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SANDIA NATIONAL LABORATORIES  
CHEMICAL & DISPOSAL ROOM PROCESSES DEPARTMENT 6748  
WASTE ISOLATION PILOT PLANT PROJECT

TOP-537

CALIBRATION, USE, AND MAINTENANCE OF  
THE ASAP-2000 B.E.T. SURFACE AREA ANALYZER

Revision 0

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## 1.0 REVISION HISTORY

This document replaces TOP-6119-03 draft 2. The only purpose for this revision is to comply with SNLA-WIPP QA requirements.

## 2.0 PURPOSE

This procedure provides for the calibration, operation, maintenance of the ASAP 2000 BET Surface Area Analyzer as part of the laboratory geochemistry research activities in support of the Waste Isolation Pilot Plant (WIPP) Project.

## 3.0 SCOPE

This procedure is applicable only for the Micromeritics ASAP 2000.

This document is not meant to substitute for the manufacturer's instruction manual for the ASAP 2000. The user is responsible for reading and understanding the manual (see references).

## 4.0 SAFETY

This document does not address ES&H issues. Laboratory ES&H procedures described in the SOPs of the laboratory in which the equipment is used shall be adhered to.

## 5.0 RESPONSIBILITIES

The Principal Investigator (PI), or designee, whose activities warrant the use of this procedure is responsible for implementing the requirements of this procedure.

The Project Scientist (PS), or designee, is responsible for performing the calibrations and measurements following the requirements of this procedure, documenting calibrations, and assuring that the latest revision of this document is followed.

The Quality Assurance Manager (QA Manager) is responsible for monitoring the work to assure proper implementation of the procedure and for assuring its continued effectiveness.

## 6.0 CONTROLS

Controls are established by written procedures or instructions prepared in accordance with QAP 5.3, PREPARING, REVIEWING, AND APPROVING TECHNICAL OPERATING PROCEDURES (Revision 1, effective date: 7/31/95) of the Sandia National Laboratories WIPP Quality Assurance Program. Procedures are issued in accordance with QAP 6.1, DOCUMENT CONTROL SYSTEM (Revision 1, effective date: 7/31/95) of the Sandia National Laboratories WIPP Quality Assurance Program.

## 6.1 STANDARDS

Calibration will be verified using commercially obtained surface area standards that are traceable to NIST or other nationally recognized standards. The lot numbers and expiration dates (if any) of the standards used shall be recorded in the laboratory notebook.

The standards will not be used past the expiration date listed on the container by the manufacturer.

## 6.2 FREQUENCY

The instrument will be recalibrated upon failure of a performance test.

The instrument's calibration shall be verified with performance tests immediately prior to and immediately after use. If a set of analyses are done, a performance test will be done once every 3 analyses.

## 6.3 PERFORMANCE TEST CRITERIA

Performance tests will be done by measuring the surface area of an appropriate surface area standard. If the difference between the surface area of the measurement and the known surface area of the standard is greater than 5%, the instrument shall be recalibrated.

## 6.4 SAMPLE PREPARATION

Samples will be degassed as per instructions in section 2-4F of the operator's manual (see Appendix 1), including a minimum degassing time of 3 hours, a visual inspection for condensation on the walls of the sample tube, and use of the AUTO DEGAS CHECK feature to verify that degassing is complete.

The manual's suggested degas temperature of 250°C, however, may alter the chemical and/or physical properties of some samples. In this case, the maximum safe temperature will be used. If no information about the effects of temperature on the surface area of the sample is known to exist, a temperature of 100°C for at least 12 hours will be used. If a standard has specific instructions about the temperature and duration of degassing, those instructions shall be followed.

## 6.5 GAS REQUIREMENTS

The minimum certified purity of helium to be used is 99.995%. The minimum certified purity of nitrogen to be used is 99.9%.

## 7.0 CALIBRATION

The instrument shall be recalibrated if it fails a performance test. The calibration of the manifold temperature sensor, the zeroing of the pressure gauge, and the calibration of system volumes shall be performed as specified in sections 2-4A, 2-4B, and 2-4C of the operator's manual (see pages 3 through 6 of Appendix 1).

## 7.1 CORRECTIVE ACTION

A performance test will be done immediately after calibration. If the instrument still fails, maintenance will be done (see section 9.0). If, following maintenance, the instrument still fails its performance test, it shall be tagged and placed out of service and the manufacturer (Micromeritics Instrument Corporation, Norcross, GA) shall be contacted to initiate repair.

Failures of performance tests and the remedial action taken shall be documented on the analysis printout. Failures of more than one performance test in a given day shall be documented in the appropriate scientific notebook.

## 8.0 PROCEDURE: SURFACE AREA MEASUREMENT

The sample should be weighed and analyzed immediately after degassing,

Analyses will be performed as per instructions in the operator's manual. The instructions listed in section 2-4K of the operator's manual (see pages 21 through 24 of Appendix 1) will suffice for standard BET analysis.

An  $r^2$  (correlation coefficient) of 0.995 or greater is required for the analysis to be considered valid.

## 9.0 MAINTENANCE

Maintenance and troubleshooting will be performed on the instrument as instructed in the operator's manual (see Appendix 2) with the following exception:

The Preventive Maintenance Schedule (table 9-2 in the operator's manual, see page 5 of Appendix 2) assumes the instrument will be used daily. The particular instrument used in this lab will be used much less frequently. Therefore, less frequent preventive maintenance is needed. A log of days used will be kept: After 20 days of actual use, "every 30 days" preventative maintenance will be performed. After 200 days of actual use, "annual" preventative maintenance will be performed.

## 10.0 QA RECORDS

The performance test results (including the lot numbers of the standards used), drying/degassing temperatures and durations, the  $r^2$  (correlation coefficient), and all sample analysis results will be recorded in the laboratory notebook in accordance with Sandia National Laboratories WIPP Quality Assurance Program Procedure 20-2, "PREPARING, REVIEWING, AND APPROVING SCIENTIFIC NOTEBOOKS" (Revision 1, effective date: 7/31/95). All printouts will be submitted to the SWCF.

## 11.0 REFERENCES

Micromeritics Instrument Corporation, 1993, *ASAP 2000 Accelerated Surface Area and Porosimetry System Operator's Manual*, Micromeritics Instrument Corporation, Norcross, GA

QAP 5.3, PREPARING, REVIEWING, AND APPROVING TECHNICAL OPERATING PROCEDURES (Revision 1, effective date: 7/31/95)

QAP 6.1, DOCUMENT CONTROL SYSTEM (Revision 1, effective date: 7/31/95)

QAP 20.2, PREPARING, REVIEWING, AND APPROVING SCIENTIFIC NOTEBOOKS (Revision 1, effective date 7/31/95)

## APPENDIXES

### Operator's Manual - ASAP 2000

APPENDIX 1:      Analysis              24 pages

APPENDIX 2:      Maintenance              24 pages

## 2-4. VERIFYING OPERATION

Installation of the ASAP 2000 system is now complete. It is important to perform a checkout procedure to verify proper operation. This procedure is designed to assist you in verifying that all installation procedures were performed properly and that the ASAP 2000 is in proper working order. If two analyzers are used, the procedure is designed to be performed (in its entirety) on the first analyzer, and then on the second one. This procedure should be performed following installation, and may be performed following any subsequent equipment repairs, replacements, or relocation.

The equipment and materials required for each procedure are listed in the following table.

*Table 2-2. Procedures Required to Verify Operation*

| Procedure  | Items Supplied by Micromeritics  | Items Supplied by User  |
|--|--|---|
| A. Calibrating Manifold Temperature Sensor       | None   | Thermocouple gauge or mercury thermometer   |
| B. Zeroing Pressure Gauge                        | None   | None  |
| C. Calibrating System Volumes                    | None   | None  |
| D. Cleaning and Labeling Glassware               | Sample tube<br>Filler rod<br>Sample tube brush<br>Stopper or seal frit for sample tube<br>Sample tube rack | Drying oven<br>Ultrasonic cleaning unit<br>Alconox <sup>®</sup><br>Rubber gloves or lint-free cloth<br>Acetone<br>Safety glasses<br>Waste container |
| E. Weighing Sample                               | Sample data sheet (Appendix A)<br>Sample weighing support<br>Reference material                            | Balance (scale)<br>Pipe cleaners<br>Forceps   |
| F. Degassing Sample                              | Sample tube set<br>Degas heating mantle<br>Clip for degas heating mantles                                  | None  |
| G. Unloading Sample Following Degassing          | None   | None  |
| H. Transferring Degassed Sample to Analysis Port | Sample tube set<br>Isothermal jacket for sample tube   | None  |

**Table 2-2. Procedures Required to Verify Operation (continued)**

| Procedure                               | Items Supplied by Micromeritics   | Items Supplied by User                                 |
|---|---|--|
| <b>I. Installing Cold Trap Dewar</b>    | 1.9 Liter Dewar<br>Dewar stopper/insulator (cold trap)                                | Liquid nitrogen<br>Safety glasses<br>Insulating gloves |
| <b>J. Installing Analysis Dewar</b>     | 1.9 Liter Dewar<br>Dewar stopper/insulator (analysis)<br>Dipstick (Dewar depth gauge) | Liquid nitrogen<br>Safety glasses<br>Insulating gloves |
| <b>K. Performing Analysis of Sample</b> | None  | None   |

The control module, video monitor, keyboard, printer, and Analysis Program must have been installed as described earlier in this section of the manual.

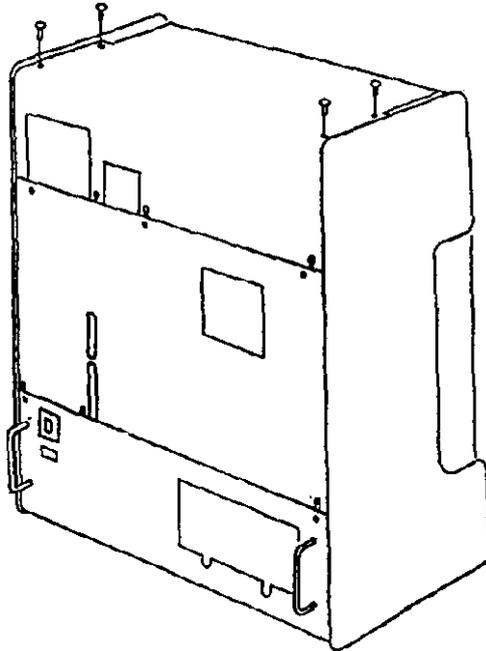
#### NOTE

The steps must be followed exactly as specified. Read each step carefully before attempting to complete it.

### A. CALIBRATING MANIFOLD TEMPERATURE SENSOR

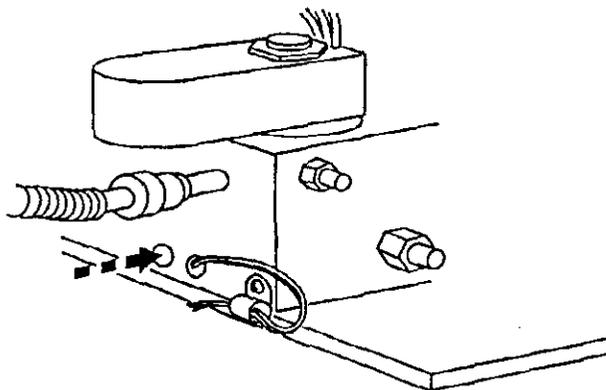
The temperature sensor calibration may change due to aging of the sensor. Annual calibration is recommended. Control of the system during this and the remaining procedures will be through keyboard entries specified by the procedure. However, the step-by-step procedure must be followed exactly as specified. To calibrate the manifold temperature sensor:

1. Remove the four screws (two each side) at the top of the analyzer. Then loosen the three screws along the lower edge of the analyzer top panel. Remove loose shipping material. Lift off the metal enclosure above the analysis valves to gain access to the manifold.



