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COULTER SA 3100 Series Surface Area and Pore Size Analyzers

Product Manual



PN 4237215AA (October 2011)

Beckman Coulter, Inc.
250 S. Kraemer Blvd.
Brea, California 92821

BECKMAN COULTER

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REVISION STATUS

Initial Issue, 8/96

Analyzer Software version 2.11.
SA-VIEW Software version 1.01.

Issue AA, 10/2011

Updated trademark information
Updated all instances of Coulter Corporation or Coulter Electronics to Beckman Coulter, Inc.
Revised the address to the Beckman Coulter address
Deleted the Coulter Care Symbol (CC)

This document applies to the latest software listed and higher versions. When a subsequent software version changes the information in this document, a new issue will be released.

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1.1 GENERAL

This manual contains all necessary information and instructions for installing, operating, and performing routine maintenance on the COULTER SA 3100 and SA 3100 Plus surface area and pore size analyzers. Features available only on the SA 3100 Plus analyzer are identified as necessary, but unless specified otherwise, both models are referred to as COULTER SA 3100 analyzers. Each chapter, and each section within a chapter, begins with general information and moves on to step-by-step procedures where appropriate.

1.2 CONVENTIONS USED IN THIS MANUAL

Certain conventions are used in the text of the manual when discussing the touch-screen interface that is used to control the instrument. These conventions are as follows:

- **Screens**. Titles of the main screens (**Analysis** and **Outgas**) and titled subscreens, such as the **Profiles** screen are in Helvetica bold.
- Touch-screen Keys Key graphics are used to denote keys on the touch screen.
- *User messages*. Messages prompting you to perform an action or notifying the you of the instrument status (which appear in the center of the **Analysis** screen) are in italics.

1.3 CONTENT OF THE MANUAL

Chapter 1

This introduction.

Chapter 2

Chapter 2 covers Precautions and Hazards. Handling the cryogenic liquids and compressed gases required for the SA 3100 analyzer presents certain hazards, and proper precautions must be taken when handling these substances. If you are not an experienced user of cryogenic liquids and compressed gases, we strongly recommend that you read Chapter 2 first.

Chapter 3

Installation instructions are presented in Chapter 3. A Beckman Coulter representative will install your instrument after delivery. If you are installing the SA 3100 analyzer yourself because you have stored it or moved it, you must read Chapter 3 before attempting to set up or operate the instrument.

Chapter 4

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Chapter 4, Introducing the SA 3100 Analyzer, describes the instrument and its function. Basic concepts, including the structure of the touch-screen user interface, are discussed in this chapter. Reading this brief overview will make learning how to use the instrument much easier. Chapter 4 also lists those features of the SA 3100 analyzers which can be accessed using the 'COULTER SA 3100 Advanced Configuration Kit'.

Chapter 5

One of the accessories provided with your SA 3100 analyzer is a COULTER control sample for surface area. Chapter 5, Getting Started - Instrument Verification, will lead you through the surface area analysis of this sample to verify that your instrument is operating correctly. The chapter also demonstrates the simplicity of the SA 3100 analyzer.

Chapter 6

Chapter 6, Principles of Operation, provides a brief overview of gas sorption measurements and the measurement methods used in the SA 3100 analyzer.

Chapters 7 and 8

Chapters 7 and 8 offer detailed operating and cleaning/routine maintenance instructions. These chapters are your long-term reference to the use of the SA 3100 analyzer.

Chapter 9

Chapter 9 covers instrument specifications.

Appendix A

Appendix A covers COULTER SA-VIEW software. The SA-VIEW program is a 32-bit Microsoft Windows -based program provided with your SA 3100 analyzer for reviewing, comparing, and printing SA 3100 analyzer data. SA-VIEW software also allows you to copy SA 3100 analyzer data to other Windows applications.

Appendix B

Appendix B provides some guidelines for sample preparation and outgassing.

References

A bibliography of references used in this manual.

Index

An Index is provided to facilitate location of items in the manual.

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2.1 GENERAL

Operating the SA 3100 analyzer involves the use of compressed gases and cryogenic liquids. Cryogenic liquids and compressed gases pose many hazards if handled improperly. You should become familiar with the use of the standard containers and connectors used in your country, as well as standard laboratory safety practices for the use of these materials.

In the USA, the Compressed Gas Association (CGA), Inc. publishes several pamphlets covering the safe use of compressed gases and cryogenic liquids. Of these, the following are most relevant to the use of compressed gases and cryogenic liquids with the SA 3100 analyzer:

- CGA P-1, *Safe Handling of Compressed Gases in Containers*
- CGA P-9, *The Inert Gases - Argon, Nitrogen and Helium*
- CGA P-12, *Safe Handling of Cryogenic Liquids*

These publications, or the equivalent in your country, should be studied by all laboratory personnel. In addition, the CGA publishes the *Handbook of Compressed Gases*[i], which is an excellent general reference on the use of compressed gases and cryogenic liquids. The pamphlets, however, are generally more up-to-date and therefore take precedence over the *Handbook of Compressed Gases*.

As most users will restrict their cryogen use to nitrogen, this chapter serves only as a quick reference to the hazards posed by liquid nitrogen and compressed inert gases, and the precautions to be taken in their use. It is not a substitute for general training in the use of these materials, and makes no reference to the use of less common cryogens and adsorbate gases.

2.2 LIQUID NITROGEN

Cryogenic Liquids

Liquid nitrogen is a cryogenic liquid. Cryogenic liquids are gases that have been transformed into extremely cold refrigerated liquids which are stored at temperatures below -90° Celsius.[ii] Liquid nitrogen is stored at approximately -196°C, and is used in the SA 3100 analyzer to control the temperature of the sample and saturation tube.

Hazards

The potential hazards which accompany liquid nitrogen are:

1. Extreme cold, which can cause burns on contact with skin or eyes and embrittle materials such as carbon steel, rubber, and plastics.

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2. Extreme pressure, which can result from rapid vaporization of refrigerated liquid. This can happen if the storage container has thermal leaks or if contained liquid is suddenly exposed to high temperature.
3. Asphyxiation, due to escaping liquid and the displacement of air by the resulting rapidly expanding gas.

4. Explosion due to condensation of oxygen within open dewars of liquid nitrogen, and subsequent exposure to an open flame or spark.

Precautions

Observe the following precautions when handling liquid nitrogen:

1. Wear eye protection, cryogen-proof gloves, and closed shoes at all times when handling liquid nitrogen. Avoid contact with skin and eyes.

2. Use only in well-ventilated areas.

3. Do not smoke or use open flames in the vicinity of liquid nitrogen.

4. Do not transfer liquid nitrogen into uninsulated vessels. Use vacuum dewars only. These dewars must be tempered before first use by swirling a small quantity of liquid nitrogen around in the vessel to avoid thermal shock and implosion.

5. Even after tempering, a vacuum dewar containing liquid nitrogen may implode at any time - always wear safety glasses in the vicinity of liquid nitrogen dewars.

6. Do not pour liquid nitrogen into sinks or onto wet surfaces.

2.3 COMPRESSED NITROGEN AND OTHER INERT GASES

Compressed Gas

Compressed gases are stored in containers at high pressure and must be handled with extreme care.

Hazards

Compressed nitrogen presents these potential hazards:

1. A gas cylinder with a broken valve head can become a missile capable of penetrating walls due to rapidly escaping gas.

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2. Escaping compressed gas (even without cylinder damage) can accelerate dust or other particles to velocities capable of injuring eyes or penetrating skin.

Precautions

Observe the following precautions when handling compressed inert gases:

1. Store and secure compressed gas cylinders in an upright position. That is, strap them to a heavy laboratory bench or wall using standard gas cylinder securing equipment.

2. Do not use gas cylinders unless they are legibly labeled according to local regulation. Any cylinder without correct markings, or with illegible markings, must not be used and must be returned to the vendor.

3. Make sure storage rooms are well-ventilated.

4. Do not store cylinders at ambient temperatures above 50°C, or expose them to sunlight for prolonged periods of time.

5. If removable caps are provided for valve protection, make sure the caps remain on the containers, except when they are in use and connected to gas pressure regulators.

6. Do not drop, slide, or roll gas cylinders, or allow them to come into contact with sharp objects.

7. Do not expose cylinders to fire or flame from welding torches or other high temperature devices.

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3.1 GENERAL

Do not unpack the unit yourself. Schedule an installation time with your Beckman Coulter representative. Your COULTER SA 3100 analyzer will be installed by the Beckman Coulter representative after delivery to your site. You should, however, inspect the cartons for possible shipping damage, see Unpacking, below. Refer to the Special Requirements below to determine an appropriate location for the SA 3100 analyzer and to ensure that your laboratory is equipped for installation and operation of the instrument. If, for any reason, you need to re-install the unit at a later time yourself, complete installation instructions are provided in this chapter.

3.2 SPECIAL REQUIREMENTS

Consider these special requirements before choosing the location best suited for your instrument.

Environment

Install the instrument in a clean, smoke-free environment. Avoid areas with rapid temperature fluctuations if possible. See also Chapter 9, Specifications.

The SA 3100 system requires a sturdy laboratory bench or table at least 61 cm x 61 cm (24 in. x 24 in). The instrument must be placed at the front of the table or bench so that dewars can be conveniently placed in the unit.

Gas cylinders should be placed as close to the instrument as possible, using securing straps on a nearby wall or heavy laboratory bench.

Electrical

The SA 3100 analyzer needs a supply voltage of 100, 120, 220, or 240 V at 50/60 Hz. Beckman Coulter recommends a surge protector to ensure proper operation in the presence of electrical transients and noise. One outlet is required for the instrument. An additional outlet is necessary if you are using a printer.

Gases

Cylinders of high-purity 99.99% adsorbate gas (usually N_2) and helium are required. Gas cylinder pressure must be at least 100 psig. The pressure supplied to the SA 3100 analyzer must be 12 psig to 15 psig.

Hardware

Two-stage gas regulators with 30 psig second stages and CGA-580 regulator-to-cylinder couplings (standard in the USA - check the equivalent in your country) are required to interface gas cylinders to the instrument. An optional gas regulator

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kit is available from Beckman Coulter. Contact your Beckman Coulter Representative for details.

A leak-free connection must be made from the gas pressure regulators to 1/8 in. nylon tubing connected to the SA 3100 analyzer.

3.3 UNPACKING

Do not unpack the unit yourself. Schedule an installation time with your Beckman Coulter representative.

Your SA 3100 system is shipped in three boxes. The largest box, labeled 'SA 3100, 1 of 3', contains the instrument and the packing list. The box labeled 'SA 3100, 2 of 3' contains the vacuum pump, and the one labeled 'SA 3100 3 of 3' contains the accessory kit, glassware, and control sample.

Your instrument is tested rigorously before it is shipped. International symbols and special handling instructions tell the carrier how to treat this electronic instrument. Carefully inspect all cartons when they arrive. If you see any sign of mishandling or damage, file a claim with the carrier and contact your Beckman Coulter representative immediately. If you have insured the instrument separately, file a claim with your insurance company.

3.4 HARDWARE SETUP

General

These instructions are provided in case you need to install the unit yourself in a different location from the original installation by a Beckman Coulter representative, or because you have stored the unit.

Setting up your COULTER SA 3100 analyzer takes about 1/2 hour if you have gas cylinders already equipped with gas pressure regulators, or about 1 hour if you need to install a regulator kit.

Removing Shipping Insert

Place the instrument on the laboratory bench. The back of the unit must be accessible as you will need to reach into the vacuum pump shelf. The shipping insert, Figure 3.1, reinforces the instrument for shipping, but you must remove it before operating the unit.

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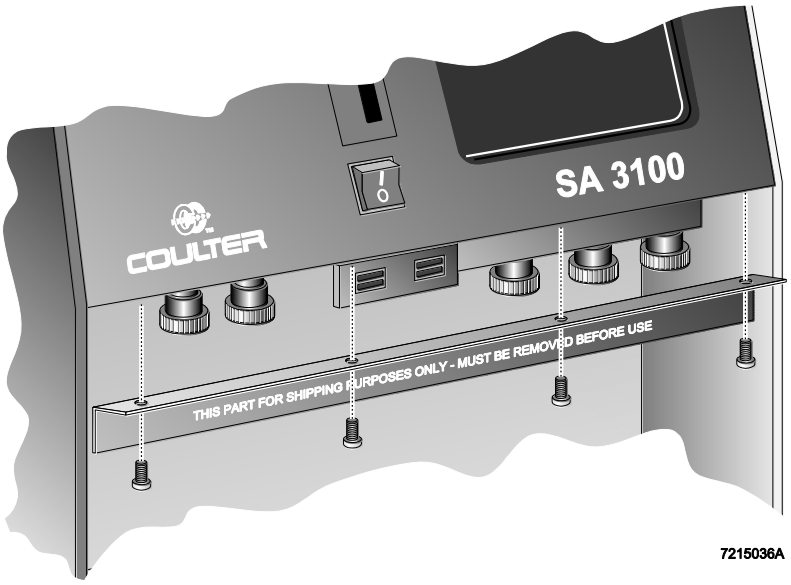


Figure 3.1 Shipping Insert

Required Item

Phillips-head screwdriver

Procedure

See Figure 3.1. Unscrew the four screws that hold in the insert and remove it. Store the insert and screws with the packing materials.

