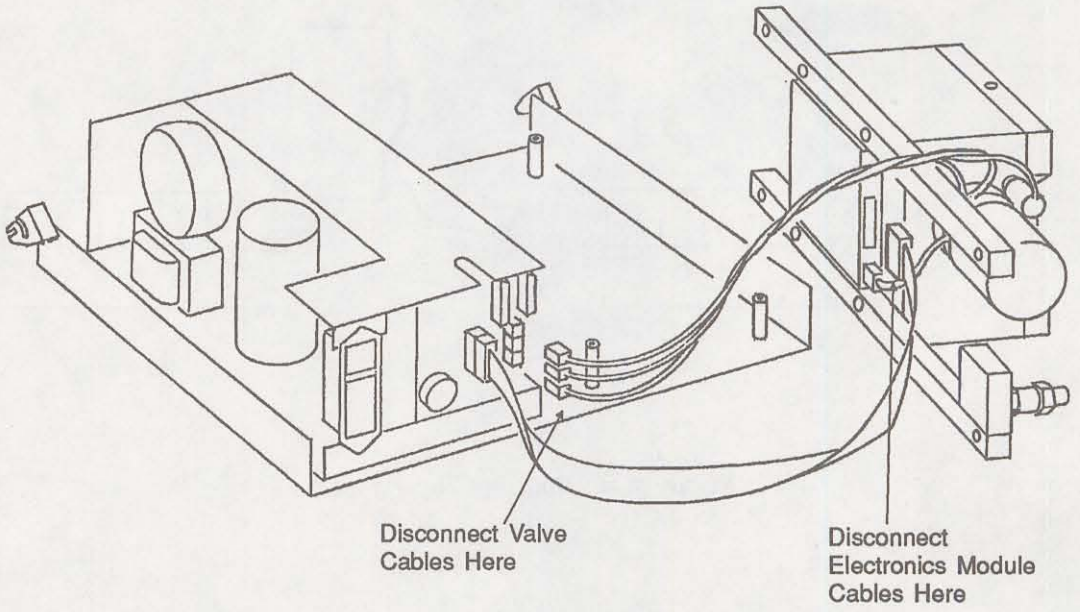


Figure A-6. Disconnecting Cable Assemblies

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 Vent valves to J4, J5, and J6 as shown in Figure A-7.

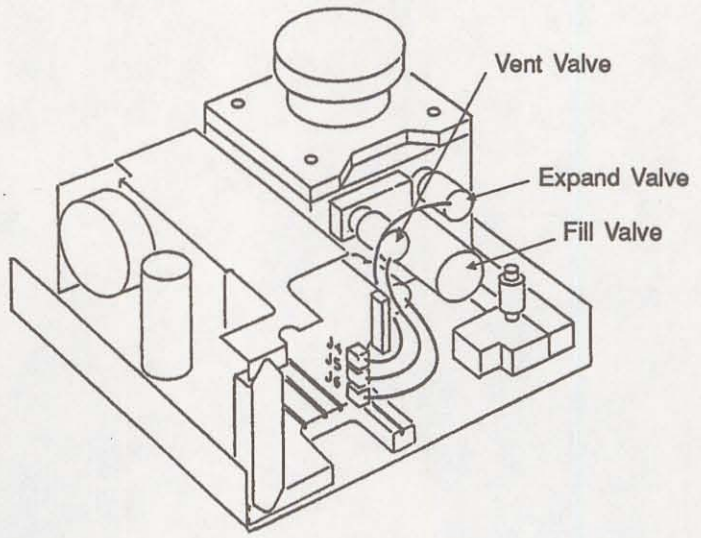


Figure A-7. Connecting Cable Assemblies

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.