*Figure 2-28. Removing Analyzer Top Panel*

2. Determine the temperature of the analysis manifold by inserting a calibrated standard device (e.g., thermocouple gauge or mercury thermometer) into the hole in the side of the manifold as shown in the following figure. Allow the temperature to stabilize.



*Figure 2-29. Determining Analysis Manifold Temperature*

3. With the Main Function Menu displayed on the video monitor screen, press **F6**. The Status/Control Menu is displayed. Then press **F6**. The Calibrate Manifold Temperature Sensor screen is displayed.

Observe the Unit Number indicated on the video monitor. This number should be 1. If this procedure is being used for operational verification of a second analyzer, press **5** on the cursor control keypad, and then press **←**. However, if this procedure is being used for operational verification of either a single analyzer or the first in a dual-analyzer system, just press **→**.

Enter the manifold temperature indicated on the calibrated device inserted into the hole in the manifold. Press **F2** to store the new value. Then press **F2** to return to the Main Function Menu.

4. Remove the calibrated device, replace the metal enclosure, then replace analyzer top panel. Insert four screws at top of analyzer and tighten those on the lower edge.

## B. ZEROING PRESSURE GAUGE

The status of the unit must be IDLE before this procedure can be performed. On krypton units, make sure the molecular drag pump is ON. To zero the pressure gauge:

1. Set vacuum level to 10  $\mu\text{mHg}$  (5  $\mu\text{mHg}$  for krypton units) using the ANALYSIS VAC SET switch. Press the + switches to increase the value or the — switches to decrease the value. or krypton units, set the
2. With the Main Function Menu displayed on the video monitor, press **F8**. The Status/Control Menu is displayed. Then press **F7**. The Zero Pressure Gauge screen is displayed.

Observe the Unit Number indicated on the video monitor. This number should be 1. If this procedure is being used for operational verification of a second analyzer, press **F5** on the cursor control keypad, and then press **←**. If the analyzer was installed for krypton use, you may choose to zero the 1000 mmHg transducer only or both the 1000 mmHg and 10 mmHg transducers. The selection is made at the Pressure gauge selection? prompt by pressing **F5** on the cursor control keypad.

Press **F6**. Automatic zeroing occurs and the Status/Control Menu is displayed. When the Status/Control Menu is displayed, press **F2** to return to the Main Function Menu.

3. Set VAC Vacuum level to 25  $\mu\text{mHg}$  (leave at 5  $\mu\text{mHg}$  for krypton units) using the ANALYSIS VAC SET switches. Press the + switches to increase the value or the — switches to decrease the value.

### C. CALIBRATING SYSTEM VOLUMES

This procedure must be completed after calibrating the manifold temperature sensor and zeroing the pressure gauge. The status of the unit to be calibrated must be IDLE before beginning the calibration. To calibrate system volume:

1. With the Main Function Menu displayed on the video monitor, press **F8**.
2. The Status/Control Menu is displayed. Press **F5**. The Calibrate System Volumes screen is displayed.

Observe the Unit Number indicated on the video monitor. This number should be 1. If this procedure is being used for operational verification of a second analyzer, press **F5** on the cursor control keypad, and then press **←**. However, if this procedure is being used for operational verification of either a single analyzer or the first in a dual-analyzer system, just press **←**.

Press **←** twice. Then press the **F5** key on the cursor control keypad to select yes for calibrating system volumes. Press **←**.

Open the door to the calibration chamber on the rear of the analyzer. Remove the chamber cap by unscrewing it. Verify that the reference ball is in the chamber. If the reference ball is not in the chamber, place it inside the chamber, replace the cap, close the door, and allow 10 minutes for thermal equilibration. If the reference ball was in the chamber, replace cap, close the door, and proceed.

Press **PageDn** to start the automatic calibration process.

When the Remove reference volume... message is displayed, open the calibration chamber door, remove the cap, remove the reference ball, replace the cap and close the chamber door.

Press **PageDn** to continue the automatic calibration process.

When the Replace reference volume... message is displayed, open the calibration chamber door, replace the reference ball, and close the chamber door.

Press **PageDn**. Automatic calibration will continue until complete.

Record the measured system volume and measured lower volume displayed on the video monitor screen.

Press **PageDn** to enter the measured system volume and the measured lower volume. The Status/Control Menu is displayed. Then press **F2** to display the Main Function Menu.

#### D. CLEANING AND LABELING GLASSWARE

In order to obtain accurate analysis results, the glassware (sample tube, filler rod, etc.) must be clean. The following is recommended:

1. Turn on the drying oven to be used for heating the sample, and set the oven temperature to 110°C.
2. Check the bowl of the ultrasonic cleaning unit to make sure that it is clean.
3. Using a ratio of 5 grams of Alconox (or equivalent) per 500 mL of warm water, fill bowl of ultrasonic unit with enough water to cover the entire sample tube and filler rod. Make sure Alconox is dissolved before placing sample tube and filler rod into water. If too much Alconox is used, it may be difficult to rinse from the sample tube.
4. Fill the sample tube with warm water and place it in the bowl of the ultrasonic cleaning unit. Place the filler rod in the bowl also. Turn on the ultrasonic cleaning unit for approximately 15 minutes.
5. Using either rubber gloves or a lint-free cloth (but not bare hands), remove the filler rod from the bowl. Remove the sample tube from the bowl.
6. Clean the interior of the sample tube with the brush supplied with the ASAP 2000 system.

#### WARNING

When using acetone, it is recommended that safety glasses be worn to protect the eyes if splashing occurs and that the operation be conducted under a vent hood.

#### WARNING

Do not pour acetone down sink drain. Consult the appropriate Material Safety Data Sheet (MSDS) for proper disposal procedure.

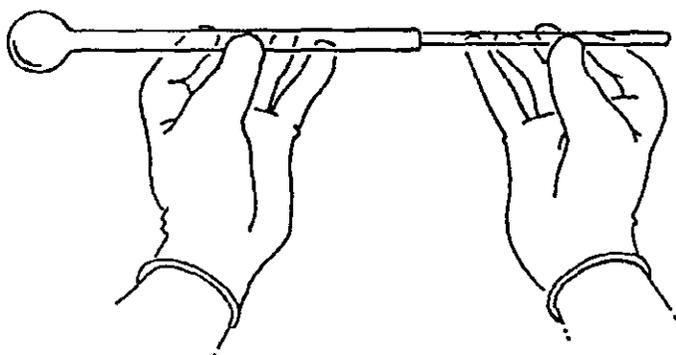
7. Rinse the sample tube and filler rod thoroughly with hot water. Then rinse the sample tube and filler rod with acetone, using a waste container to collect used acetone.
8. Dry the interior of the sample tube using nitrogen, under a vent hood, with a tubing extension long enough and small enough in diameter to fit inside the sample tube. Also dry the filler rod.

- Stand the sample tube on a rack in the oven (or on the sample tube rack supplied with the ASAP 2000 system) and place the filler rod in a basket or in the rack. Bake the sample tube and filler rod for two hours.

**CAUTION**

Do not hold the sample tube in a vertical position when inserting the filler rod. Dropping the rod directly into the sample tube can break the sample tube.

- Remove the sample tube from the oven and place a filler rod in each sample tube by holding the sample tube horizontally and sliding the filler rod into the sample tube slowly.



*Figure 2-30. Inserting Filler Rod into Sample Tube*

- Wipe a rubber stopper (or seal frit, if used with 1/2-inch tubes) with a lint-free cloth and place the stopper or seal frit into the opening in the sample tube. Label the sample tube and stopper or seal frit for identification.

## E. WEIGHING SAMPLE AND TUBE SET AND SAMPLE

Since the analysis results are expressed in units of surface area per gram of sample, the true weight of the sample must be known. This procedure is used to determine the weight of the sample before it is degassed. The procedure is as follows:

1. Fill out the top portion of a Sample Data Sheet (Appendix A) for the sample to be used as follows:

|                       |   |
|-----------------------|---|
| <b>SAMPLE NUMBER:</b> | 1 if during installation of the system; or the next consecutive number if sample files already exist in the system. |
| <b>SAMPLE ID:</b>     | Reference Material  |
| <b>SUBMITTER ID:</b>  | N/A   |
| <b>OPERATOR ID:</b>   | your name   |
| <b>REPORT TITLE:</b>  | Installation  |
| <b>SAMPLE WEIGHT:</b> | Enter the sample weight following degassing of sample   |

Fill out the DEGAS INFORMATION portion as follows:

|                       |               |
|-----------------------|---------------|
| <b>DEGAS STATION:</b> | Left or Right |
| <b>TEMPERATURE:</b>   | 200°C         |
| <b>TIME:</b>          | 3 Hours       |

2. Place the sample weighing support on the balance for weighing sample. Tare balance and allow it to stabilize at zero (0).
3. Place the sample tube set with stopper or seal frit onto sample weighing support. Record stabilized weight on Sample Data Sheet as EMPTY SAMPLE TUBE WEIGHT. Remove sample weighing support and sample tube set from balance.
4. Place a sample weighing container onto the balance. Tare the balance and allow it to stabilize at zero (0).

### NOTE

Do not touch sample (reference material) with bare hands while completing Step 5. This could affect the accuracy of results.

5. Add 10 to 14 pellets (approximately 0.5 to 0.7 grams) of reference material to the weighing container slowly, using forceps. Record weight of reference material on Sample Data Sheet as SAMPLE WEIGHT (Before Degas).

#### NOTE

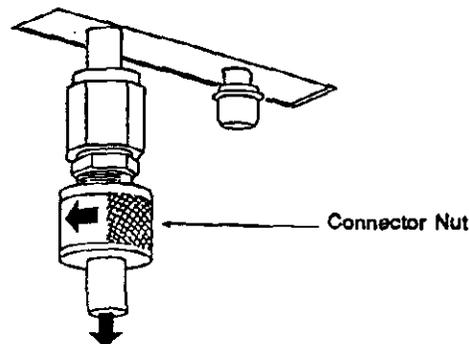
When removing filler rod from sample tube in Step 6, do not touch filler rod with bare hands. Also, hold finger over sample tube opening while rubber stopper or seal frit is removed. If some reference material clings to the top 5 cm of the inside of the sample tube, remove it using pipe cleaners.

6. Remove rubber stopper or seal frit and filler rod from sample tube. Pour reference material from weighing container into sample tube using funnel.
7. Replace filler rod in sample tube, and insert rubber stopper or seal frit.
8. Weigh the sample tube/sample (reference material)/rod/stopper or seal frit combination and record the weight on the Sample Data Sheet as SAMPLE PLUS SAMPLE TUBE WEIGHT (Before Degas).