Vacuum Pump Installation

Required Items

- Vacuum pump
- Vacuum pump oil
- High vacuum silicone grease
- Vacuum pump clamps
- Oil mist filter
- 1-inch adjustable wrench

Procedure

To prepare and install the pump:

1. Add pump oil, see Figure 3.2.

    a. Ensure that the oil drain plug is tight. The plug should be hand-tight - do not use a wrench.

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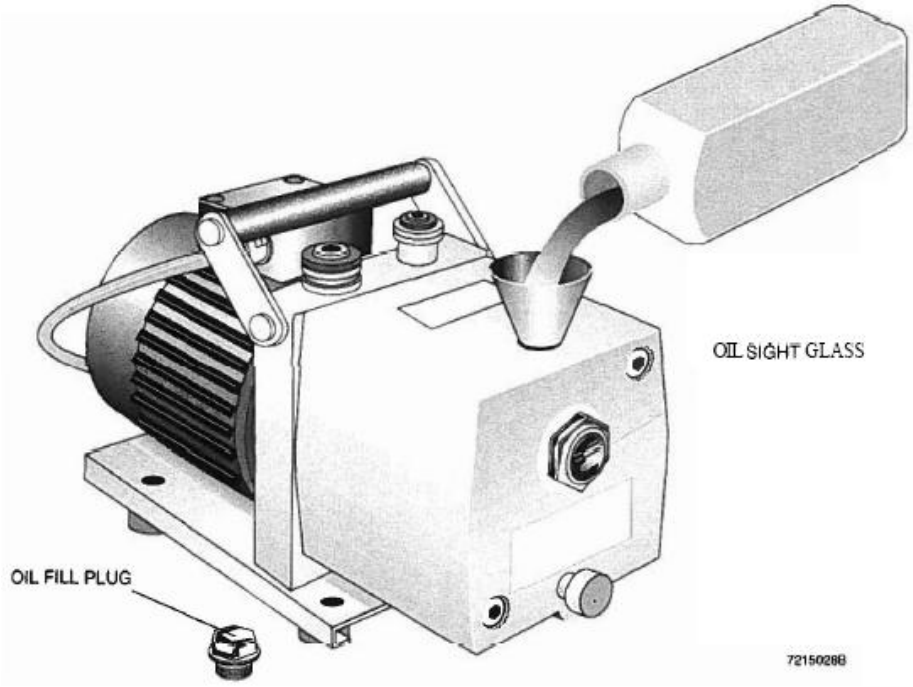


Figure 3.2 Adding Vacuum Pump **Oil**

CAUTION

Use only Balzers P3 pump oil in the vacuum pump. Use of other oil may damage the pump and will void the pump warranty.

b. Remove the oil fill plug and add pump oil SLOWLY until the sight glass is 3/4 filled.

c. Replace the oil fill plug and tighten the plug using the adjustable wrench.

2. Install the oil mist filter. See Figure 3.3.

a. Remove the shipping cover from the oil mist filter connector (a red arrow points away from the oil mist filter connector).

b. Clamp the filter onto the oil mist filter connector.

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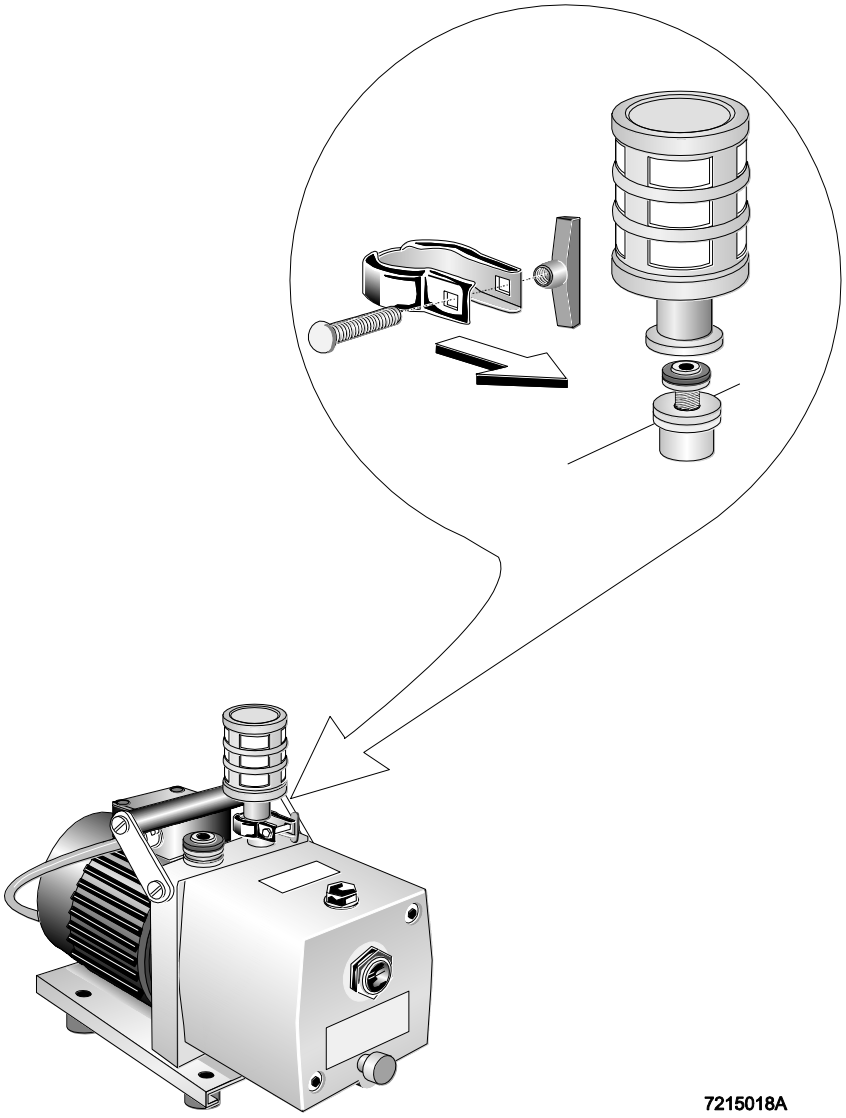


Figure 3.3 Installing Oil Mist Filter

CAUTION

Do not attach the vacuum line to the oil mist filter connector. Doing so may damage the pump and the SA 3100 analyzer.

3. Connect the vacuum line. See Figure 3.4.

   a. Remove the shipping cover from the vacuum line input (a red arrow on the pump chassis points toward the vacuum line input).

   b. Lightly grease the O-ring attached to the mesh basket with silicone high vacuum grease.

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c. Clamp the vacuum line onto the vacuum line input.

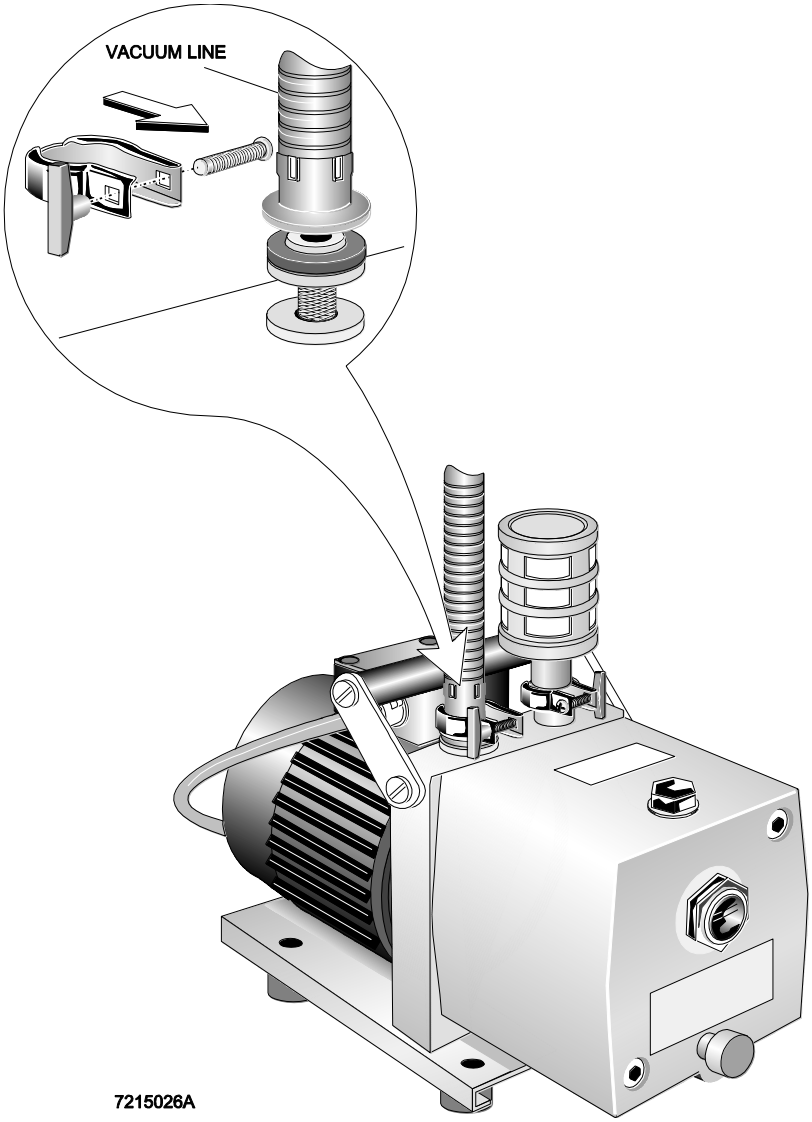


Figure 3.4 Connecting Vacuum Line to the Vacuum Pump

The vacuum pump is now ready for installation into the instrument. See Figure 3.5, illustrating a correctly prepared vacuum pump and make sure that your pump is identically fitted with oil mist filter and vacuum line.

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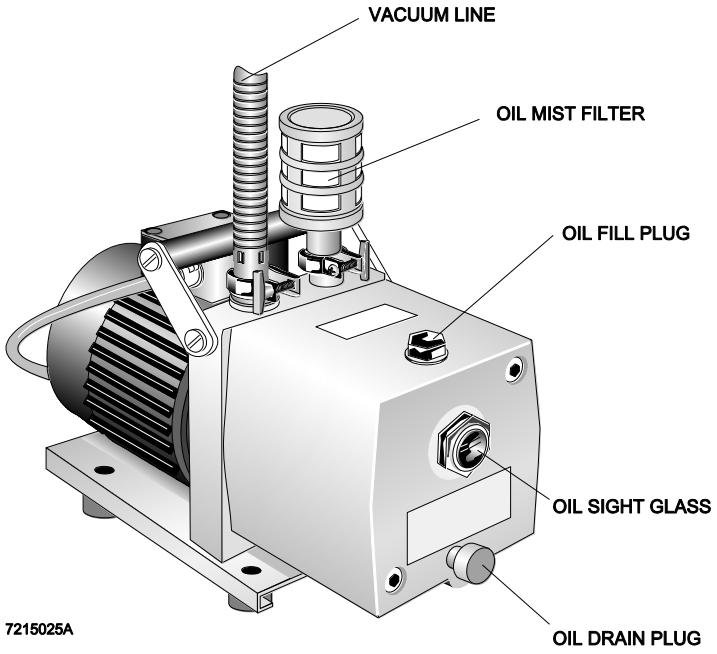


Figure 3.5 Vacuum Pump Prepared for Installation

4. See Figure 3.6. Install the vacuum pump into the SA 3100 analyzer's pump shelf:

   a. Plug the pump power cable into the pump power outlet.

   b. Place the pump into the pump shelf so that the rubber feet fit into the provided depressions.