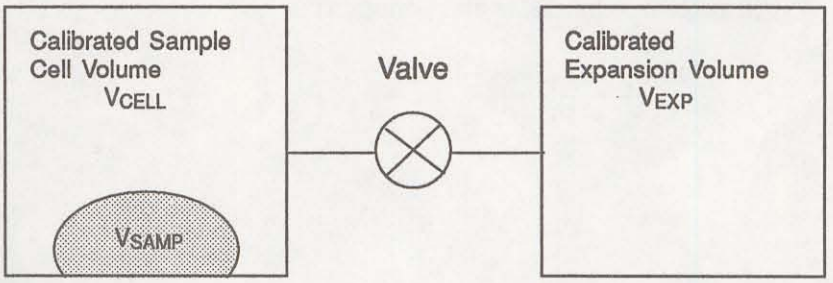


Figure B-1. Simplified Block Diagram

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

$$P_{1g} = P_1 - P_a \quad (10)$$

$$P_{2g} = P_2 - P_a \quad (11)$$

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.

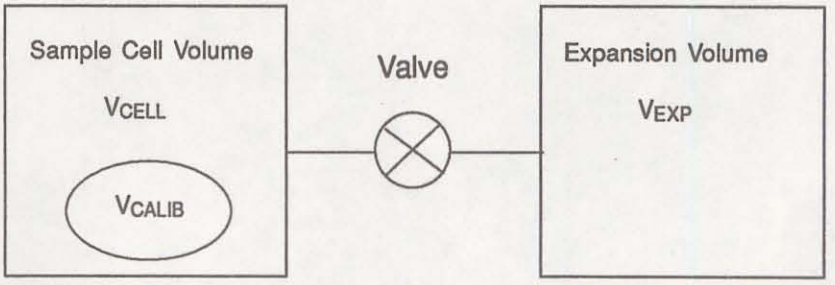


Figure C-1. Simplified Diagram for Calibration

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

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

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

The following tables describe the format and meaning of data in transmitted reports. Each record is terminated by a carriage return and line feed.

Table D-1. Calibration Report Format

Record Number	Information Conveyed	Form
1	Carriage return/linefeed	
2	Version number of the AccuPyc software	20 characters
3	Serial Number	1 integer
4	Report type = calibration	11 characters
5	Calibration standard size	1 floating pt.
6	Number of purges	1 integer
7	Equilibration rate	1 floating pt. + 9 characters
8	Average cell volume	1 floating pt.
9	Cell volume standard deviation	1 floating pt.
10	Average expansion volume	1 floating pt.
11	Expansion volume standard deviation	1 floating pt.
12	Number of runs	1 integer
13	Raw data - Pressure of data points P1 P2 P1* P2* Units (psig or kPag) xxx.xxxxbxxx.xxxxbxxx.xxxxbxxx.xxxxbcccc b = blank	 1 floating pt. each 4 characters
14	Reduced data Cell volume Cell volume deviation Expansion volume Expansion volume deviation xxx.xxxxcxxx.xxxxbxxx.xxxxcxxx.xxxx c = * or blank b = blank An asterisk after the cell or expansion volume indicates that the value is excluded from the average calculation. Records 12 and 13 are repeated for each run.	 1 floating pt. 1 floating pt. 1 floating pt. 1 floating pt.
15	No data message	40 characters

Table D-2. Analysis Report Format

Record Number	Information Conveyed	Form
1	Carriage return/linefeed	
2	Version number of the AccuPyc software	20 characters
3	Serial Number	1 integer
4	Report type = analysis	8 characters
5	Sample ID	20 characters
6	Sample weight	1 floating pt.
7	Number of purges	1 integer
8	Equilibration rate	1 floating pt. + 9 characters
9	Cell volume	1 floating pt.
10	Expansion volume	1 floating pt.
11	Average volume	1 floating pt.
12	Volume standard deviation	1 floating pt.
13	Average density	1 floating pt.
14	Density standard deviation	1 floating pt.
15	Number of runs	1 integer
16	Raw Data - Pressure of data points P1 P2 Units (psig or kPag) xxx.xxxxbxxx.xxxxbcccc b = blank	 1 floating each 4 characters
17	Reduced Data Volume Volume deviation Density Density deviation xxx.xxxxcxxx.xxxxbxxx.xxxxcxxx.xxxx c = * or blank b = blank An asterisk after the density or volume value indicates that the value is excluded from the average calculation. Records 15 and 16 are repeated for each run.	 1 floating pt. 1 floating pt. 1 floating pt. 1 floating pt.