## F. DEGASSING SAMPLE

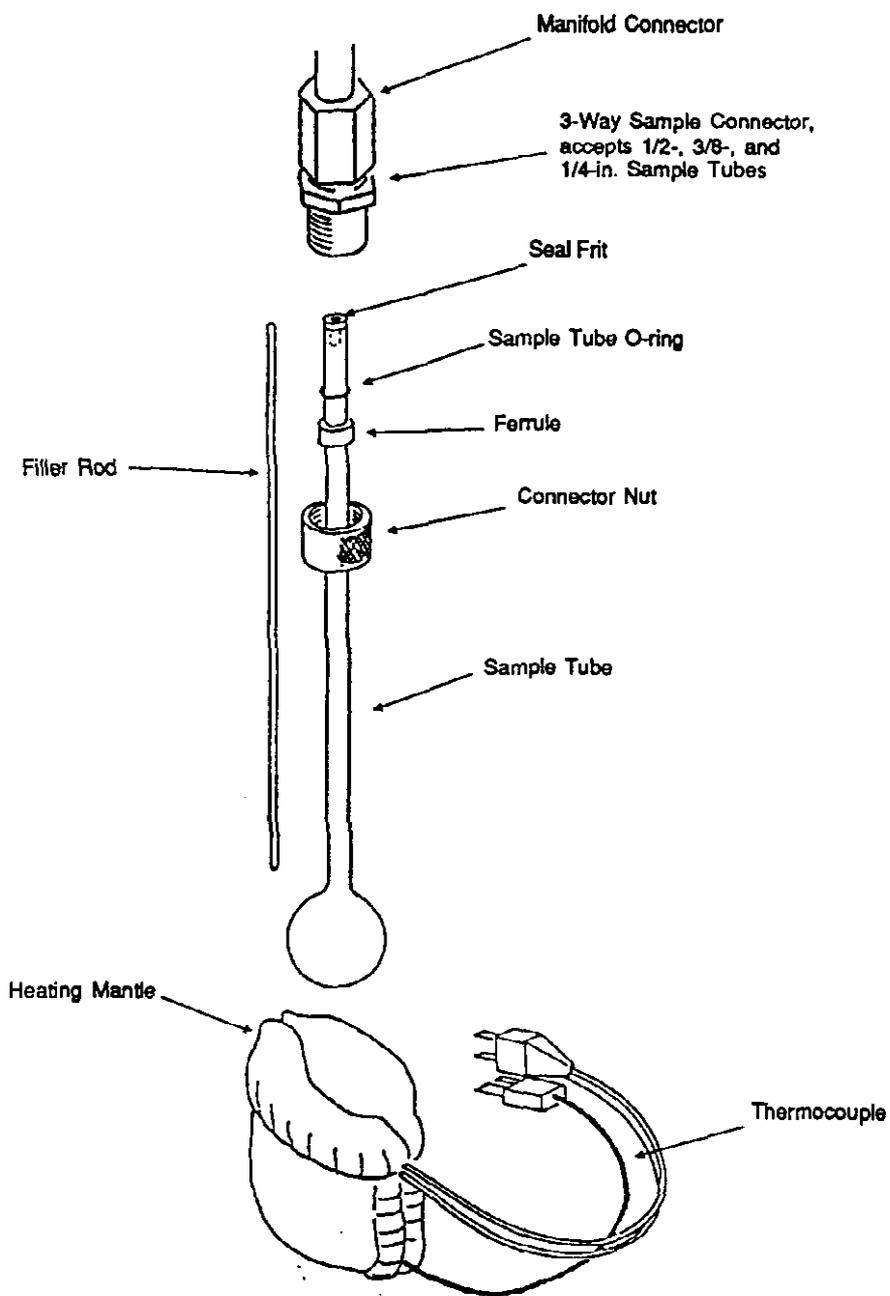
Most solid materials absorb moisture and other contaminants when exposed to the atmosphere. The sample must be clean when analysis is performed. The sample is heated and placed under vacuum to remove the moisture and other contaminants. This process is referred to as degassing the sample. The procedure is as follows:

1. While holding the steel degas port plug, remove the connector nut and plug from the degas port by turning the connector nut counterclockwise.



*Figure 2-31. Removing Degas Port Plug*

2. Place degas port connector nut, ferrule, and O-ring onto the sample tube set as shown in the following figure.



*Figure 2-32. Preparing and Attaching Sample Tube to Degas Port*

3. Remove rubber stopper or seal frit from sample tube, and attach sample tube set to degas port. Be sure to push the sample tube in to a full stop. Secure sample tube set in place by sliding connector nut, ferrule, and O-ring up to degas port and turning connector nut clockwise. Tighten nut securely (by hand).
4. Place heating mantle over bulb of sample tube set, and secure mantle in place with mantle clip.
5. Insert heating mantle thermocouple plug into appropriate connector on the front of the analyzer. Then insert heating mantle power plug into appropriate connector on the front of the analyzer. Make sure that both plugs are inserted completely.

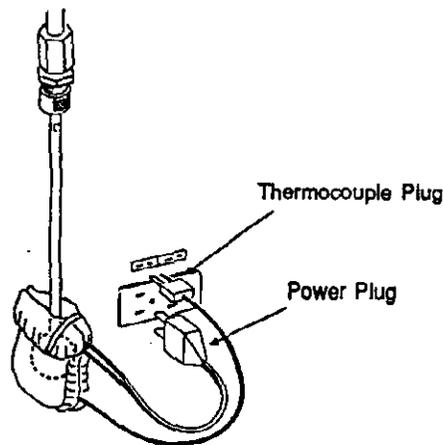


Figure 2-33. Attaching Heating Mantle to Sample Tube

6. Observe the degas portion of the analyzer control panel. Press the AUTO/MANUAL switch down to place the degas system in AUTO (automatic) mode. The indicator on the left side of the switch will be illuminated.

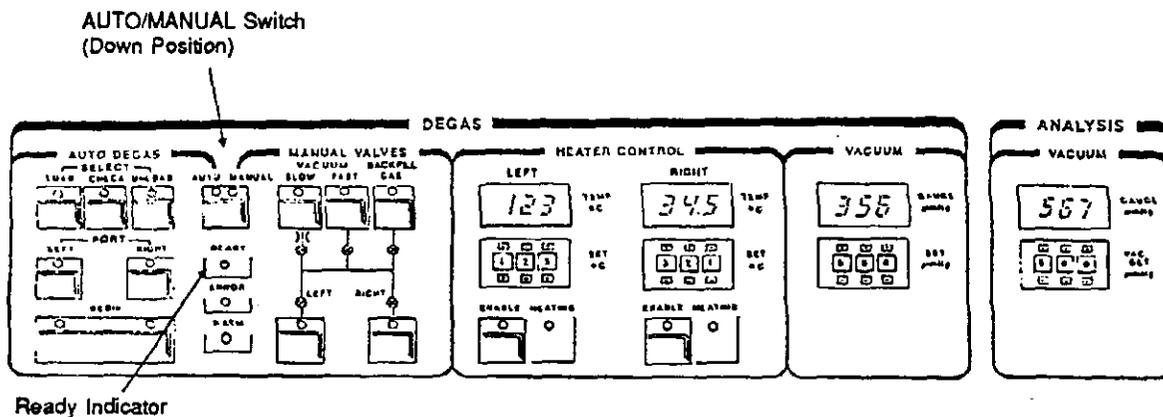


Figure 2-34. ASAP 2000 Analyzer Control Panel

- Set the degas vacuum level to 500  $\mu\text{mHg}$ . The current setting is shown in the DEGAS VACUUM SET indicator. To increase the setting, press the + switches. To decrease the setting, press the - switches.

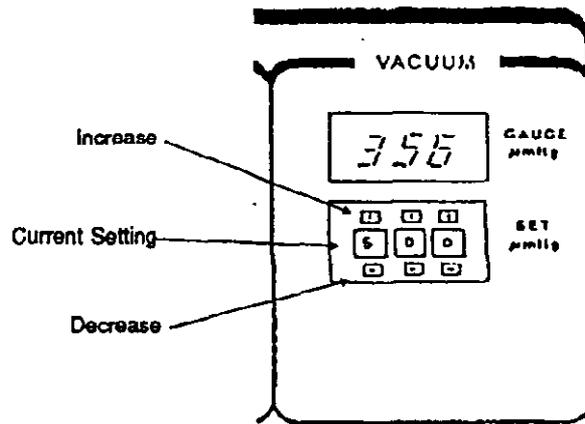


Figure 2-35. Setting Degas Vacuum Level

- Set the temperature for the degas port to 250°C. The current temperature is shown in the DEGAS TEMP SET indicator. To increase the temperature, press the + switches. To decrease the temperature, press the - switches. Refer to the following figure.
- Enable the heater for the degas port used by pressing the ENABLE switch down. The HEATING indicator on the ENABLE switch will be illuminated. The HEATING indicator next to the ENABLE switch will flash when power is applied to the heater.

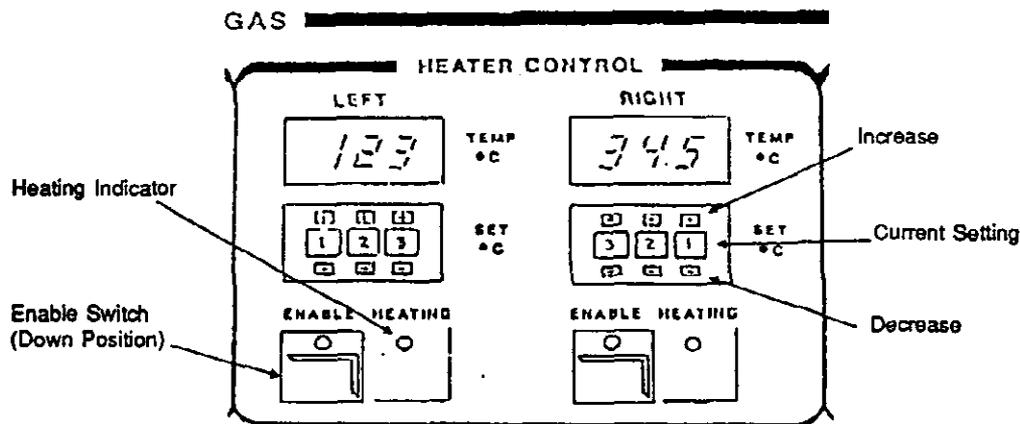
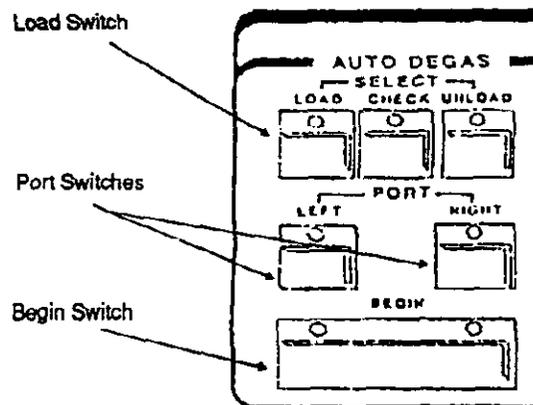


Figure 2-36. Setting Temperature and Enabling Heater for Degas Port

10. Press the **LOAD** switch as a preliminary to automatic sample degassing. The indicator on the **LOAD** switch will be illuminated.



*Figure 2-37. Loading Sample and Selecting Degas Port for Automatic Degassing*

11. Select the port containing the sample to be automatically degassed by pressing the **LEFT PORT** or **RIGHT PORT** switch. The indicator on the switch will be illuminated.
12. Press the **BEGIN** switch to start the automatic degassing operation selected (**LOAD**, in this case). The indicators on the **BEGIN** switch will be illuminated to indicate that the **LOAD** operation is activated.
13. Allow the sample to be automatically degassed for at least three hours after the **READY** indicator is turned on. If desired, the degree to which the sample has been degassed may be checked during this period as follows:
- Press the **AUTO DEGAS CHECK** switch. The indicator on the switch will be illuminated.
  - Press the appropriate switch (either **LEFT PORT** or **RIGHT PORT**) for the degas port to be checked. The indicator on the switch will be illuminated.
  - Press the **BEGIN** switch to start the **CHECK** operation. The appropriate indicator on the switch (either left or right) will be illuminated.
  - Observe the **DEGAS VACUUM GAUGE** to see if there is an increase in the pressure indicated. If so, moisture is still being released from the sample and further degassing is required.
  - Wait for the **READY** indicator to illuminate or press **BEGIN** to resume degassing.

#### NOTE

If the **DEGAS ERROR** indicator is illuminated, refer to Table 9-1, Chapter 9, for corrective action.