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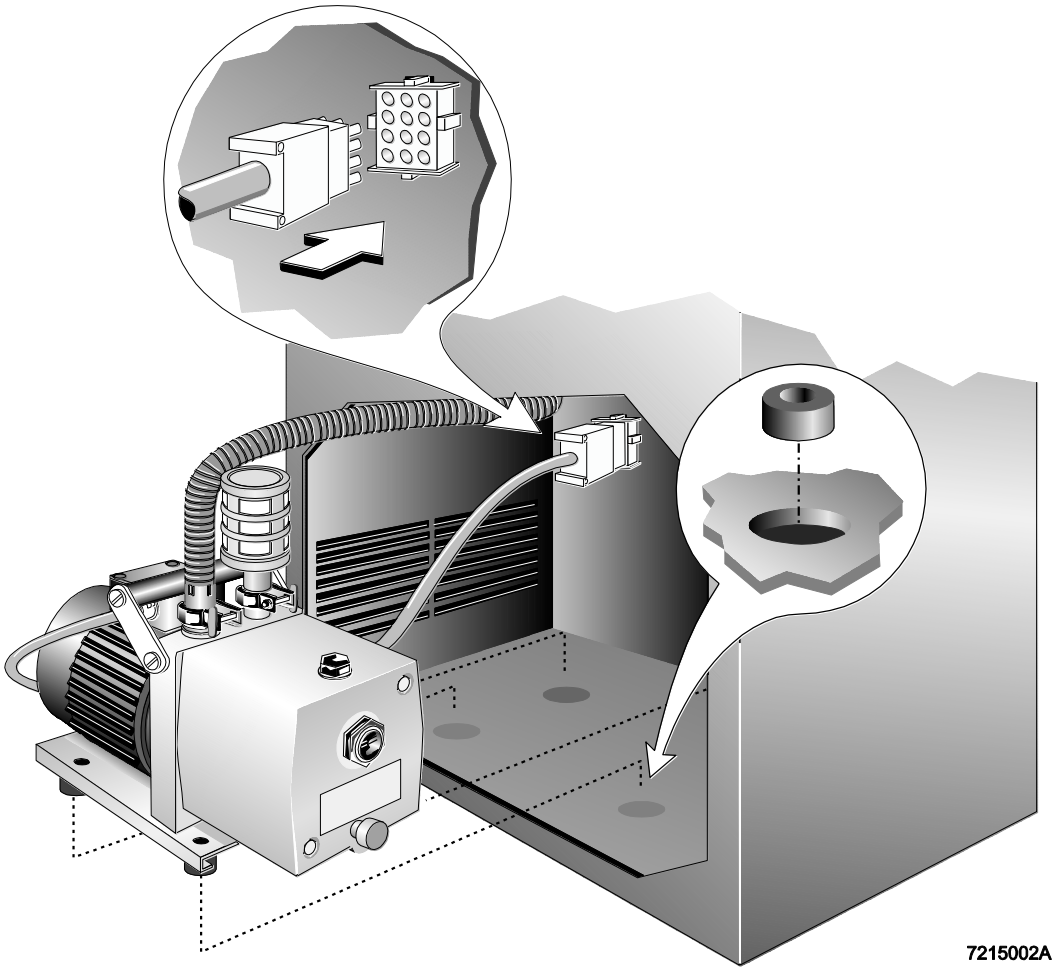


Figure 3.6 Placing Vacuum Pump into Pump Shelf

Installing Gas Cylinder Pressure Regulators (optional)

Before proceeding with any of the following setup procedures involving compressed gases, see Chapter 2, Precautions and Hazards.

Use this procedure to install the optional pressure regulator kit supplied by Beckman Coulter. Refer to Figure 3.7.

Note: In countries other than the USA, an adaptor may be necessary to connect the cylinder to the regulator.

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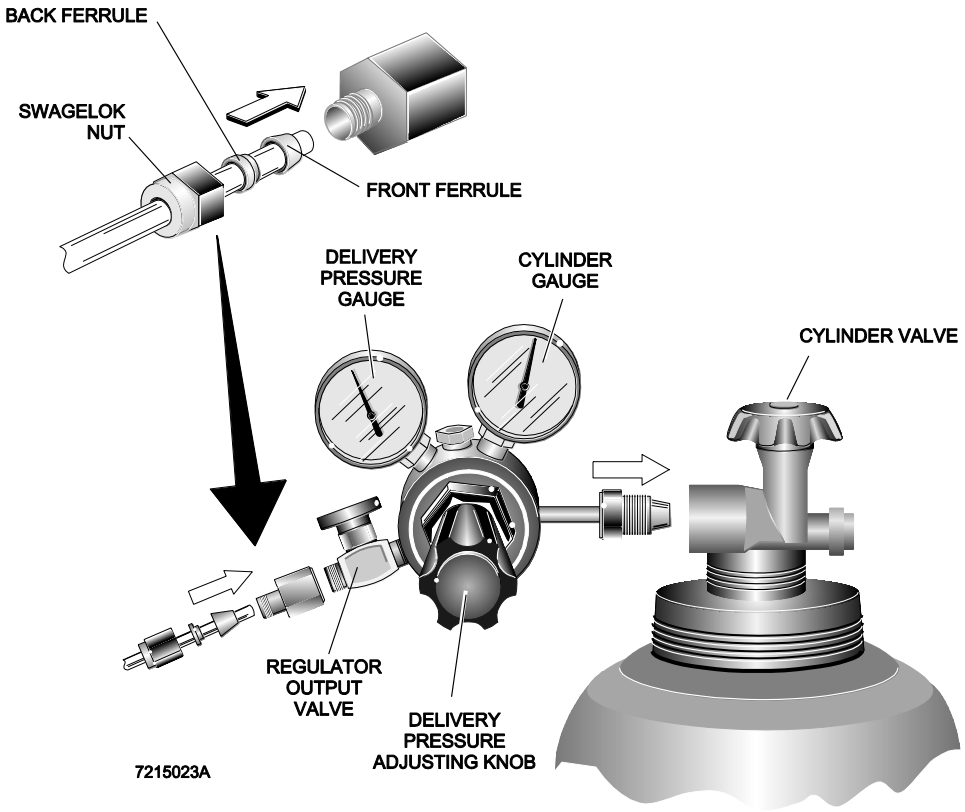


Figure 3.7 Optional Pressure Regulator Kit Installation

Required Item

1 1/8-inch AF (Across Flat) wrench

Procedure

WARNING

The regulator supplied is designed for only nitrogen, helium, and other inert gases. The regulator must not be used for any corrosive or hazardous gas.

WARNING

To prevent valve damage and potential personal injury, never use a nonstandard wrench or tool on any cylinder valve.

1. Make sure the cylinder valve is closed by tightening the knob or other valve mechanism clockwise.

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2. Carefully inspect the regulator for signs of contamination or damage. Examine all exposed and connecting threads for signs of wear or abuse. Make sure that there are no loose parts in the regulator other than the adjustment knobs and the connector nut.

WARNING

To prevent valve damage and personal injury, do not force the regulator-cylinder connection. If the regulator does not screw into the cylinder valve outlet easily, check for cross-threading or contamination.

CAUTION

Never use oil, grease, or Teflon® tape on any cylinder or regulator valve or connection threads.

3. Screw the regulator onto the gas cylinder valve outlet by hand. Never force a regulator connection. When the connection is hand tight, use the 11/8-inch AF wrench to secure the connection by tightening approximately 1/8 turn clockwise.

4. Close the regulator output valve by turning the small wheel clockwise.

5. Close the delivery valve by turning the delivery pressure adjusting knob counterclockwise until it turns freely.

6. Open the cylinder valve SLOWLY. When opening a valve, always turn the knob counterclockwise until it stops, then clockwise 1/4 turn. This prevents jamming while ensuring that the valve is completely open. The cylinder gauge (right-hand, high pressure gauge) registers the cylinder pressure.

7. Check for leaks in the regulator-cylinder connection by using soapy water around the threads. Bubbles from the joint will indicate a leak.

   If there are any leaks:

   a. Close the cylinder output valve.

   b. Turn the delivery pressure adjusting knob clockwise to open the regulator.

   c. SLOWLY open the regulator output valve to release pressure within the regulator. Both regulator gauges should descend to zero.

   d. Remove the regulator and inspect the joint components for cleanliness and repeat steps 3 through 7. If there is still a leak, either the regulator or gas cylinder may be damaged. Try another cylinder and repeat steps 3 through 7. If a leak persists, contact your Beckman Coulter Representative.

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IMPORTANT

The cylinder pressure must be at least 100 psig.

8. Make a note of the cylinder pressure.

9. Close the cylinder output valve. The measured pressure should not change. If the pressure changes, see step 7, above, about leaks.

10. Proceed with the installation with the cylinder valve closed, but check the pressure in 15 min to ensure there are no slow leaks. In the meantime, proceed with the setup. In the event of a slow leak, see step 7, above.

Connecting Gas Supply to the Analyzer

Required Item

7/16-inch AF wrench or adjustable wrench

Making the Connections

1. Close the pressure regulator output valves from the gas supply.

2. Make connections from the gas supply to the 1/8 in. gas lines:

   - If you have the optional pressure regulator kit supplied by Beckman Coulter, see Figure 3.7 and,

     a. Loosen the nut of the Swagelok fitting attached to the regulator output.

     b. Remove the nut, taking care not to lose the two ferrules inside the nut.

     c. See Figure 3.8. Thread the tubing through the nut, back ferrule, and conical front ferrule, ensuring that the orientations of the parts are correct as in the illustration.

     d. Push the tubing into the conical opening in the fitting on the regulator output, then slide the nut forward and finger tighten the nut onto the fitting.

     e. Use the 7/16-inch AF wrench (or adjustable) to tighten the nut 3/4 of a turn clockwise past finger-tight. This will 'swage' the ferrules onto the tubing to form a seal.

   - If you do not have the optional pressure regulator kit supplied by Beckman Coulter, make appropriate gas-tight connections from the gas supply to the 1/8-inch nylon tubing.

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3. Attach the gas lines to the labeled ports on the instrument, see Figure 3.8:

   a. Loosen the nut of the Swagelok fitting on the gas input to the instrument.

   b. Remove the nut, taking care not to lose the two ferrules inside the nut.

   c. Thread the tubing through the nut, back ferrule, and conical front ferrule, ensuring that the orientations of the parts are correct as in the illustration.

   d. Push the tubing into the conical opening in the fitting on the instrument, then slide the nut forward and finger tighten the nut onto the fitting.

   e. Use the adjustable wrench to tighten the nut 3/4 of a turn clockwise past finger-tight. This will 'swage' the ferrules onto the tubing to form a gas-tight seal.

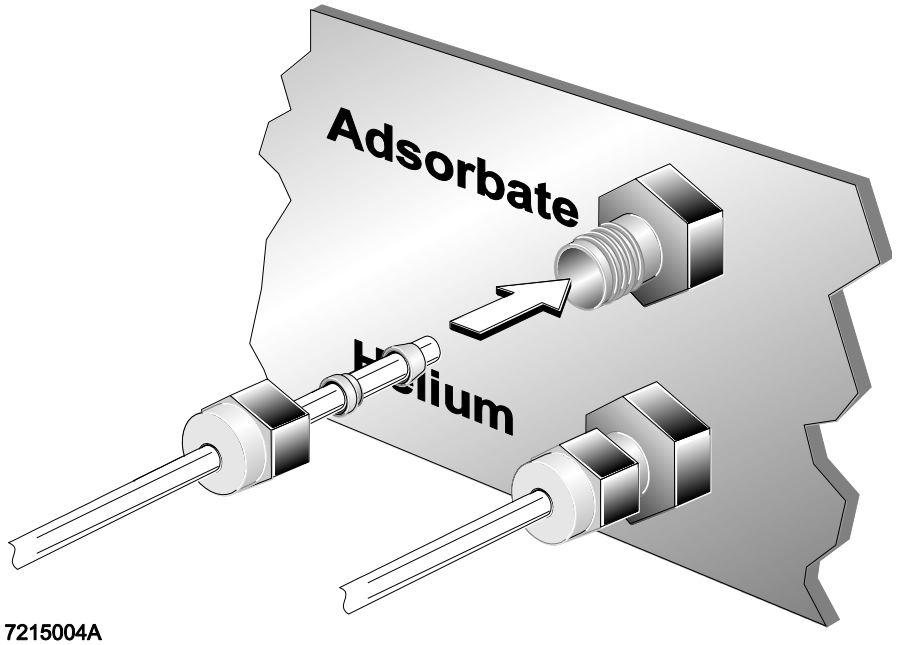


Figure 3.8 Gas Line Connections to the Analyzer

Purging the Gas Lines

1. Supply gas pressure to the gas lines:

   If your gas supply is in cylinders:

   a. Open the cylinder output valve.

   b. Turn the delivery pressure adjusting knob clockwise to set the regulator output pressure to between 12 psig and 15 psig.

   c. Slowly open the regulator output valve.

   If you have an alternative gas supply:

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Supply 12 psig to 15 psig to the gas lines.

2. Purge the gas lines:

   a. At the instrument, hold the gas line while loosening the Swagelok nut. Stop loosening the nut when you feel the tube starting to back out of the connection (you will also hear the gas escaping). Do not remove the nut.

   b. Hold the tube for 20 to 30 s to purge the tube, then push it back into the connector and finger-tighten the nut. Finger tighten the nut, then use the wrench to tighten the nut approximately 1/2 turn more.

   c. Repeat the procedure for the other gas.

Checking Gas Lines for Leaks

1. Make a note of the cylinder pressure. If you have the optional pressure regulator, the right-hand, or high-pressure gauge will display the cylinder pressure.

IMPORTANT

The cylinder pressure must be at least 100 psig.

---

2. Close the cylinder output valve. The measured pressure should not change.

   If the pressure changes, re-connect the gas line to the instrument and repeat the test.

   If the pressure does not change, proceed with the installation with the cylinder valve closed, but check the pressure in 15 min to ensure there are no slow leaks.

   If leaks persist, call your Beckman Coulter Representative.