## G. UNLOADING SAMPLE FOLLOWING DEGASSING

Once sample degassing is complete, the sample tube must be allowed to cool before it is handled. Also, the pressure in the degas port must be increased to a level just above atmospheric pressure so that when the sample tube is removed, contaminants are not drawn into the sample tube. To unload the sample tube:

1. Disable the heater for the appropriate degas port (either left or right) by pressing the LEFT ENABLE switch or the RIGHT ENABLE switch. The switch will be in the UP position; the indicator on the switch and the HEATING indicator will turn off.

### WARNING

Do not touch the sample tube and/or the heating mantle until they have been allowed to reach room temperature. Touching the sample tube or mantle could result in personal injury, such as burns.

2. Remove heating mantle clip and heating mantle from sample tube, and allow sample tube to cool to room temperature (approximately 15 minutes).
3. Press the AUTO DEGAS UNLOAD switch. The indicator on the switch will be illuminated.
4. Press the appropriate switch (either LEFT PORT or RIGHT PORT) for the degas port to be unloaded. The indicator on the switch will be illuminated.
5. Press the BEGIN switch to continue the UNLOAD operation. The indicators on the switch will be illuminated indicating the operation is in process.
6. Wait until the READY indicator is turned on. This indicates that the UNLOAD operation is complete and that the degassed sample can be removed from the degas port for analysis.

## H. TRANSFERRING DEGASSED SAMPLE TO ANALYSIS PORT

The sample tube must be removed from the degas port, weighed and then placed onto the analysis port before the sample can be automatically analyzed.

### NOTE

**If the sample tube is not to be mounted to the analysis port immediately, either leave it on the degas port or remove it and insert the rubber stopper or seal frit into the sample tube opening.**

1. While holding the sample tube, loosen the port connector nut, and remove sample tube from degas port. Stopper immediately. Remove connector nut, ferrule, and O-ring from sample tube stem.
2. Weigh sample tube set. Enter weight on Sample Data Sheet as **SAMPLE PLUS SAMPLE TUBE WEIGHT (After Degas)**. Copy **EMPTY SAMPLE TUBE WEIGHT** from previous entry. Subtract **EMPTY SAMPLE TUBE WEIGHT** from **SAMPLE PLUS SAMPLE TUBE WEIGHT** to determine weight of sample. Enter weight of sample on Sample Data Sheet as **SAMPLE WEIGHT**.
3. Slide isothermal jacket down over sample tube stem until it touches sample tube bulb.
4. Place sample tube Dewar cover over sample tube stem just above isothermal jacket. Place connector nut, ferrule, and O-ring onto sample tube stem. For straight wall tubes, attach the sample tube clip. (See Figure 2-38.)
5. Attach sample tube to analysis port. Secure in place by screwing connector nut onto analysis port connector. Hand tighten connector nut.

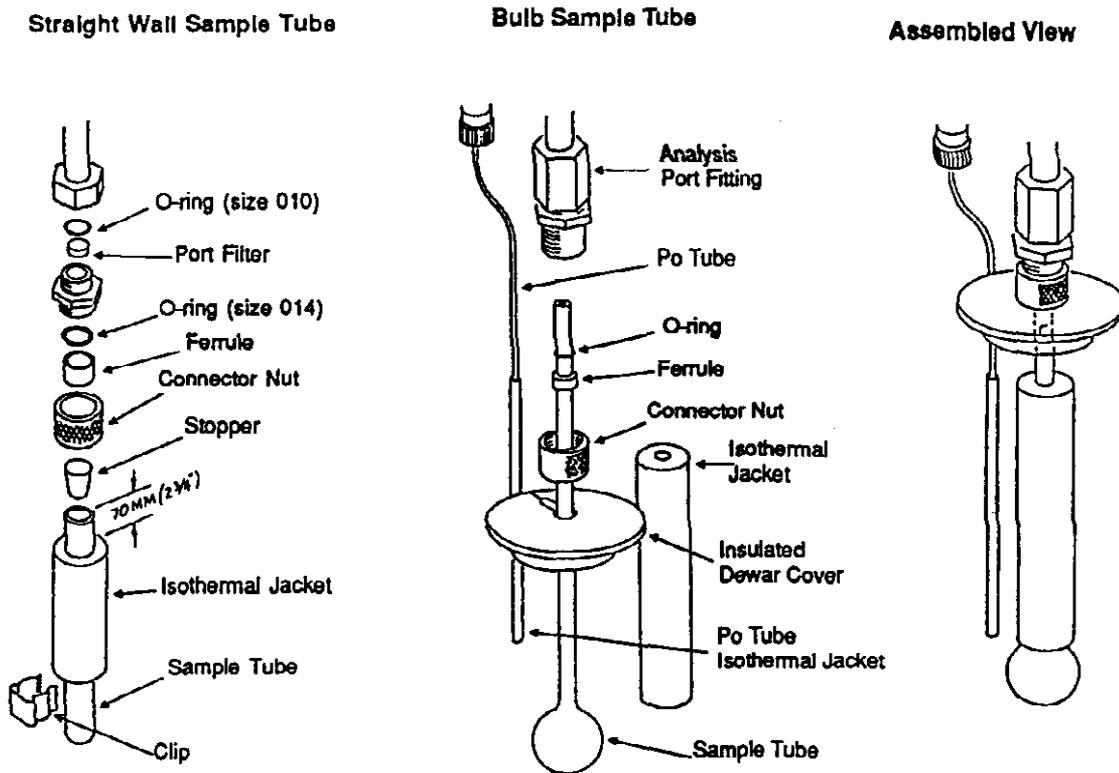


Figure 2-38. Preparing and Attaching Sample Tube to Analysis Port

## I. INSTALLING THE COLD TRAP DEWAR

### WARNING

Liquid nitrogen can cause burns. Wear safety glasses and insulating gloves when handling liquid nitrogen.

1. Fill the cold trap Dewar with liquid nitrogen to about 5 cm (2 inches) from the top.
2. Hang the cold trap Dewar around the cold trap port as shown in the following illustration.

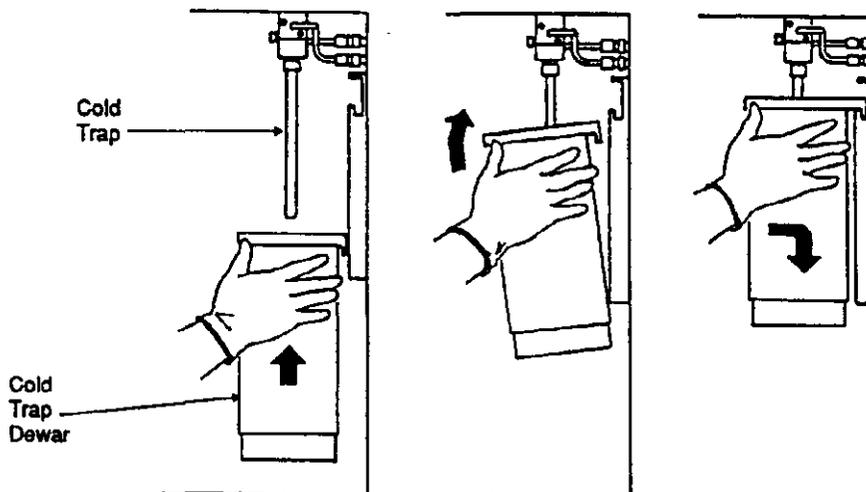


Figure 2-39. Cold Trap Dewar Installation

3. Place the insulator/stopper over the Dewar flask opening as shown below.

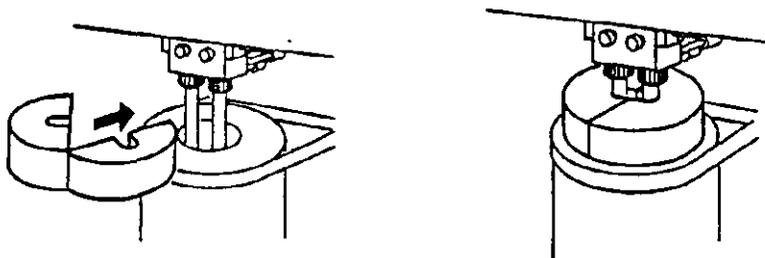
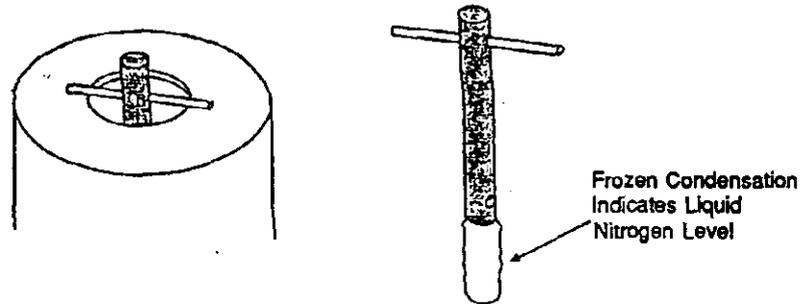


Figure 2-40. Cold Trap Dewar Insulator/Stopper

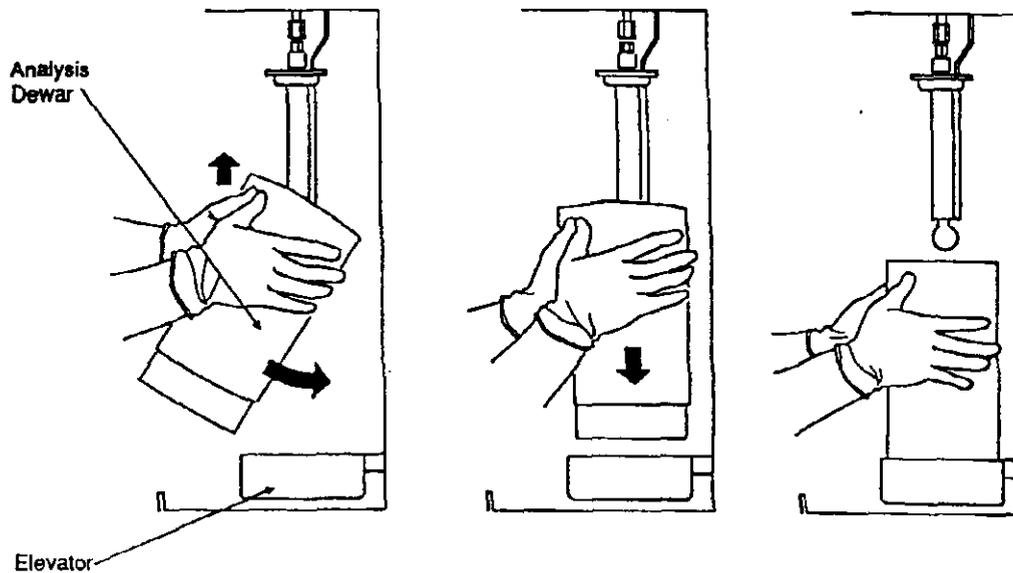
**J. INSTALLING ANALYSIS DEWAR**

1. Fill the analysis Dewar with liquid nitrogen to about 5 cm (2 inches) from the top.
2. Check the liquid nitrogen level with the dipstick as shown below. The liquid nitrogen level is indicated by the frost mark on the dipstick.



*Figure 2-41. Liquid Nitrogen Dipstick*

3. Insert the analysis Dewar onto the elevator as shown in the following illustration.



*Figure 2-42. Analysis Dewar Installation*

4. Lower the safety shield.

#### NOTE

The safety shield should remain lowered at all times except when loading a sample.

#### K. PERFORMING ANALYSIS OF SAMPLE

This procedure involves completing an analysis of the sample and providing printed results of that analysis which can be compared to information supplied with the reference material. To perform an analysis:

1. With the MAIN FUNCTION MENU displayed on the video monitor, press **F3**. The Sample Information Menu is displayed. Then press **F3** again. The Add Sample Information screen is displayed.

Observe the sample number on the screen. It should be the same number entered on the Sample Data Sheet as SAMPLE NUMBER. Press **↵**.

Enter the SAMPLE ID from the Sample Data Sheet using the alphanumeric keypad, and press **↵**.

At the SUBMITTER ID prompt, just press **↵**.

Enter the OPERATOR ID from the Sample Data Sheet, and press **↵**.

Enter the REPORT TITLE from the Sample Data Sheet, and press **↵**.

Enter the SAMPLE WEIGHT (after Degas) from the Sample Data Sheet, and press **↵**.

Leave the TYPE OF DATA set to automatically collected.

The screen should now look similar to the one shown below.

|                 |        |        |               |
|-----------------|--------|--------|---------------|
| ASAP 2000 V1.XX | UN1/N2 | UN2/N2 | DATE:01/09/89 |
| SAMPLE DIR/NUM  |        |        | TIME:08:35:10 |
| PRESSURE        | 8.00   | 8.00   | CURRENT DIR:  |
| TEMP            | 14.2   | 35.1   | DATA          |
| VOL ABSORBED    |        |        |               |
| STATUS          | IDLE   | IDLE   |               |

3.3d1 ADD SAMPLE INFORMATION

Sample number: 1

Sample ID: Reference Material

Submitter ID:

Operator ID: Brown

Report Title: Installation

Sample wt: 1.5000 g

Type of data? automatically collected

[PgDn]

*Figure 2-43. Add Sample Information Screen*

Press **[PgDn]**. The Add Sample - Run Conditions screen is displayed.

2. Press **[PgDn]**. The Add Sample - Pressures screen is displayed.

Press **[↓]**. The Number of surface area points? prompt is highlighted. Press **[5]** on the cursor control keypad three times to select a value of 5.

3. Press **[PgDn]**. The Add Sample - Report Options screen is displayed.

Press **[↓]**. The Print analysis log? prompt is highlighted. Press **[5]** on the cursor control keypad to change the value to no.

Press **[↓]** twice. The Report BJH ads? prompt is highlighted. Press **[5]** on the cursor control keypad to change the value to no.

Press **[→]**. The Report BJH des? prompt is highlighted. Press **[5]** on the cursor control keypad to change the value to no.

The values in the fields should now match those shown on the following screen.

|                 |        |        |               |
|-----------------|--------|--------|---------------|
| ASAP 2000 V1.XX | UNI/M2 | UNI/M2 | DATE:01/09/89 |
| SAMPLE DIR/NUM  |        |        | TIME:08:37:44 |
| PRESSURE        | 0.00   | 0.00   | CURRENT DIR:  |
| TEMP            | 14.1   | 15.1   | DATA1         |
| VOL ADSORBED    |        |        |               |
| STATUS          | IDLE   | IDLE   |               |

3.3p5 ADD SAMPLE - REPORT OPTIONS [PgUp]

|                            |                |                     |                |
|----------------------------|----------------|---------------------|----------------|
| Report options set number: | 0              | Report ID:          |                |
| Print analysis log?        | no             | Plot isotherms?     | yes F4 to edit |
| Report surf area?          | yes F5 to edit | Report micropore?   | no F6 to edit  |
| Report BJH ads?            | no F7 to edit  | Report BJH des?     | no F8 to edit  |
| Print 2500 BJH ads?        | no             | Print 2500 BJH des? | no             |
| Print summary?             | yes            |                     |                |

Figure 2-44. Add Sample - Report Options Screen

4. Press **[PgDn]**. The Sample Information Menu is displayed. Then press **[F2]** to display the Main Function Menu.
5. Press **[F7]**. The Start Run screen is displayed.

Observe the Unit Number indicated on the video monitor. This number should be 1. If this procedure is being used for operational verification of a second analyzer, press **[5]** on the cursor control keypad, and then press **[←]**. If this procedure is being used for operational verification of either a single analyzer or the first in a dual-analyzer system, just press **[←]**.

Enter the Sample Number (from the Sample Data Sheet) using the alphanumeric keypad.

Press **[PgDn]**. This starts the automatic analysis, which takes approximately 1 to 2 hours. The Main Function Menu is displayed.

6. Lower the safety shield.
7. Press **[F8]**. The Status/Control Menu is displayed. Then press **[F4]** to monitor the analysis. The Run Status/Control screen is displayed.

8. Remove the reports from the printer when the analysis is complete. Compare these results with the information on the Product Bulletin (Part No. 004-16821-00) supplied with the reference material.

This completes the procedure for verifying operation. If the results obtained match the referenced information within the specified limits, verification of proper system operation is complete. However, if the results do not match, refer to the Troubleshooting section of this manual. After corrective procedures in the Troubleshooting section have been performed, repeat the procedures for verifying operation. If the desired results are not obtained, service to the system or operational assistance may be required.

## MAINTENANCE AND SERVICE

### 9-1. INTRODUCTION

The ASAP 2000 system has been designed to provide efficient and continuous service. However, in order to get the best results over the longest period of time, certain maintenance and service procedures must be followed. Additionally, when operator problems are encountered, the appropriate corrective action must be taken. This section contains information regarding operator problems and corrective action, along with maintenance and service procedures.

### 9-2. TROUBLESHOOTING

Most operational problems are caused by leaks (commonly around the sample tube O-ring at the analysis port), sample weighing errors, use of too much LN<sub>2</sub> in the analysis Dewar at the start of an analysis, and entry of incorrect system volume for analysis. Always check these first when expected analysis results are not obtained. Some common operational problems, which are not indicated on the video monitor screen, and their respective causes and solutions are provided in Table 9-1.

*Table 9-1. Operational Problems Not Displayed on Monitor*

| WHAT HAPPENED   | WHY   | WHAT TO DO  |
|---|---|---|
| LN <sub>2</sub> Dewar used for analysis cannot be raised (or lowered).            | Elevator which moves Dewar stuck in up position, down position, or somewhere in between.        | Check for possible obstruction to elevator movement.  |
| Analyzer controls and indicators not operating or extraordinary values displayed. | 1. Ribbon cable which connects analyzer and control panel partially or completely disconnected. | 1. Check cable connection; correct as necessary.  |
|   | 2. Analyzer Power/Breaker switch in OFF position.   | 2. Place Power/Breaker switch in ON position. If switch returns to OFF position, refer problem to appropriate service personnel.                                  |
|   | 3. Breaker(s) placed in OFF (out) position.   | 3. Check position of both breakers on the analyzer rear panel. If breaker is out, push breaker in. If it will not stay in, contact appropriate service personnel. |

**Table 9-1. Operational Problems Not Displayed on Monitor (continued)**

| WHAT HAPPENED   | WHY   | WHAT TO DO  |
|---|---|---|
| Analyzer controls and indicators not operating or extraordinary values displayed. (continued) | 4. Electrical power to analyzer turned off.   | 4. Check power cord connections at both the analyzer and the power source. If power cord is connected properly, verify power at power source. |
|   | 5. Internal fuse blown.   | 5. Replace fuse. (See Replacing Control Panel Fuse, Section 9.)   |
| Analysis vacuum pump gurgles continuously   | 1. Sample tube O-ring or fitting loose.   | 1. Tighten fitting. Replace O-ring.   |
|   | 2. Sample tube cracked.   | 2. Replace with new sample tube or install steel plug.  |
|   | 3. No sample tube loaded on a selected port.  | 3. Install steel plug.  |
|   | 4. Gas inlet valve open while vacuum valve open.  | 4. Use manual mode control screen to close gas inlet valve.   |
| Degas temperature display shows three decimal points (...) and any number.                    | Thermocouple unplugged.   | Plug thermocouple into correct connector.   |
| Degas Error light illuminated.  | 1. O-ring seal at sample tube fitting is loose or damaged.  | 1. Tighten or inspect O-ring for damage. Replace.   |
|   | 2. Excessive sample outgassing.   | 2. Start degas again or pretreat sample in a drying oven.   |
|   | 3. Leak at filter fitting above sample connector.   | 3. Replace O-ring around filter.  |
|   | 4. Leak at internal fitting.  | 4. Tighten fitting slightly. Replace.   |
|   | 5. Previously loaded samples were evacuated slowly for 2 hours and did not reach set point on vacuum gauge. | 5. Reset vacuum gauge if necessary. Restart samples. Samples which are badly contaminated may benefit from evacuation in a vacuum oven.       |

Table 9-1. Operational Problems Not Displayed on Monitor (continued)

| WHAT HAPPENED  | WHY  | WHAT TO DO   |
|--|--|--|
| Degas Error light illuminated. (continued)   | 6. System was unable to admit backfill gas in 2 minutes to above atmospheric pressure. | 6. Check gas regulator and valves. Replace gas supply if it is exhausted. Regulator to be set at about 8 to 12 psig. |
| Vacuum gauge shows reading above 20 $\mu$ mHg, even after extended pumping through unrestricted valve with analysis or degas ports closed. | 1. Vacuum pump oil is low, causing ineffective evacuation.                             | 1. Add or change vacuum pump oil. Be sure to add oil to proper level according to sight glass.                       |
|  | 2. Filter in port being used is dirty.   | 2. Clean filter in port. (See Cleaning Analyzer Port Filter, Section 9.)   |
|  | 3. Leak in vacuum plumbing.  | 3. Check and tighten all connections in vacuum plumbing, including cold traps.                                       |
|  | 4. Vacuum pump turned off or unplugged.  | 4. Check pump power plug and power switch.   |
|  | 5. The alumina in the oil vapor trap is holding moisture.                              | 5. Replace or dry the alumina.   |
|  | 6. Cold trap obstructed by condensation.   | 6. Clean the cold trap tube. (See Cleaning Cold Trap Tubes in this chapter.)   |
| Degas Load light will not come on when LOAD button pressed.  | 1. Degas system is completing another task.  | 1. Wait for Ready light to turn on.  |
|  | 2. Degas system is in Manual Mode.   | 2. Set AUTO/MANUAL switch to AUTO position, then wait for Ready light to turn on.                                    |
| Control module display is off or only some screen text is visible.   | 1. Display has been turned off.  | 1. Turn on display.  |
|  | 2. Display has been adjusted.  | 2. Adjust brightness and contrast as necessary.  |

**Table 9-1. Operational Problems Not Displayed (continued)**

| WHAT HAPPENED  | WHY  | WHAT TO DO  |
|--|--|---|
| Control module display is off or only some screen text is visible. (continued) | 3. Power to monitor is off.  | 3. Make sure power cable is plugged into power source and monitor power switch is on. Check control module power to verify that control module is on. If control module is off, turn it on. |
| Valves cannot be operated.   | 1. Circuit opened by circuit breaker (analysis valves and degas valves). | 1. Push in breaker button. If it will not stay in, contact appropriate service personnel.   |
|  | 2. Internal fuse blown.  | 2. Replace fuse. (See Replacing Valve Control Fuse, Section 9.)   |

Some operational problems are indicated by messages on the video monitor screen. These messages are referred to as **ERROR MESSAGES**, and are provided by the system software. Information concerning these messages is provided in Appendix B.

### 9-3. PREVENTIVE MAINTENANCE

The ASAP 2000 system has been designed to provide efficient and continuous service. However, some disposable parts (e.g., filters, fuses, O-rings, plungers, etc.) are used in the system; and, the fluid in the vacuum pumps loses its effectiveness over a period of time. Therefore, in order to get the best results over a longer period of time, a conscientiously applied program of regular preventive maintenance can prove to be effective. The Preventive Maintenance Schedule and the Preventive Maintenance Checklist may serve as convenient aids to such a program.