Greasing and Seating O-rings

Required Items

High vacuum silicone grease
Clean glass shipping plug or sample tube

Procedure

Perform the following procedure for all five ports (saturation port, sample port, and three outgassing ports). Refer to Figure 4.1 if you are unsure of the location of these ports.

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CAUTION

To prevent damage to ports or fittings, do not use any tools to remove or install port fittings. Fittings are designed to seal when finger-tight.

1. See Figure 3.9. Loosen the knurled nut, slide the glass shipping plug out of the port, and then remove the nut. Ensure that there is a sleeve and O-ring inside the nut. If the O-ring is not inside the nut, it may still be inside the fitting, and can be removed with a small blunt object.

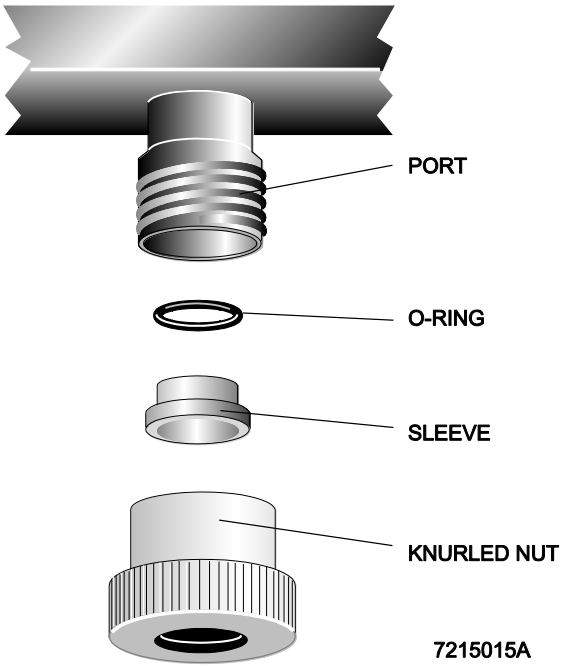


Figure 3.9 Port Fittings

2. Check the O-ring for dust or particles. If the O-ring is damaged, replace the O-ring. If the O-ring is contaminated, wipe it clean with lint-free laboratory tissue.

IMPORTANT

Excess grease on O-rings will be deposited on sample tubes, causing weighing and analysis errors.

3. Grease the O-ring very lightly with a thin film of high vacuum silicone grease. The surface of the O-ring should appear to be just slightly wet. There should be no excess grease on the O-ring.

4. See Figure 3.10. Clean the glass shipping plug or sample tube with lint-free laboratory tissue. Slide the knurled nut, sleeve (ensure the sleeve is narrow side up), and O-ring over the end of the plug or sample tube.

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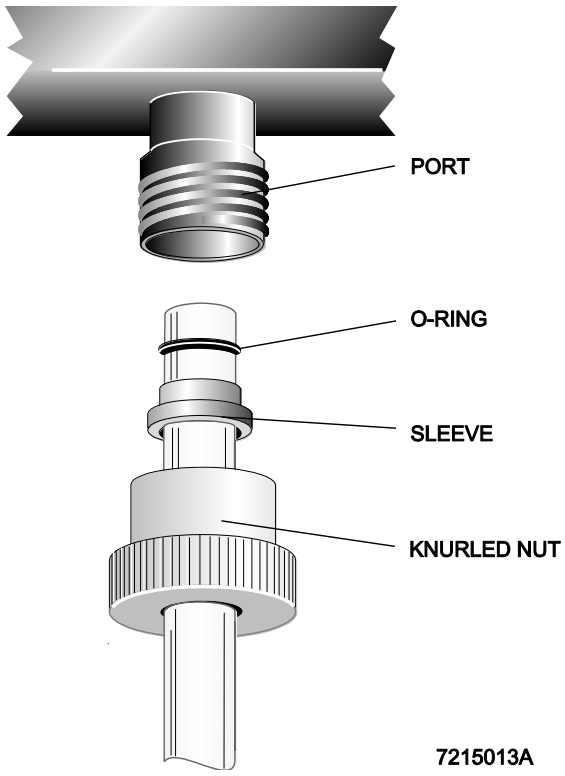


Figure 3.1 0 Seating the O-ring

5. Insert the plug or tube into the port until it stops against the manifold, then slide the knurled nut up the tube and tighten until finger-tight. The O-ring is now seated.

   Note. The tube may appear to be loose within the fitting, but if a gentle downward pull does not pull the tube out of the fitting, the O-ring is correctly seated.

6. Loosen the nut 1/4 turn and remove the plug or tube. Retain the shipping plugs in case you need to store or ship the unit in the future. These prevent dust from entering the manifolds.

Installing the Saturation Tube

Ensure that the saturation tube is clean and dry before installing.

Required Items

Saturation tube
Tube insert
Saturation tube heat sink

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Procedure

CAUTION

Do not use any tools to remove or install saturation port, adsorption port, or outgas port fittings. Fittings are designed to seal when finger-tight.

1. See Figure 3.11. Slide the saturation tube heat sink onto the end of the saturation tube so that the bottom of the tube is flush with the bottom of the heat sink. To loosen the heat sink and make it easier to install, hold the left end of the heat sink while twisting the right end away from you. Make sure the heat sink is secure on the tube.

2. Insert the tube insert into the saturation tube.

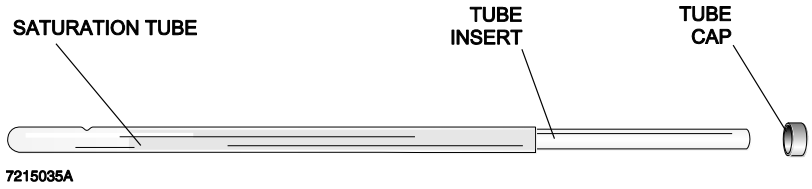


Figure 3.1 1 Saturation Tube, Insert and Heat Sink

3. See Figure 3.12. Loosen the knurled nut on the saturation port.

4. Insert the saturation tube into the saturation port until it stops against the manifold.

5. Tighten the knurled nut until finger-tight.

The sleeve compresses the O-ring against the glass tube, forming a gas-tight seal. The tube does not have to feel rigid in the fitting to be sealed. If a gentle downward pull on the tube does not pull it out of the fitting, it is installed properly.

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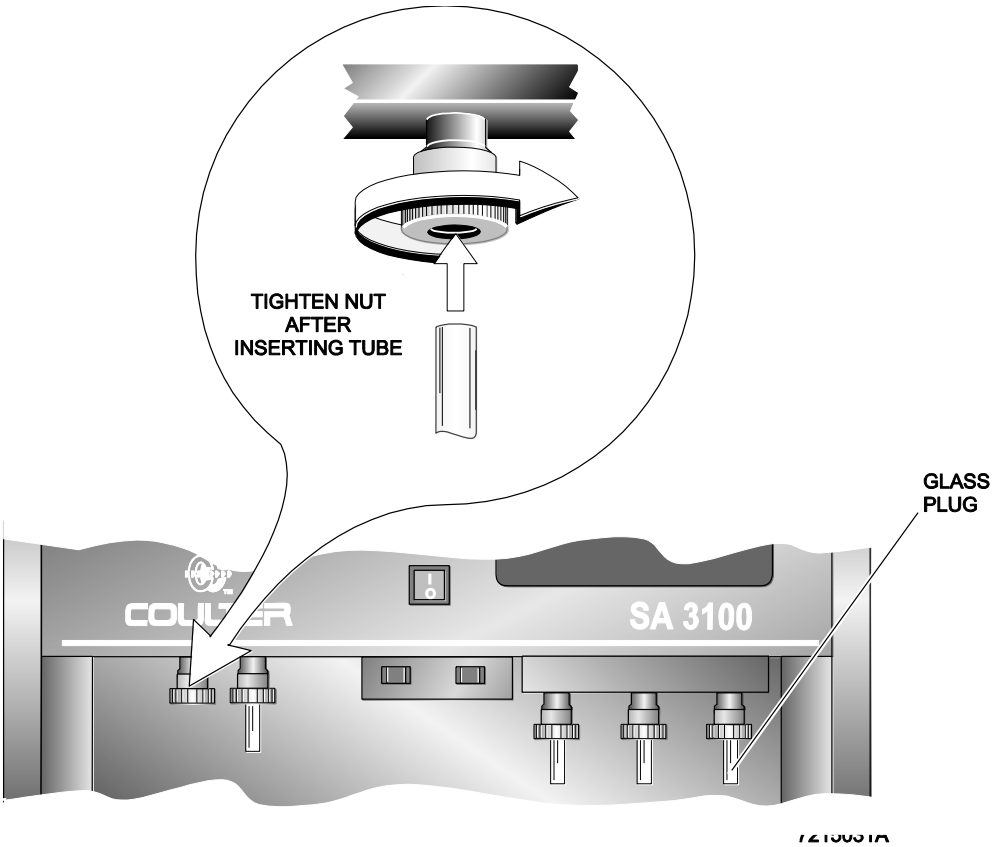


Figure 3.1 2 Saturation Tube Installation

3.5 POWER-UP

Required Item

IBM®-formatted 3.5 in. diskette

Procedure

See Chapter 10, Specifications, for the local power specifications for the SA 3100 analyzer.

See Figure 3.13.

1. Insert the diskette into the disk drive.

2. Plug the power cord from the instrument into an electrical outlet.

3. To turn the instrument on, press the top of the red rocker switch (labeled I) until it clicks.

The unit takes about 15 s to boot up. The **Analysis** screen, Figure 3.14, appears on the touch screen display when the software has been loaded. The Analysis Status key contains a message reading *Initializing* for 5 min or until the manifold equilibrates at

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45°C, whichever is longer. If the instrument is cold, equilibration may take 1 h or longer. When the equilibration is complete, the Analysis Status key displays a message reading *Ready for Next Analysis*, and the SA 3100 analyzer is ready for use. If a message other than *Ready for Next Analysis* appears call your Beckman Coulter representative.

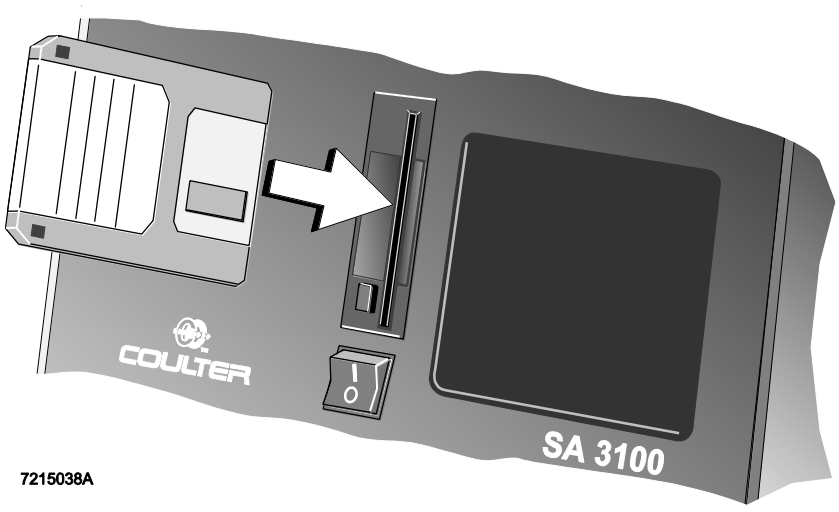


Figure 3.1 3 Analyzer Power-up

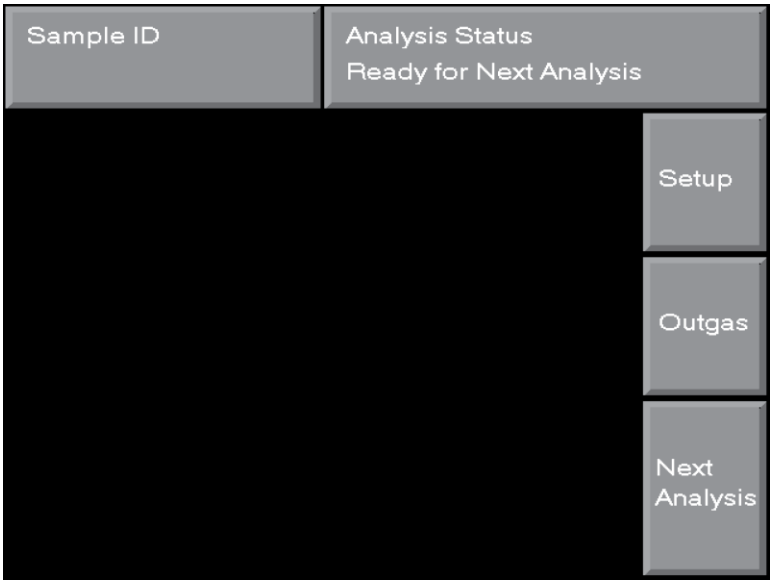


Figure 3.1 4 Analysis Screen

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3.6 FLUSHING GAS LINES

General

The SA 3100 analyzer has an automatic procedure to flush the internal gas lines and manifolds.

Procedure

To flush the internal gas lines and manifolds:

Note. If you are not familiar with touch screens, you can make selections by touching the appropriate button on the touch screen using your finger or other soft, blunt implement.

a. On the **Analysis** screen, Figure 3.14, press Setup, the **Setup and Print** screen displays.

b. Press Flush Gas Lines, the key will depress momentarily and you will hear a click. Press OK to return to the **Analysis** screen. This initiates an automatic procedure which flushes both the Helium and Adsorbate gas lines and manifolds inside the instrument. The procedure takes about 5 min per gas line (10 min total). You can monitor the process by pressing Analysis Status to display the system schematic.

3.7 PRINTER INSTALLATION

General

The SA 3100 analyzer is compatible with 300 dpi laser printers, some inkjet printers, and dot matrix printers. Graphs are only available when using laser printers which support the HPGL2 printer language. Contact your Beckman Coulter representative to find out if a printer is compatible with the SA 3100 analyzer.

Required Items

Parallel interface printer
Printer power cable
Parallel printer interface cable

Procedure

See Figure 3.15.

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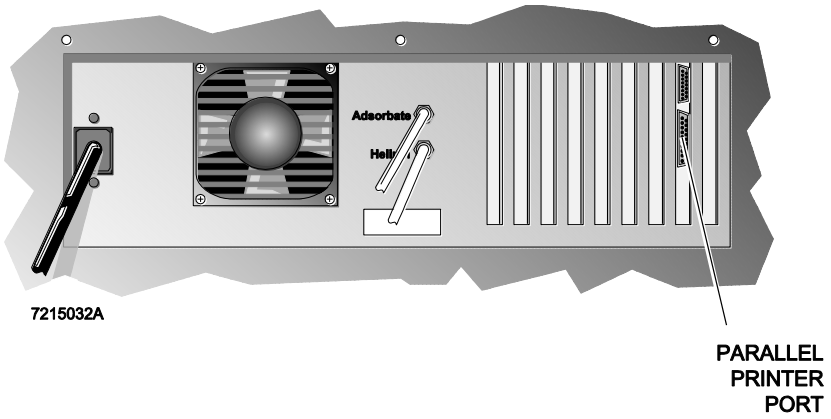


Figure 3.1 5 Printer Installation

1. Attach the parallel printer cable to the printer port on the instrument and the appropriate port on the printer.

2. Refer to the relevant printer manufacturer's manual for printer setup and power-up instructions.

3. After the printer completes any self-tests, configure the printer at the SA 3100 analyzer as follows:

   Note. If you are not familiar with touch screens, you can make selections by touching the appropriate button on the touch screen using your finger or other soft, blunt implement.

   a. Press Setup on the **Analysis** screen, Figure 3.14. The **Setup** screen appears.

   b. Press Configuration on the Setup screen, the **Configuration** screen appears.

   c. Press Printer on the **Configuration** screen, the **Printers** screen appears.

   d. Select Dot Matrix, Laser, or Inkjet by pressing the appropriate square key.

   e. Press OK to confirm your choice and return to the **Configuration** screen. Press OK twice more to return to the **Analysis** screen.

The printer is now installed and configured. The type of printed output is defined as part of the analysis profile, see the subheading Profiles in section 4.4 USER INTERFACE.

3.8 ADVANCED SETUP - USER INTERFACE

To set the date, the time, the format for filenames to be used, and whether or not users can modify or define new profiles (analysis protocols), use the **Advanced Setup** screen. For details, see Chapter 7, heading 7.15, Advanced Setup.

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4.1 SYSTEM OVERVIEW

The COULTER SA 3100 and SA 3100 Plus analyzers are bench-top Surface Area and Pore Size Analyzers with built-in vacuum pumps. They measure surface areas, pore size distributions, and pore volume parameters of powdered, pelleted or solid piece samples. The SA 3100 Plus analyzer has the added capability to measure desorption isotherms and perform the pore parameter calculations associated with desorption isotherms. Unless specified otherwise when discussing to features specific to the SA 3100 Plus analyzer, both instruments are referred to as SA 3100 analyzers.

The instrument is very easy to learn and use due to the simple, graphical touch screen user interface. The interface allows you to create analysis profiles which store the run parameters you use most often. Run setup is therefore streamlined. In addition, isotherms (see Chapter 6, A Brief Overview of Gas Sorption) for commonly run materials can be stored and used by the instrument to speed analysis through the use of a special algorithm.

The SA 3100 analyzer has some features which can only be activated using the 'SA 3100 Advanced Configuration Kit'. These features are discussed in heading 4.5, ADDITIONAL FEATURES.

The SA 3100 analyzer provides highly accurate results for both quality control and research and development applications.

4.2 SYSTEM FUNCTION

The SA 3100 analyzer is a completely integrated sample preparation, sample analysis, and data processing system. This section gives a brief overview of system operation.

Control

The instrument is controlled by a simple touch screen user interface. The interface displays the real-time status of all instrument functions, provides touch-key operation, and allows you to customize the SA 3100 analyzer for your applications.

Sample Preparation - Outgassing

Samples are prepared for surface area or pore size analysis on the SA 3100 analyzer by outgassing. Outgassing removes adsorbed gases and moisture from the sample surface. Outgassing is performed by evacuating gases from the sample tube(s) with the vacuum pump. The sample tube(s) are heated for outgassing by the outgassing furnace. Three samples can be simultaneously outgassed at the three outgassing ports. Sample analysis can proceed independently of and simultaneously with outgassing.

The COULTER SA-PREP™ six-station outgasser is also available if increased throughput is required. The COULTER SA-PREP outgasser uses the flowing gas method to purge samples of adsorbed gases and moisture. This accessory has six purging stations and six cooling stations, and can therefore keep up to six SA 3100 analyzers continuously supplied with

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samples for analysis to avoid any wasted sample preparation time. Contact your Beckman Coulter representative for more information.

Sample Analysis

General

Many types of analyses can be performed with the SA 3100 analyzer, see Chapter 6, Principles of Operation, for further details. All analyses, however, involve the adsorption of a gas (called the adsorbate gas) onto the surface and into the pores of a sample prepared by outgassing. (Adsorption is the condensation of gas molecules onto the surface of a material.) The adsorbate gas is added in known doses into an evacuated tube containing the sample, incrementally increasing the quantity of adsorbed gas and the pressure in the sample tube. During this process, the sample tube is kept at a constant temperature. When the pressure in the tube has equilibrated following each dose of adsorbate gas, the pressure is recorded. The pressure readings are used, in turn, to calculate the volume of gas adsorbed.

The volume of gas adsorbed is measured as a function of relative pressure. Relative pressure is the ratio of the pressure in the sample tube to the saturation vapor pressure of the adsorbate gas (the pressure at which the adsorbate gas liquefies). The saturation pressure is measured for every sample tube pressure data point. During the measurement, the sample is maintained at a temperature where liquefaction of adsorbate gas can take place on the sample. To maintain this temperature, the sample tube is immersed in a dewar of liquid cryogen (usually nitrogen) during analysis. Some types of analysis measure the desorption of gas from the surface and pores of the sample. Desorption is the removal of gas molecules from the material. Only the SA 3100 Plus analyzer can perform those types of analysis which require desorption measurements. The resulting data set is called an adsorption or desorption isotherm, because the measurement is performed at constant temperature.

The surface area, pore size distribution, and pore volume parameters are calculated from the isotherm data. Depending on the type of analysis required, an adsorption or desorption isotherm may consist of as few as three points or as many as 100 to 200. Analysis time is a function of the number of points required.

Fast BET Analysis

The SA 3100 analyzer has a special Fast BET mode (see Chapter 6, Principles of Operation for an explanation of BET analysis) which permits accelerated surface area analysis when increased throughput is required. See heading 7.4, Profiles, for information on running Fast BET analyses.

Output

General

Results from analyses are automatically written to the diskette, and may be printed automatically at the completion of analysis if a printer is installed and configured. Output to the diskette is in ASCII format. The COULTER SA-VIEW Microsoft Windows-based program can be used for reviewing and comparing data files, printing data, and copying data to other Windows applications. See Appendix A for details of SA-VIEW software.

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RS232C Port and Networking

SA 3100 analyzer data can also be directed to network servers or other computers. To configure the unit to do this, the optional 'SA 3100 Advanced Configuration Kit' is required. Contact your Beckman Coulter representative for details.

4.3 COMPONENTS

Figures 4.1 (front view) and 4.2 (back view) illustrate the SA 3100 system. The main components are called out in the figures and described below.

Control Components

Power Switch

The red rocker switch is the power switch. To turn the instrument on, press the top of the switch (labeled I) until it clicks and is flush with the instrument case. To turn the instrument off, rock the switch back to its original position by pressing the bottom of the switch (labeled O).

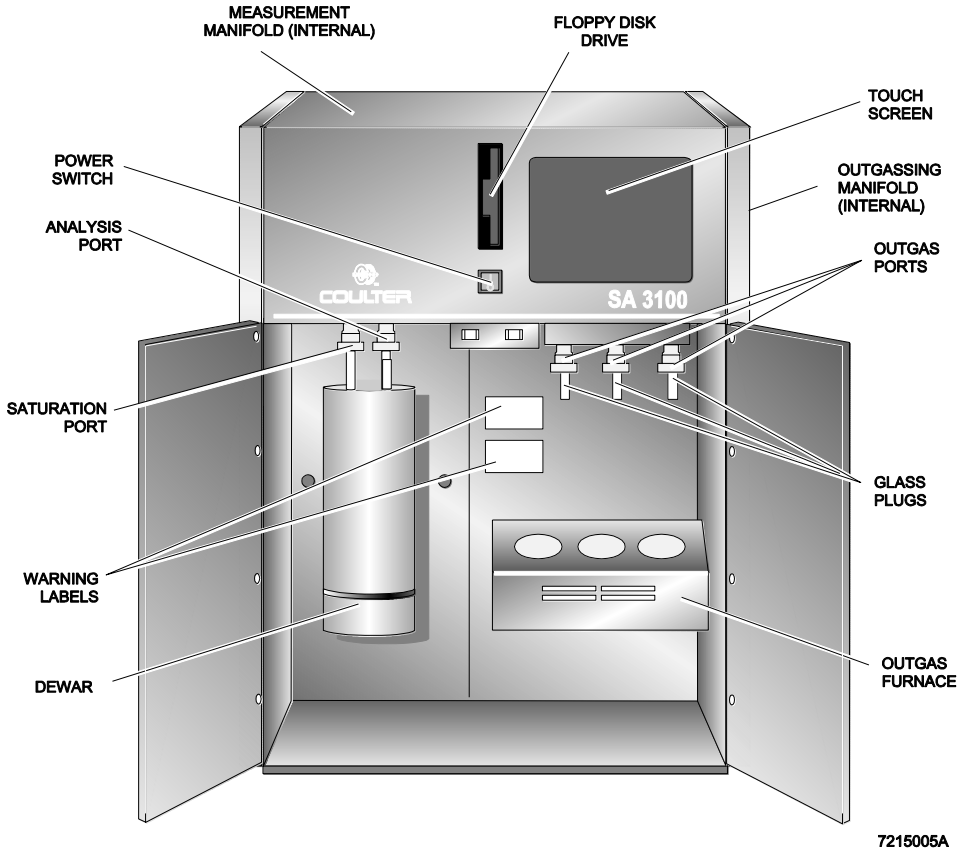


Figure 4.1 SA 31 00 Analyzer Front View

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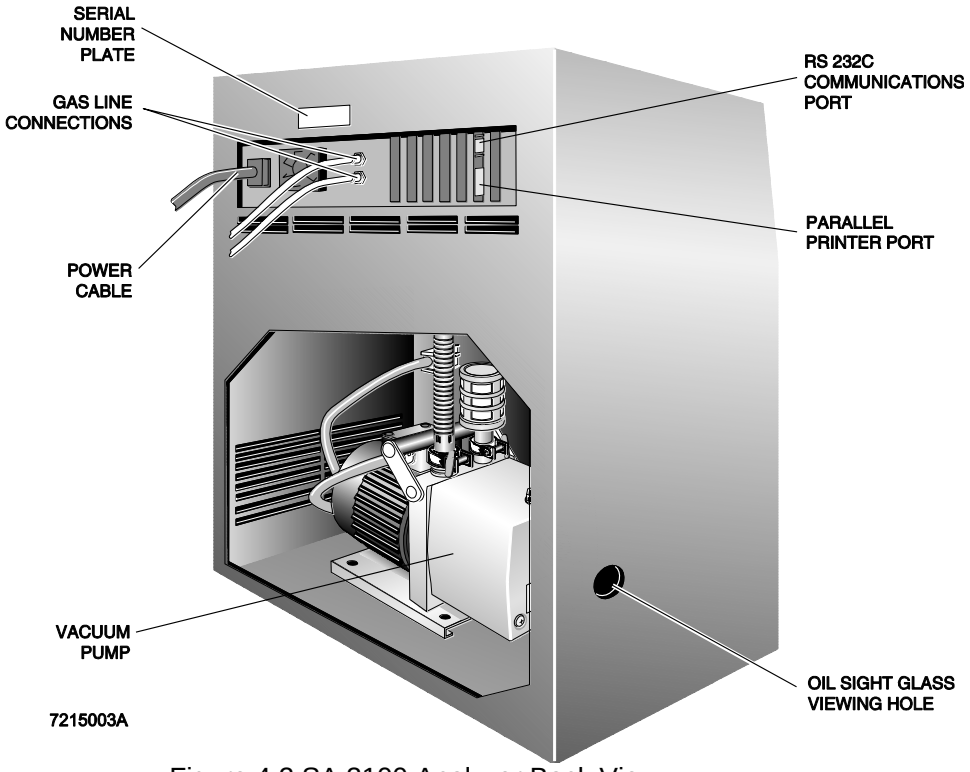


Figure 4.2 SA 3100 Analyzer Back View

Touch Screen

When the instrument is turned on, the touch screen is lit and displays an interface screen with touch keys. All instrument control is accomplished by the touch screen. The keys are activated by touching with your finger or other soft blunt object (such as a pencil eraser). If the screen is not touched for 15 minutes, a screen saver becomes active, displaying only the status of the instrument. Touch the screen to restore the full display.