#### A. PREVENTIVE MAINTENANCE SCHEDULE

Most equipment users are commonly assigned multiple duties and responsibilities; thus, preventive maintenance is frequently either forgotten or irregularly scheduled. The Preventive Maintenance Schedule (Table 9-2) can be used both as a convenient reminder and as a tool for maintaining schedule regularity.

**Table 9-2. Preventive Maintenance Schedule**

| Maintenance Required   | Frequency                     |
|--|-------------------------------|
| 1. Replacing analyzer port filters.  | Every 30 days                 |
| 2. Cleaning diskette drive.  | Every 30 days                 |
| 3. Cleaning analyzer.  | As required                   |
| 4. Testing analyzer for leaks.   | As required or every 6 months |
| 5. Overhauling analyzer valves.  | As required                   |
| 6. Replacing Vacuum pump exhaust filters.                                      | Annually*                     |
| 7. Inspecting and changing vacuum pump fluid.                                  | Annually*                     |
| 8. Lubricating molecular drag pump (krypton, chemi, and micropore units only). | Annually*                     |
| 9. Calibrating manifold temperature sensor.                                    | Annually                      |
| 10. Cleaning cold trap tubes.  | As required                   |
| 11. Replacing alumina in oil vapor traps (if installed).                       | As required                   |
| *Heavy use may require more frequent maintenance.                              |                               |

**B. PREVENTIVE MAINTENANCE CHECKLIST**

The following checklist can be used as a maintenance record and in determining if additional accessories must be ordered. A blank checklist is included in Appendix A for your convenience.

### PREVENTIVE MAINTENANCE CHECKLIST

This Preventive Maintenance Checklist can be used as a maintenance record (for task verification) and in determining if additional accessories must be ordered.

Instrument/System \_\_\_\_\_ Date \_\_\_\_\_

| Description of Task        | Completed | Ordering Information   |
|----------------------------|-----------|--|
| Cleaned analyzer           | —         |  |
| Overhauled degas valves    | —         | Need to order valve housing<br>O-ring, P/N: 004-25471-00<br>Yes _____ No _____<br><br>Need to order valve plunger,<br>P/N: 250-25267-00<br>Yes _____ No _____<br><br>Need to order valve spring,<br>P/N: 250-25628-00<br>Yes _____ No _____<br><br>Need to order valve seats, re-<br>stricted, P/N: 240-25897-00<br>Yes _____ No _____<br><br>Need to order valve seat, unre-<br>stricted, P/N: 240-25817-00<br>Yes _____ No _____<br><br>Need to order size 010 seat<br>O-ring, P/N: 004-25466-00<br>Yes _____ No _____ |
| Overhauled analysis valves | —         | Need to order valve plunger,<br>P/N: 250-25267-00<br>Yes _____ No _____<br><br>Need to order valve spring,<br>P/N: 250-25628-00<br>Yes _____ No _____<br><br>Need to order Kel-F gasket,<br>P/N: 250-25608-00<br>Yes _____ No _____  |

Figure 9-1. Preventive Maintenance Checklist (page 1)

### C. REPLACING ANALYZER PORT FILTER

Refer to the Preventive Maintenance Checklist in Appendix A for part numbers of components. A 2-micron filter is located in the analysis port (20-micron for krypton) and in each degas port of the analyzer. (See Figure 9-2.) If a filter on a degas port is contaminated, the contaminant may adsorb atmospheric gases when the port is not plugged (with either sample tube or steel plug), resulting in extended degassing time for samples on that port. Likewise, a contaminated filter on the analysis port may result in extended time for achieving a vacuum at that port. More importantly, the contaminant may adsorb or desorb during analysis, affecting the analysis results. A contaminated filter on the analysis port may be detected by a leak test (if the contaminant outgasses) or by a free space reading much lower than normal.

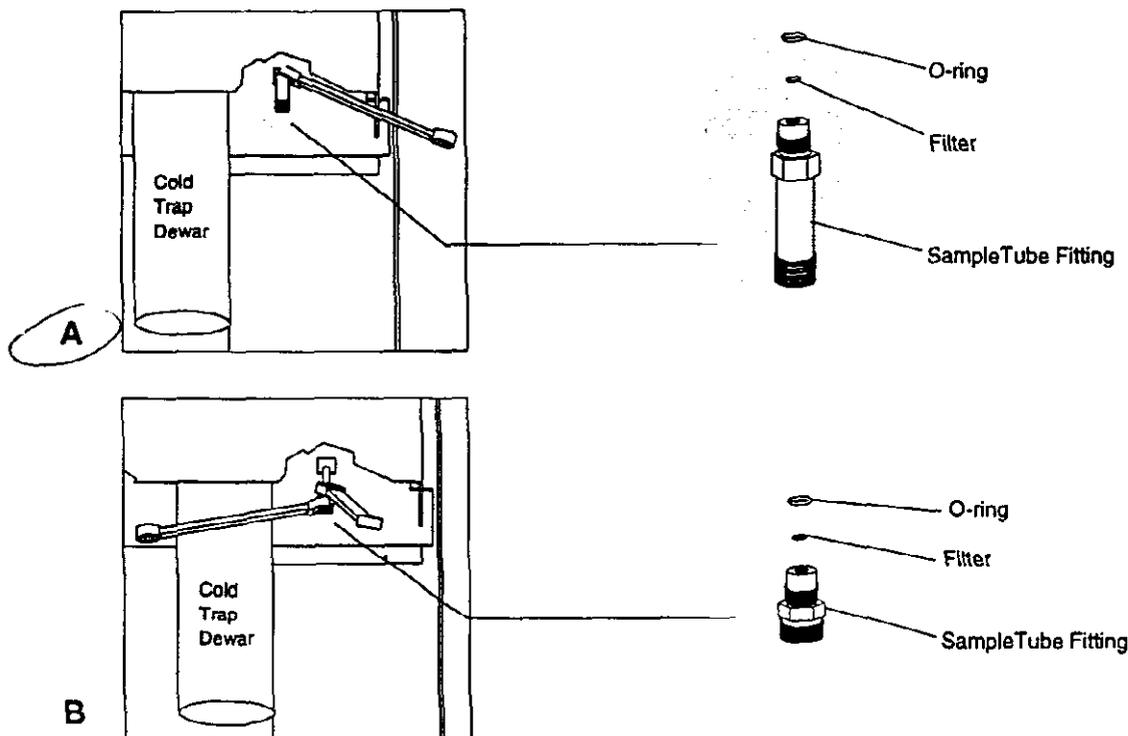


Figure 9-2. Analyzer Port Filter Location

To replace the filter:

1. Use one wrench to remove the analysis port fitting in analyzers with a serial number of 347 and above (refer to Figure 9-2A). Use two wrenches to remove the analysis port fitting in analyzers with serial numbers below 347 (refer to Figure 9-2B).

#### NOTE

To avoid degassing problems, the filter and O-ring should be clean and should not be touched with bare hands.

2. Replace the filter and the O-ring. Carefully reassemble the sample tube fitting, filter, O-ring and manifold connector, and tighten by hand. Then tighten with the two wrenches to prevent leaks when evacuated.

## E. CLEANING THE ANALYZER

### CAUTION

When cleaning the analysis compartment shield, do not use isopropyl alcohol. This could result in damage to the surface of the shield. A mild detergent in water can be used.

A clean cloth, dampened with isopropyl alcohol (IPA), a mild detergent or a 3% hydrogen peroxide solution may be used to clean the outside casing of the analyzer. It is not necessary to remove any switches or screws while cleaning.

## F. TESTING ANALYZER FOR LEAKS

This test is designed to isolate internal or external valve leaks. If the instrument passes this check and leakage is still suspected, contact appropriate service personnel.

If a leaky valve is discovered, attempt to correct the problem by tightening the housing (see Figures 9-5 and 9-6). If this is unsuccessful, replace the valve plunger and perform the test again. Contact appropriate service personnel if leak persists.

In the test procedure whenever you see the statement, *Observe the leak rate*, perform the following steps:

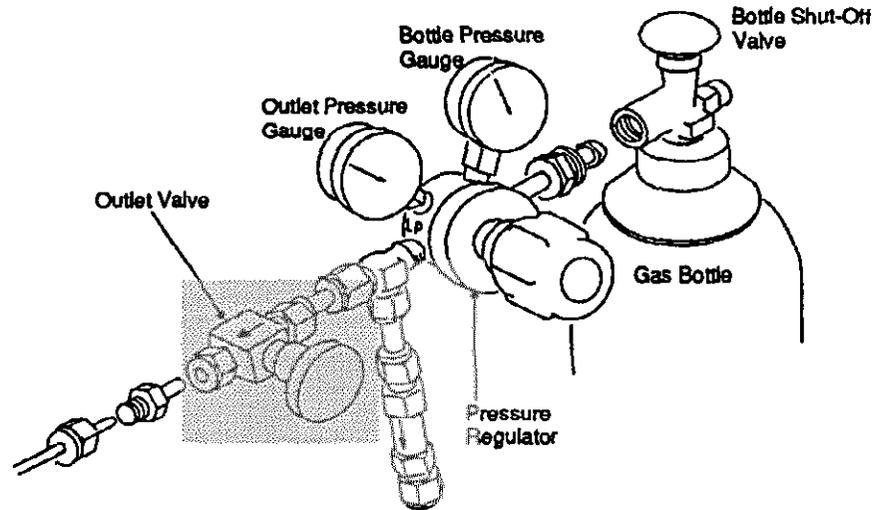
### NOTE

Between each test, it may be necessary to re-evacuate the system to remove accumulated gas.

1. Note the reading on the analysis vacuum gauge.
2. Wait 10 minutes.
3. Note the reading again on the analysis vacuum gauge.
4. Subtract the first reading from the second one.
5. Divide the result by 10 to get the leak rate in  $\mu\text{mHg}/\text{min}$ .

Perform the leak test as follows:

1. Close the outlet valves for the analyzer gas inlets (see Figure 9-3).



*Figure 9-3. Gas Outlet Valve*

2. Ensure that plug is installed in sample port, Po tube is correctly installed, and calibration volume cap is correctly installed.
3. Open valves 1 through 11.
4. Allow manifold to evacuate to approximately 15 microns.
5. Close valves 3 through 6 and valves 8 through 11.
6. Allow manifold to evacuate for 15 minutes.
7. Close valves 1 and 2.
8. Observe the leak rate. This is the base leak rate for the system. If the leak rate exceeds 2  $\mu\text{mHg}/\text{min}$ , check fittings. If the leak rate is less than 2  $\mu\text{mHg}/\text{min}$ , proceed with Step 9.
9. Close valve 7 and open valve 2.
10. Remove plug from sample port.
11. Observe the leak rate. If the leak rate exceeds 2  $\mu\text{mHg}/\text{min}$ , valve 9 is leaking. If the leak rate is less than 2  $\mu\text{mHg}/\text{min}$ , proceed with Step 12.
12. Re-install plug in sample port.