Mouse

The SA 3100 analyzer can be configured for use with a mouse for those who cannot or do not wish to use the touch screen. The 'SA 3100 Advanced Configuration Kit' is required to configure the system for a mouse.

Measurement and Outgassing Components

Measurement Manifold (Internal)

The measurement manifold is the heart of the SA 3100 analyzer. It consists of a gas manifold, valves, and pressure transducers, and connects the adsorption port, the vacuum pump, and the gas inputs.

Analysis Port

The sample tube is attached to the analysis port for sample analysis.

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Saturation Port

The saturation tube is attached to the saturation port. The saturation tube is used to measure the saturation vapor pressure of the adsorbate gas, functioning as a vapor pressure thermometer.

Outgassing Manifold (Internal)

The outgassing manifold consists of gas valves and connections to the measurement manifold.

Outgassing ports

Sample tubes are attached to the outgassing ports for outgassing. Three samples can be outgassed simultaneously. Sample analysis can proceed while other samples are being outgassed.

Outgassing Furnace

The outgassing furnace heats the samples to the required temperature for outgassing. You set the temperature appropriate for your sample.

Dewar

The dewar holds liquid for a constant temperature bath to keep the sample tube and saturation tube at the temperature required for analysis.

Vacuum Pump

The vacuum pump achieves the low pressures necessary for outgassing and sample analysis.

Gas Lines

Helium and the adsorbate gas lines connect to the respective gas cylinders (see Heading 3.1, Special Requirements). Gas must be supplied to the SA 3100 analyzer at a pressure of 12 psig to 15 psig.

Output Components

Floppy disk drive

The high density floppy disk drive is provided for data storage. Preformatted diskettes (IBM format) must be used.

Printer Port

This is a standard parallel printer port. Beckman Coulter offers a range of dot-matrix, inkjet, or laser printers compatible with the SA 3100 analyzer.

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Other

Communications Port

The RS232C communications port can be used to send data to an external computer if the instrument is configured accordingly. The 'SA 3100 Advanced Configuration Kit' is required for this feature.

4.4 USER INTERFACE

The user interface of the SA 3100 analyzer is straightforward and flexible. It has the power to streamline your procedures through the use of Profiles (see Profiles, below) while its simple architecture makes it easy to learn. This section introduces the interface by describing its basic structure. For complete operating instructions see Chapter 7, Operating Instructions.

Touch Screen

The instrument is controlled entirely through the touch screen. The touch screen allows you to perform all instrument functions without a keyboard, keypad or mouse. The touch screen also displays results of analyses.

When a message requiring your attention appears on the screen an audible beep sounds. If you are currently viewing a screen other than the one on which the message is displayed, an asterisk also appears in the upper right hand corner.

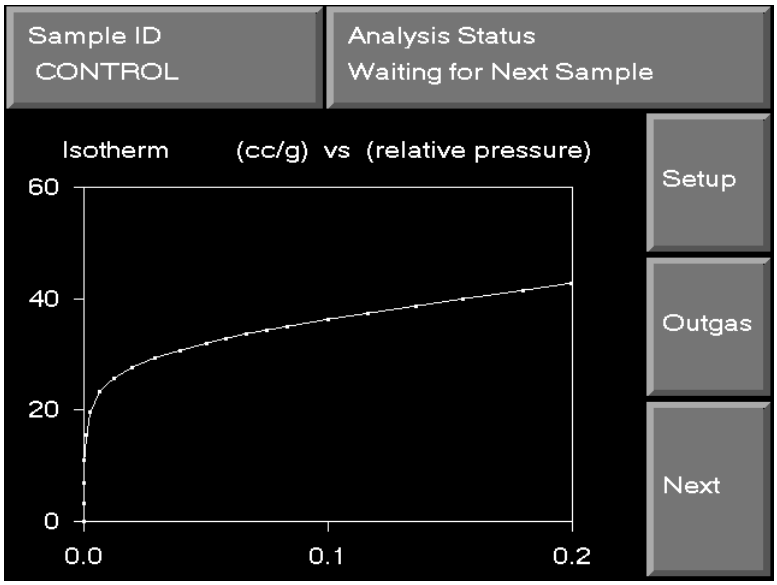


Figure 4.3 Analysis Screen

Keys on the Touch Screen

When you turn your system on, the **Analysis** screen is displayed, see Figure 4.3. Along the top and right-hand sides of the screen are several keys. Each key represents a command

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or provides access to another screen where commands may be executed or choices made. Some screens contain numeric keypads, Figure 4.4, or alphanumeric (QWERTY) keypads, Figure 4.5.

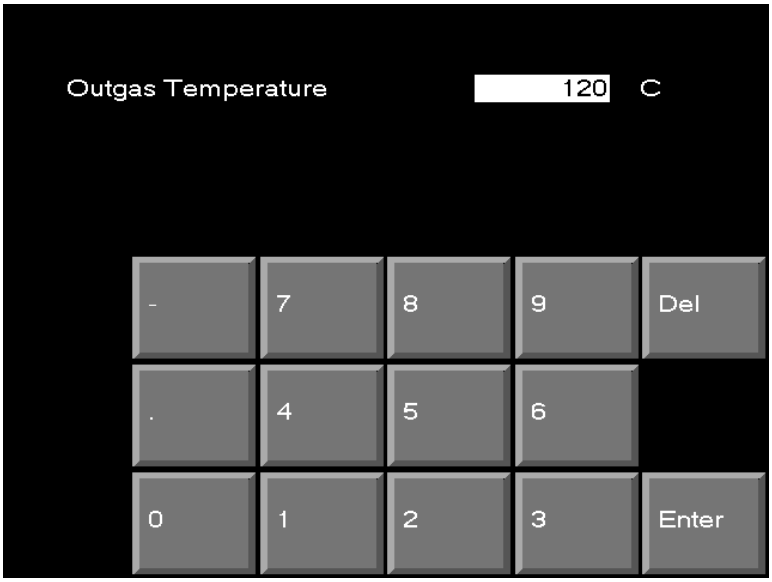


Figure 4.4 Example of Nurneric Keypad on the Touch Screen



Figure 4.5 Example of Alphanumeric **(QWERTY)** Keypad on the Touch Screen

To activate the function or character represented by a key, simply touch the key on the screen. You will see the key depress. If Key Click is enabled in the system configuration, you will also hear a click when the key is depressed. If for some reason the system cannot respond immediately, the key will remain depressed until the system is free.

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OK, Cancel, and Enter Keys

These keys are common to several screens that allow you to enter information or make choices. The keys function as follows:

OK — Confirms choices made in that screen and takes you out of the screen to your original position in the interface.

Cancel — Cancels any choices made in a screen and exits the screen.

Enter — Confirms an entry made on a QWERTY or numeric keypad and exits the screen.

Interface Structure

The interface consists of two main screens from which all important functions can be accessed. The two screens correspond to the two principal functions of the SA 3100 analyzer: outgassing samples and analyzing samples. Each main screen can be accessed from the other by touching a key. The main screens and their component keys are described below.

Analysis Screen

The first screen to appear when you power on the instrument is the **Analysis** screen, Figure 4.3. During and after an analysis, the central area of the screen displays analysis data or status messages, depending on the current state of the system. In Figure 4.3, isotherm data from a BET surface area analysis is displayed on the screen.

After an analysis has been started, and the instrument has begun collecting data, the central area of the **Analysis** screen is active; touching anywhere within the central area will provide you with a screen with which you can choose different displays as necessary. After a BET analysis, for example, you may choose to view the numerical BET surface area results or the adsorption isotherm.

Note. If a command message is displayed, requiring you to perform an action before the system can proceed, the central area of the **Analysis** screen is not active.

The **Analysis** screen is used to initiate analyses. The keys on this screen are:

Sample ID — Displays the ID of the current (prepared and outgassed) sample. Touching the key accesses the sample information screen, which allows you to edit information for the current sample (including the analysis profile selected, see Profiles, below), choose from a list of other outgassed samples, or enter information for new samples.

Analysis Status — Displays the current status of the analysis module. Touching the key accesses the analysis status display screen, which graphically displays

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the state of the analysis manifold, including valve states, pressures and manifold temperature.

Setup — Accesses the setup screen, which allows you to print data, change the configuration of the system, and edit profiles.

Outgas — Takes you to the **Outgas** screen.

Next Analysis — Clears results of the previous analysis from the screen and memory and allows you to choose the next sample from the list of logged-in outgassed samples, or enter information for a new sample. Also allows you to delete samples from the outgassed samples list.

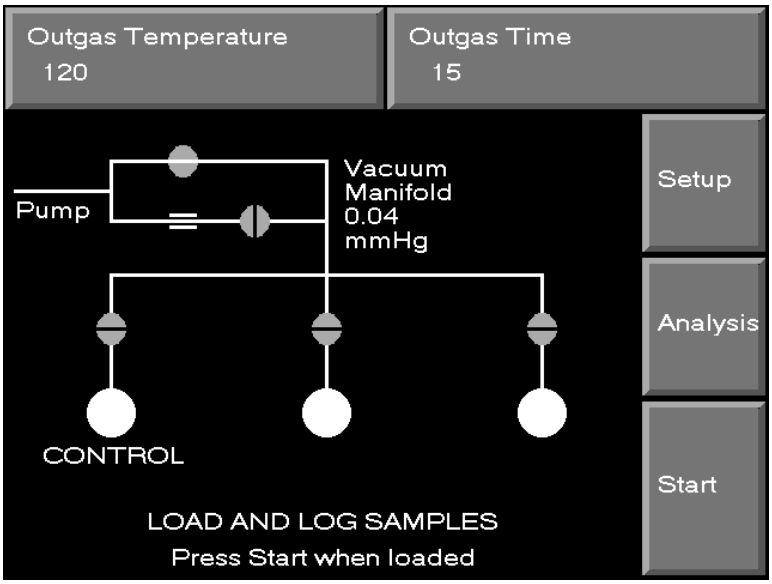


Figure 4.6 Outgas Screen

Outgas Screen

The other main screen is the **Outgas** screen, Figure 4.6. The central area of the **Outgas** screen graphically displays the status of the outgassing manifold. The circular graphics representing the sample tubes on the outgas ports are also active keys. Each key allows you to enter sample information for the samples that are to be outgassed on the respective outgas ports. The other keys on this screen are:

Outgas Temperature — Displays the temperature for sample outgassing, and allows you to change the outgas temperature.

Outgas Time — Displays the time for which to outgas the sample at the specified temperature, and allows you to change the outgas time.

Setup — Same as the setup key on the **Analysis** screen.

Analysis — Takes you to the **Analysis** screen.

Start Outgas — Initiates the outgassing procedure.

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Profiles

The Setup key in both main screens provides access to, among other functions, the **Profile** screen, Figure 4.7. Profiles allow you to set the parameters for sample analyses, such as the adsorbate gas, the measurement type, the desired output, and others. Profiles can be stored for repeated use, and so streamline operation of the SA 3100 analyzer by saving setup time. When you enter sample information during sample log-in, you also choose the profile for analysis of that sample.