13. Open valve 8 and allow pressure to equilibrate approximately 30 seconds.
14. Observe leak rate. If the leak rate exceeds 5  $\mu\text{mHg}/\text{min}$  above the base leak rate, leak is in the area of the calibration volume chamber. If the leak rate is less than 5  $\mu\text{mHg}/\text{min}$ , proceed with Step 15.
15. Close valve 8.
16. Open valve 10 and allow pressure to equilibrate approximately 1 minute.
17. Observe the leak rate. If the leak rate exceeds 2  $\mu\text{mHg}/\text{min}$  above the base leak rate, the Po tube is leaking. If the leak rate is less than 2  $\mu\text{mHg}/\text{min}$ , proceed with Step 18.
18. Close valve 10.
19. Remove Po tube.
20. Observe the leak rate. If the leak rate exceeds 2  $\mu\text{mHg}/\text{min}$  above the base leak rate, valve 10 is leaking. If the leak rate is less than 2  $\mu\text{mHg}/\text{min}$ , proceed with Step 21.
21. Re-install Po tube.
22. Open valves 1, 7, 10 and 11.
23. Allow manifold to evacuate to approximately 15  $\mu\text{mHg}$ .
24. Close valves 1, 2, 10 and 11.
25. Open the nitrogen gas regulator outlet valve to pressurize valve 6.
26. Observe the leak rate. If the leak rate exceeds 2  $\mu\text{mHg}/\text{min}$  above the base leak rate, valve 6 is leaking. If the leak rate is less than 2  $\mu\text{mHg}/\text{min}$ , proceed with Step 27.
27. Open the analysis gas regulator outlet valve to pressurize valves 4 and 5.
28. Observe the leak rate. If the leak rate is greater than 2  $\mu\text{mHg}/\text{min}$  above the base leak rate, valve 4 or 5 is leaking. If the leak rate is less than 2  $\mu\text{mHg}/\text{min}$ , proceed with Step 29.
29. Open the helium gas regulator outlet valve to pressurize valve 3.
30. Observe the leak rate. If the leak rate is greater than 2  $\mu\text{mHg}/\text{min}$  above the base leak rate, valve 3 is leaking. If the leak rate is less than 2  $\mu\text{mHg}/\text{min}$ , proceed with Step 31.
31. Remove the Po tube.
32. Observe the leak rate. If the leak rate exceeds 2  $\mu\text{mHg}/\text{min}$  above the base leak rate, valve 11 is leaking. If the leak rate is less than 2  $\mu\text{mHg}/\text{min}$ , proceed with Step 33.
33. Re-install the Po tube.

34. Open valves 1 and 2.
35. Allow manifold to evacuate to approximately 15  $\mu\text{m}$ .
36. Close valves 1, 2, and 7.
37. Open valve 3 for approximately 5 seconds.
38. Observe the leak rate. If leak rate exceeds 2  $\mu\text{mHg}/\text{min}$  above the base leak rate, valve 7 is leaking. If leak rate is less than 2  $\mu\text{mHg}/\text{min}$ , proceed with Step 39.
39. Open valves 3, 7, and 8.
40. Pressurize manifold to approximately 800 mmHg.
41. Close valves 3 and 8.
42. Open valves 1 and 2.
43. Allow manifold to evacuate to approximately 15  $\mu\text{mHg}$ .
44. Close valves 1, 2, and 7.
45. Observe the leak rate. If the leak rate exceeds 2  $\mu\text{mHg}/\text{min}$  above the base leak rate, valve 8 is leaking. If the leak rate is less than 2  $\mu\text{mHg}/\text{min}$ , the system passes the leak check

## G. OVERHAULING ANALYZER VALVES

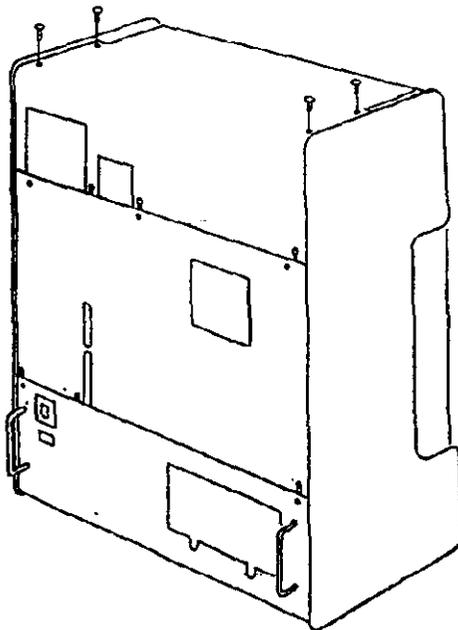
Refer to the Preventive Maintenance Checklist in Appendix A for part numbers of components. Two types of valves are used in the ASAP 2000 analyzer. One type is used on the analysis manifold, and the other type is used on the degas manifold. The only difference between the valves is the types of coils and housing used. Occasionally, due to wear, some internal valve components must be replaced for the valve to operate properly. The procedure for repairing the valves is as follows:

### Degas Valves

1. Remove the three screws which fasten the top panel at the upper rear of the analyzer. (See Figure 9-4.) Then remove the four screws (two on each side) at the top of the analyzer. Lift the top panel from the analyzer.
2. Locate the valve to be repaired on the degas manifold.
3. Remove the nut (and washer if included) at the top of the valve. Lift the coil housing from the valve.

### CAUTION

When removing the coil housing from the valve, avoid pulling or twisting the attached wires.



*Figure 9-4. Removing Analyzer Top Panel*

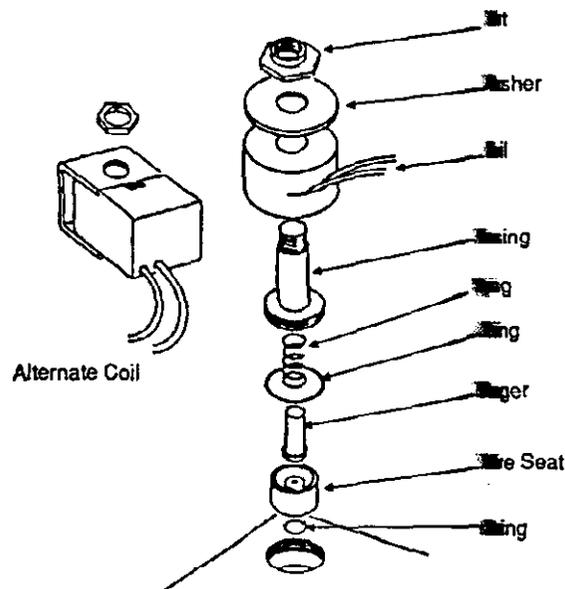


Figure 9-5. Repairing Analyzer Valves (~~D~~Manifold)

4. Place the spanner wrench supplied with the system over ~~the~~ valve housing such that the pins of the wrench fit into the two slots in the valve housing. ~~Remove~~ the valve housing by turning counterclockwise.
5. Examine the condition of the valve components. Most of ~~the~~ problems associated with valve failure can be attributed to the spring and plunger. Occasionally, the seat is damaged. Replace the worn or damaged components.
6. Reassemble the valve as follows:
  - a. Center the O-ring (size 010) inside the opening in ~~the~~ manifold.
  - b. Place the seat in the manifold.
  - c. Place the O-ring (size 018) above and around the ~~seat~~.
  - d. Place the plunger inside the seat.
  - e. Place the spring over the plunger.
  - f. Place the valve housing over the spring and plunger, ~~and~~ screw the housing into the manifold. Tighten with the spanner wrench.

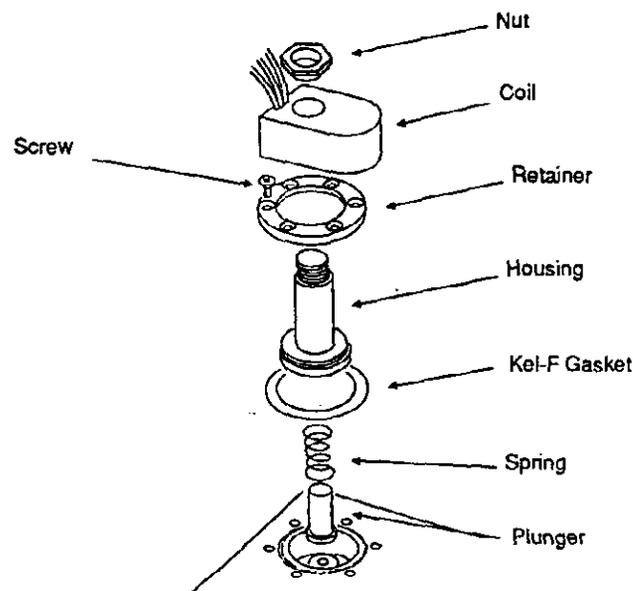
7. Place the coil housing over the valve housing. Install nut (and washer if included). Hand-tighten the nut (it is not necessary to use a wrench).
8. Replace the top panel (refer to Figure 9-4).

### Analysis Valves

1. Remove the three screws which fasten the top panel at the upper rear of the analyzer. (See Figure 9-4.) Then remove the four screws (two on each side) at the top of the analyzer. Lift the top panel from the analyzer.
2. Locate the valve to be repaired on the analysis manifold.
3. Remove the nut at the top of the valve. Lift the coil housing from the valve.

#### CAUTION

When removing the coil housing from the valve, avoid pulling or twisting the attached wires.



*Figure 9-6. Repairing Analyzer Valves (Analysis Manifold)*

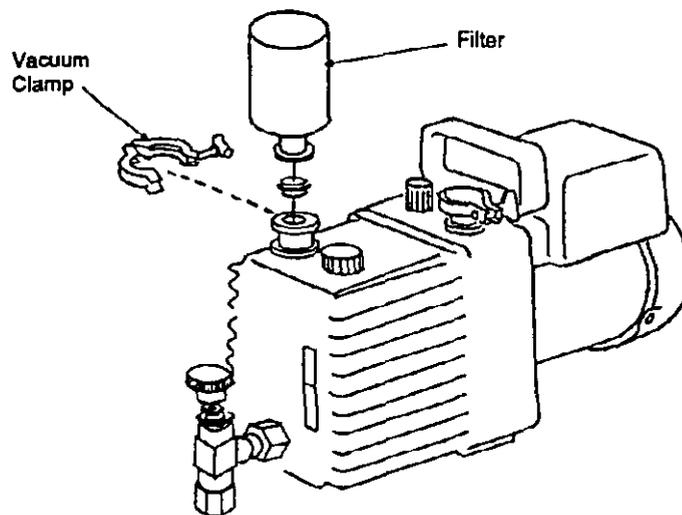
4. Remove the 6 screws holding the retainer. Lift the retainer and housing away from the manifold. Discard the Kel-F gasket.
5. Examine the condition of the valve components. Most of the problems associated with valve failure can be attributed to the spring and plunger. Occasionally, the seat is damaged. Replace the worn or damaged components.

6. Reassemble the valve as follows:
  - a. Place a new Kel-F gasket on the manifold.
  - b. Place the plunger in the manifold.
  - c. Place the spring over the plunger.
  - d. Place the valve housing onto the manifold, taking care to center the Kel-F gasket.
  - e. Place the retainer around the housing and tighten the 6 screws evenly.
7. Place the coil housing over the valve housing. Install nut. Hand tighten the nut (it is not necessary to use a wrench).
8. Replace the top panel (refer to Figure 9-4).

## H. REPLACING VACUUM PUMP EXHAUST FILTER

Refer to the Preventive Maintenance Checklist in Appendix A for the part number of the filter. The gases used by the ASAP 2000 are exhausted by the vacuum pumps. Since no exhaust line is connected, an exhaust filter is used on the exhaust port of each vacuum pump. The filter minimizes the release of oil vapor. To replace the filter:

1. Loosen (but do not remove) the screws along the lower edge of the analyzer rear panel. Then remove the screws along the upper edge of the rear panel. Remove the panel by pulling the upper edge away from the analyzer then upward.
2. Loosen the wing nut of the vacuum clamp at the vacuum pump exhaust port. Swing the clamp fastening screw away from the exhaust port, and open the clamp to remove it from the port.