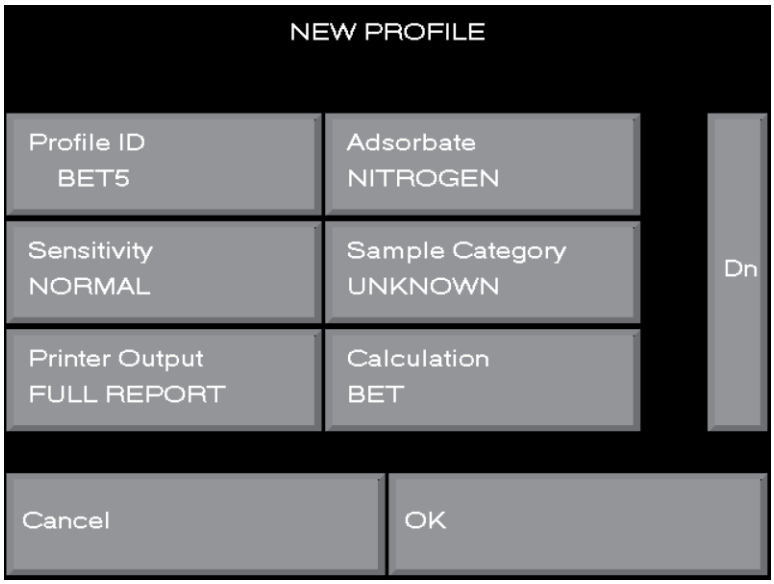


Figure 4.7 Profile Screen

4.5 ADDITIONAL FEATURES

Some features of the SA 3100 analyzer can only be activated through the use of the 'SA 3100 Advanced Configuration Kit'. This kit allows you to:

- Configure the instrument for use of a mouse.
- Configure the instrument to use Krypton and Argon as adsorbate gases.
- Configure the RS232C port to send data to an external computer.
- Configure the instrument to send data to a network server.
- Configure the instrument to take data at specific P/P_0 values, for data processing elsewhere by external calculations.
- Change the labels 'Customer' and 'Operator' on these fields on the **Sample Information** screen.

Contact your Beckman Coulter representative for more information on the 'SA 3100 Advanced Configuration Kit'.

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5.1 OVERVIEW

In order to ensure the instrument is operating correctly, and also to introduce you to the process of running a sample on the SA 3100 analyzer, we strongly recommend that you measure the specific surface area of the control sample shipped with your instrument. Beckman Coulter recommends that you run the control sample at least weekly to verify instrument performance, although the frequency with which you run the sample depends on your protocols and how much data you can afford to lose in the unlikely event the instrument is not performing to specifications. The procedure below will lead you through the process of analyzing the sample. Complete operating instructions are presented in Chapter 7, Operating Instructions.

5.2 ANALYZING THE SAMPLE

Required Items

Make sure you have the following items before beginning.

Beckman Coulter RapiTube sample tube
Sample tube insert
Sample tube cap
Control sample
Analytical balance
Sample loading funnel
Dewar
Dewar filling funnel
Insulating dewar lid

Procedure

The procedure for analyzing a sample on the SA 3100 analyzer consists of six steps:

1. Weighing the Sample Tube Assembly
2. Adding Sample to the Sample Tube
3. Logging-in the Sample
4. Outgassing the Sample
5. Weighing the Outgassed Sample
6. Analyzing the Sample

These steps are explained in order below. Reviewing the results is discussed after the analysis procedure.

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Weighing the Sample Tube Assembly

You must carefully weigh the dry, empty sample tube assembly before proceeding in order to weigh the sample accurately after outgassing. The sample tube assembly consists of the tube and tube insert.

1. Refer to Figure 5.1. Insert the tube insert into the sample tube. The tube insert decreases the free volume inside the tube, increasing sensitivity and accuracy.

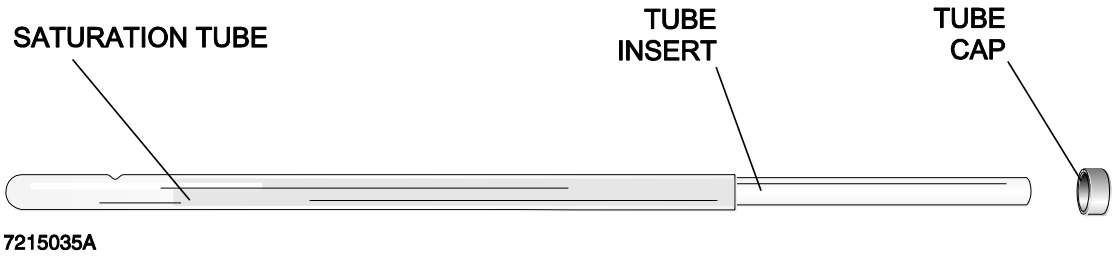


Figure 5.1 Sample Tube Assembly

2. Weigh the tube (including the tube insert) on the analytical balance to at least 0.001g precision. Make sure the following conditions are met before weighing the sample tube assembly:

   - The sample tube is at room temperature.
   - The sample tube is clean, dry, and without fingerprints. Use a lint-free laboratory tissue to remove fingerprints.
   - The weighing pan on the analytical balance is clean.
   - The sample tube is not charged with static electricity. Use an anti-static device to discharge the tube.

Make a note of the weight - you will need it later. Ensure that the insert used for this weighing stays with the sample tube. Interchanging parts after weighing introduces significant weight errors. Spaces are provided on sample tubes for marking, and indelible lab markers can be used on tube inserts to identify them uniquely. Don't make marks after weighing, however, as this changes the weight of the parts.

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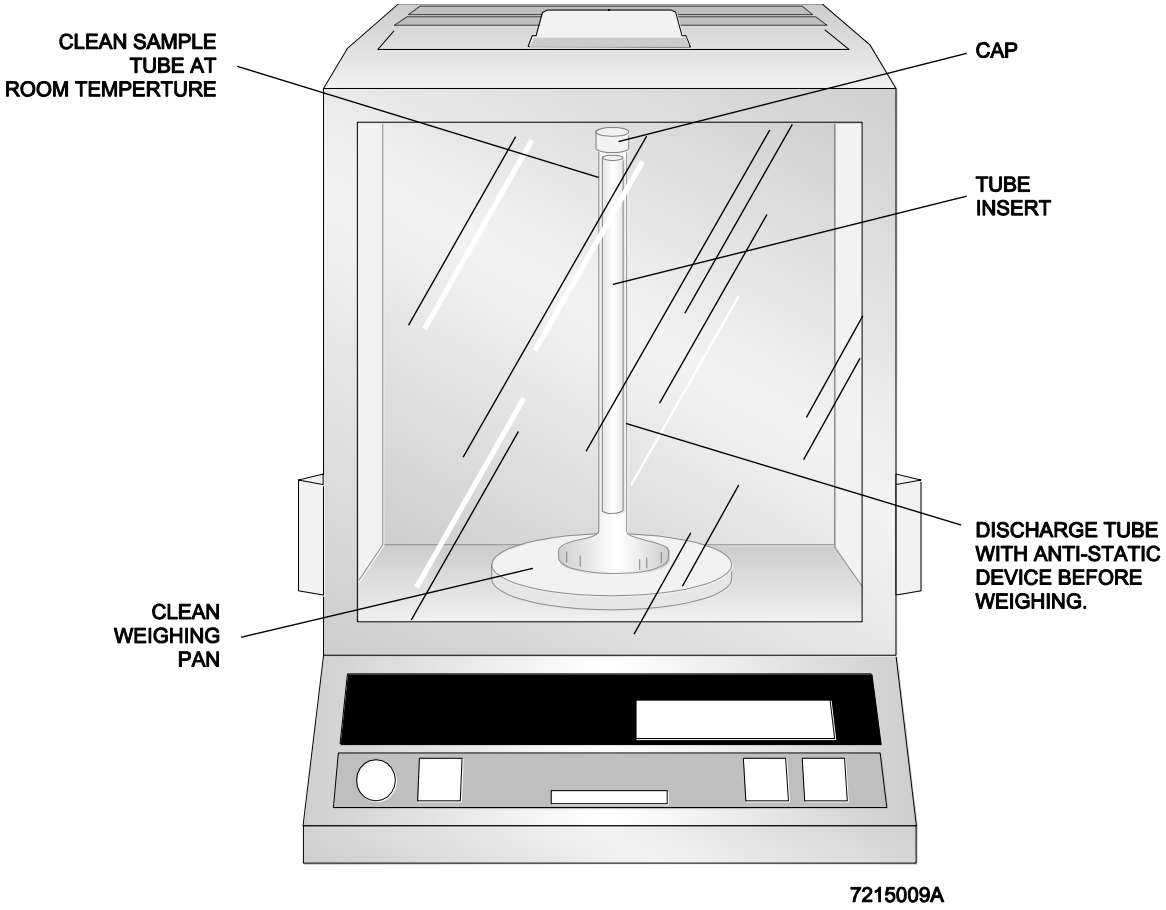


Figure 5.2 Weighing the Sample Tube Assembly

Adding Sample to the Sample Tube

The amount of sample added to the sample tube should be within certain limits to ensure the fastest and most accurate analysis.

1. Refer to the assay sheet contained in the control sample package. Weigh out a quantity of sample as recommended in the sheet under 'SAMPLE RUNNING INSTRUCTIONS'.

2. Remove the tube insert from the sample tube, then pour the weighed sample into the sample tube using the sample loading funnel, Figure 5.3.

3. Replace the tube insert in the sample tube.

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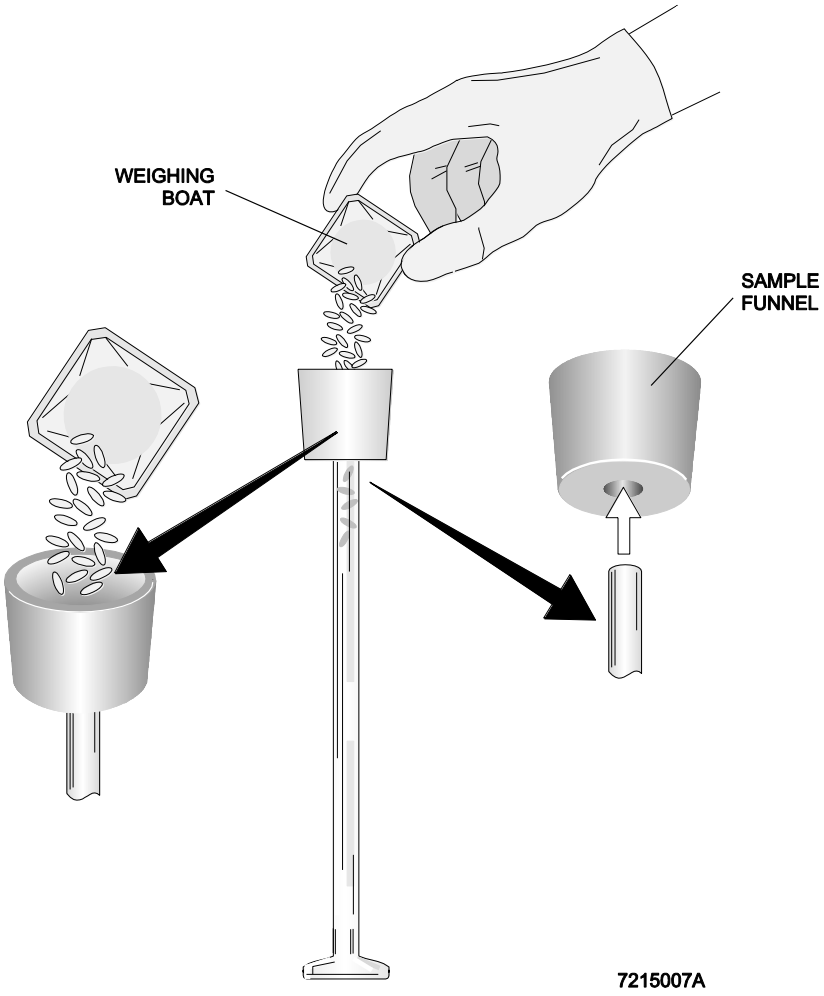


Figure 5.3 Adding Sarnple to the Sarnple Tube

Logging-in the Sample

Logging in sample information allows the SA 3100 analyzer to keep track of sample status, and perform the desired analysis. In the following procedure you will enter the proper parameters for outgassing and analysis.

1. If no keys have been pressed since the unit was turned on, the **Analysis** screen is displayed, see Figure 5.4. Press the Outgas key to move to the **Outgas** screen. Figure 5.5 illustrates the **Outgas** screen. If the **Outgas** screen is displayed, go to step 2.

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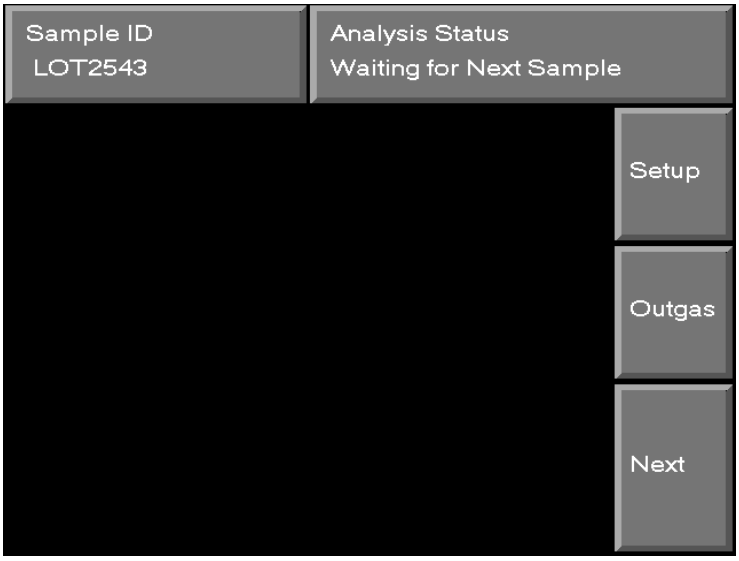


Figure 5.4 Analysis Screen

If a screen other than the **Analysis** or **Outgas** screen is displayed, press keys if available, or the key if a QWERTY or numeric keypad is displayed, or the key, until one of the two main screens is displayed.