*Figure 9-7. Replacing Vacuum Pump Exhaust Filter*

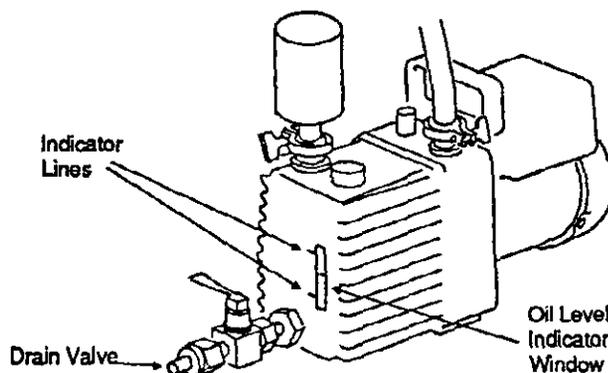
3. Lift the used exhaust filter from the exhaust port and discard it. Install a new filter on the exhaust port and push the filter against the O-ring.
4. Open the clamp and place it around the flange of the exhaust port and the flange of the exhaust filter. Swing the clamp fastening screw toward the exhaust port until it fits into the slot in the other half of the clamp. Tighten the wing nut.
5. Mount the analyzer rear panel so that the slots along the lower edge of the panel are aligned with the fastening screws on the analyzer. Insert the screws along the upper edge of the rear panel. Tighten the screws along the lower edge of the panel.

## I. INSPECTING AND CHANGING VACUUM PUMP FLUID

Refer to the Preventive Maintenance Checklist in Appendix A for part numbers of components and fluid. Annually or when the efficiency of the vacuum pump declines (requiring more time to reach vacuum levels), the fluid in the vacuum pump should be changed. The fluid can be inspected to determine if a change is necessary.

### Inspecting Fluid:

1. Rotate the analyzer so that access to the rear panel is convenient.
2. With the vacuum pump operating, open the drain valve shown in Figure 9-8 and allow a small amount of fluid to drain into a container.
3. Inspect the fluid in the container. Fluid which is in good condition is clean, honey-colored, and transparent. If the color of the fluid is darkened, the fluid should be changed. Compare it to fresh fluid.



*Figure 9-8. Vacuum Pump Drain Valve*

**Changing Fluid:**

When it has been determined that the vacuum pump fluid should be changed, the fluid should be changed as follows:

**NOTE**

**Always drain the vacuum pump while the pump is warm and switched off.**

1. Place the analyzer ON/OFF switch in the OFF position.
2. Loosen (but do not remove) the screws along the lower edge of the rear panel. Then remove the screws along the upper edge of the rear panel. Remove the panel by pulling the upper edge away from the analyzer then upward.
3. With the vacuum pump warm but not operating, remove the oil-fill plug, and open the drain valve. Allow the fluid to drain into a suitable container.
4. When the flow of fluid slows, close the drain valve.

**CAUTION**

**Adding fluid above the midway position on the fluid level indicator may cause fluid to splash into the vacuum hoses and also leak from the internal vacuum pump.**

5. Partially fill the pump with fresh fluid while observing the oil level indicator. The correct level is midway between the two indicator lines. Check the washer or O-ring used at the oil-filling port; replace if necessary. Replace the oil-fill plug.
6. Mount the analyzer rear panel so that the slots along the lower edge of the panel are aligned with the fastening screws on the analyzer. Insert the screws along the upper edge of the rear panel. Tighten the screws along the lower edge of the panel.
7. Place the analyzer ON/OFF switch in the ON position. A few hours (or overnight) will be required for air and moisture to be eliminated from the fresh fluid and for efficient vacuum operations.

**NOTE**

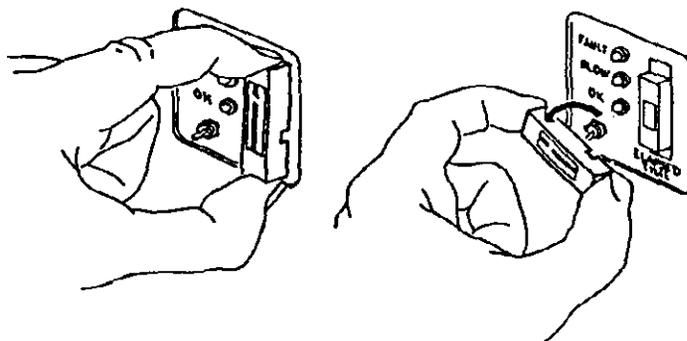
**Badly contaminated fluid will require flushing of the pump one or two times with fresh fluid in order to wash out the contamination.**

**J. LUBRICATING MOLECULAR DRAG PUMP (Krypton Unit Only)**

The molecular drag pump should be lubricated after 5000 hours (7 months) of continuous operation at 25°C. This will depend on whether the pump is turned off when not required. A syringe filled with lubricant is provided with the instrument accessories. Follow the instructions provided with the pump.

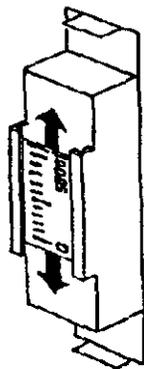
The timer, located on the MOLECULAR DRAG PUMP switch, contains an indicator that moves up the scale showing hours of use. When it reaches 5000 hours, lubricate the pump and reset the timer as follows:

1. Remove the cover and reverse it. This places the indicator downscale again.



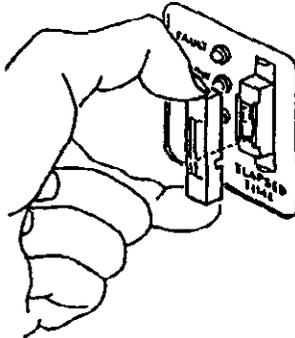
*Figure 9-9. Removing Cover from Pump Timer*

2. If necessary, slide the scale up or down so that the indicator aligns with the zero on the scale.



*Figure 9-10. Resetting the Timer Scale*

3. Snap the cover back into place.



*Figure 9-11. Replacing Pump Timer Cover*

#### **K. CALIBRATING MANIFOLD TEMPERATURE SENSOR**

Refer to Calibrating Manifold Temperature Sensor in Chapter 2 for instructions.

#### **L. CLEANING COLD TRAP TUBES**

Oil vapor from the vacuum pumps will accumulate in the cold trap. Clean the tubes as follows:

1. Turn off the instrument.
2. Remove the vent thumbscrews, then the glass tubes.
3. Rinse each tube with acetone and dry.
4. Reinstall the vent thumbscrews and glass tubes.
5. Turn on the instrument.

**M. REPLACING THE ALUMINA IN THE OIL VAPOR TRAPS (if Installed)**

The activated alumina used in the oil vapor traps becomes saturated during use. The alumina should be replaced if any of the following conditions occur:

- The alumina has been used for a 3-month period
- Oil has accumulated in the cold trap
- Most of the alumina pellets are no longer white

**NOTE**

When you replace the alumina, you should also change the vacuum pump oil.

To replace the alumina:

1. Place the analyzer ON/OFF switch in the OFF position.
2. Loosen (but do not remove) the screws along the lower edge of the rear panel. Then remove the screws along the upper edge of the rear panel. Remove the panel by pulling the upper edge away from the analyzer then upward.
3. Prepare fresh alumina as described in **Preparing Oil Vapor Traps** in Chapter 2.
4. Remove the vent thumbscrews on the cold traps to allow air into the vacuum hoses.
5. Remove the traps by opening both vacuum clamps and separating the trap from the vacuum pump and hose connections.
6. Remove one end fitting from each trap body.
7. Remove the used alumina from the trap body.

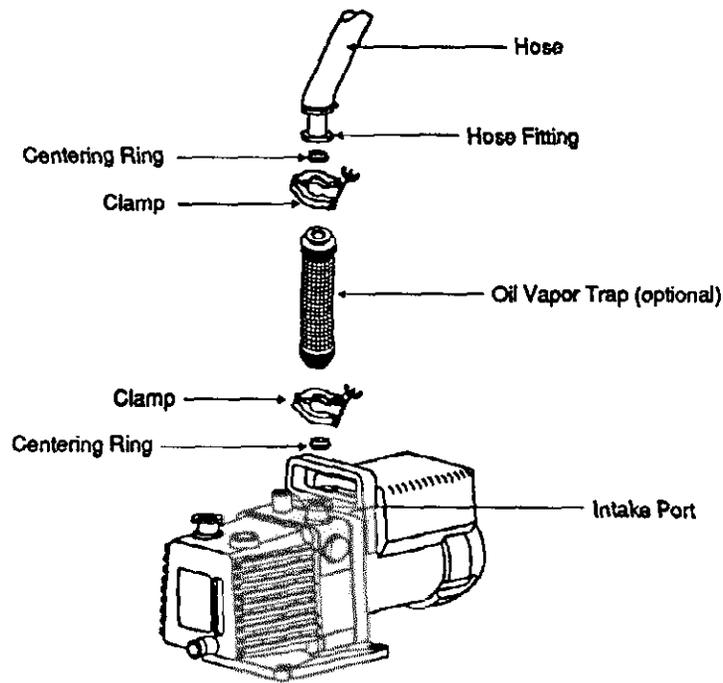


Figure 9-12. Removing the Oil Vapor Trap

- 8. If the trap body interior appears oily or dirty, it should be washed with isopropyl alcohol or ethyl alcohol and thoroughly dried.

**CAUTION**  
 Ensure adequate ventilation when using solvents for cleaning purposes.

- 9. Pour the new activated alumina pellets into each trap until they are level with the top of the trap body.

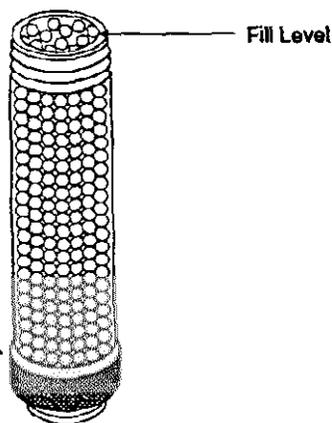


Figure 9-13. Adding Activated Alumina to Trap

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10. Screw the end fittings back onto the trap bodies.
11. Replace the traps on the vacuum pumps.
12. Reinstall the cold trap vent thumbscrews and the rear panel.
13. Place the analyzer ON/OFF switch in the ON position.

## 9-4. REPLACING FUSES

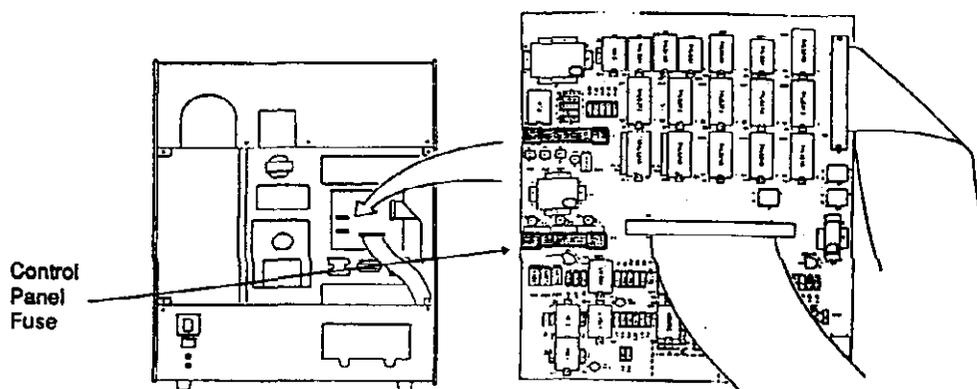
### A. REPLACING CONTROL PANEL FUSE

#### WARNING

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

A type 3AG, 2 Amp fuse is used to protect the control panel circuitry. If this fuse must be replaced, it must be replaced with a fuse of the same type and rating. To replace the fuse:

1. Loosen (but do not remove) the screws along the lower edge of the analyzer rear panel. Then remove the screws along the upper edge of the rear panel. Remove the panel by pulling the upper edge away from the analyzer then upward.
2. Locate the control panel fuse on the degas control board. Remove the blown fuse and insert a new one.



*Figure 9-14. Location of Control Panel Fuse*

3. Mount the analyzer rear panel so that the slots along the lower edge of the panel are aligned with the fastening screws on the analyzer. Insert the screws along the upper edge of the rear panel. Tighten the screws along the lower edge of the panel.