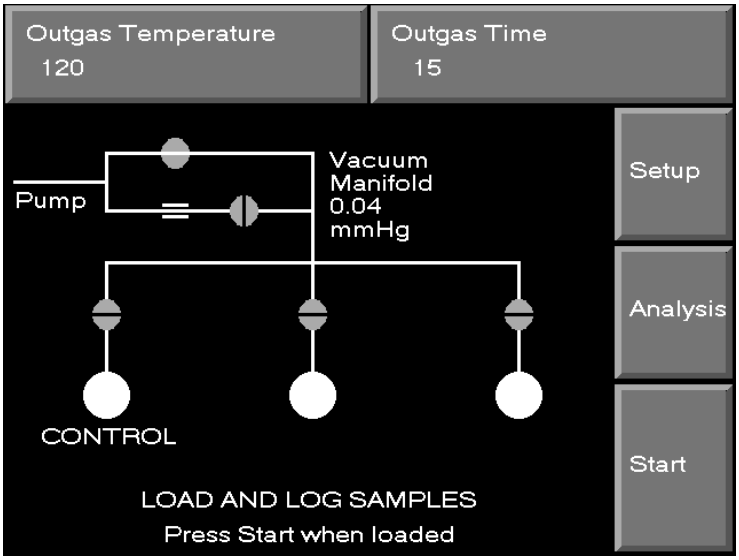


Figure 5.5 Outgas Screen

2. If no samples are currently outgassing, the instrument is ready to outgas the sample, indicated by the following message at the bottom of the **Outgas** screen

   LOAD AND LOG SAMPLES
   Press Start Outgas when loaded

3. In the central area of the **Outgas** screen, the three circles at the bottom of the vertical lines represent the three outgas ports; press the left-hand circle - this corresponds to the outgas port on which you will attach the sample tube. The **Sample Information** screen is displayed, Figure 5.6. Edit the **Sample Information screen**:

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a. The only field that *must* be correct at this stage is the one on the Profile key. If 'BET5' is not displayed on this key, press the Profile key, press the button labeled 'BET5' on the **Profiles** screen, and then press OK.

b. To enter an appropriate Sample ID for this sample, press the Sample ID key, use the displayed QWERTY keypad to enter characters, then press Enter.

Note. If you use a number for the last character of the Sample ID, the Sample ID will automatically increment the number the next time you run a sample.

c. If you wish to enter an Operator name and Customer, change these fields in the same way as you did the Sample ID.

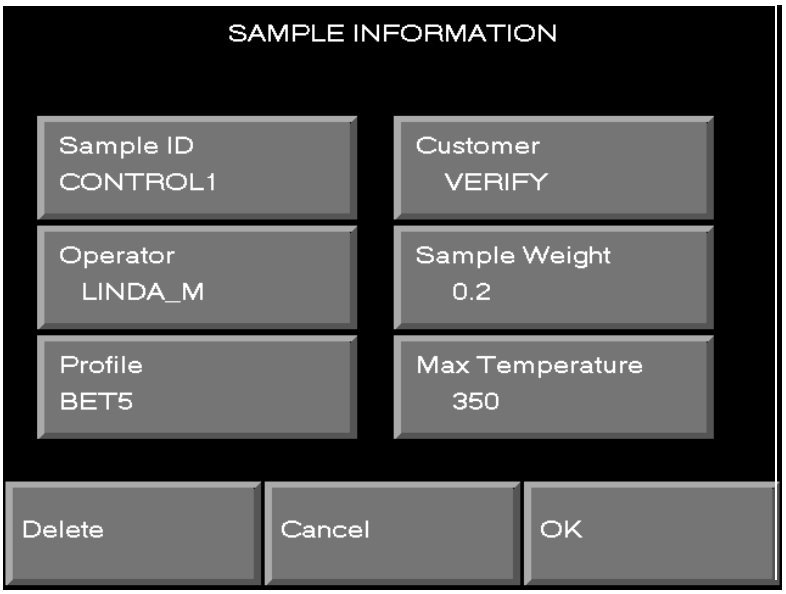


Figure 5.6 Sample Information Screen

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Outgassing the Sample

Attaching the Sample Tube to an Outgas Port

CAUTION

Do not use any tools to remove or install saturation tube or adsorption tube fittings. Fittings are designed to seal when finger-tight

1. Refer to Figure 5.7. Loosen the knurled nut on the left-hand outgas port.
2. Remove the cap from the sample tube, then insert the tube into the outgas port until it stops against the manifold.
3. Tighten the knurled nut until finger-tight.

   The tube does not have to feel rigid in the fitting to be sealed. If a gentle downward pull on the tube does not pull it out of the fitting, it is installed properly.

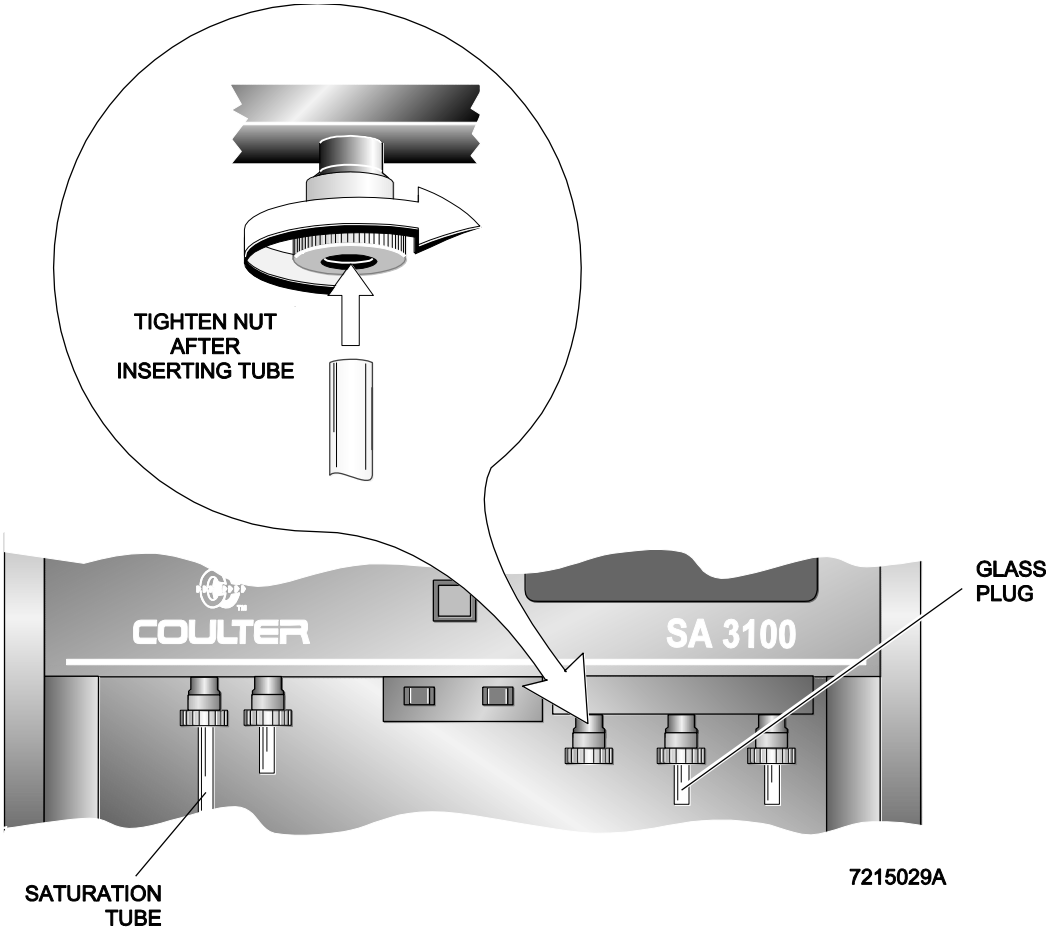


Figure 5.7 Connecting the Sample Tube to an Outgas Port

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Outgassing

1. Refer to the 'SAMPLE RUNNING INSTRUCTIONS' on the assay sheet, and note the outgas temperature and time. If the values displayed on the respective keys on the **Outgas** screen are not the same as those on the sheet, pressing each key will provide access to a numeric keypad to allow you to edit the values. After editing values, press Enter to confirm the change and return to the **Outgas** screen.

   Note. The outgas temperature and time displayed on the **Outgas** screen are in Centigrade and minutes, respectively.

2. Press Start on the **Outgas** screen. This initiates the outgassing procedure. For 60 minutes of outgassing at 300º C, the entire procedure takes approximately 1 h and 15 min. When outgassing is complete, the message *Outgassed* is displayed below the graphic for the outgassing port used. In addition, the following message is displayed at the bottom of the **Outgas** screen:

   REMOVE SAMPLES
   Press OK when removed

3. Remove the sample tube from the outgas port:

   a. Have a sample tube cap ready.

   b. Loosen the knurled nut and pull the tube out of the fitting.

   c. Promptly place the cap on the sample tube (the tube is filled with nitrogen, so waiting too long will allow the gas to mix with air).

   d. Press OK.

The sample is ready to be weighed and analyzed.

Weighing the Outgassed Sample

The outgassed sample weight is the weight used to determine the analysis results, and is therefore critical.

1. Weigh the tube (including the tube insert and sample) on the analytical balance to at least 0.001g precision. Remove the cap just before weighing and promptly replace it after weighing. Make sure the following conditions are met before weighing the sample tube after outgassing:

   - The sample tube is at room temperature.
   - The sample tube is clean and without fingerprints. Use a lint-free laboratory tissue to remove fingerprints.
   - The weighing pan on the analytical balance is clean.

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- The sample tube is not charged with static electricity. Use an anti-static device to discharge the tube.

2. Subtract the weight found earlier for the tube assembly without sample to determine the outgassed sample weight, and make a note of the result.

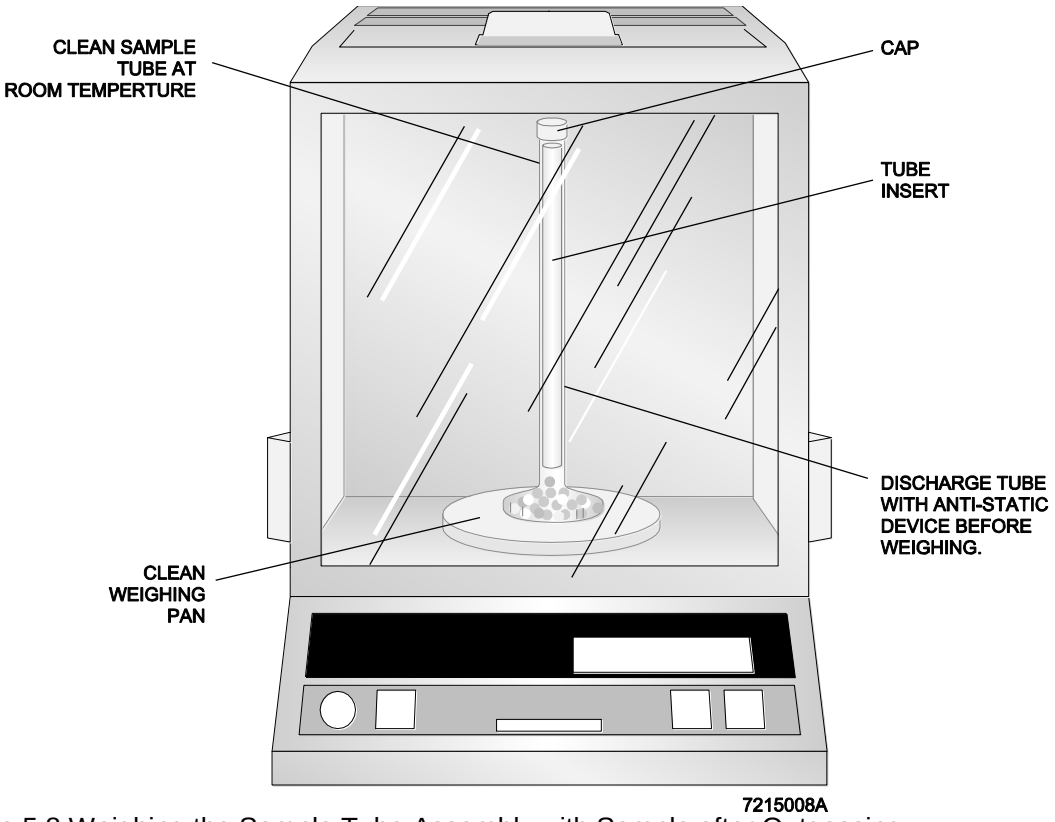


Figure 5.8 Weighing the Sample Tube Assembly with Sample after Outgassing

Analyzing the Sample

Your control sample is now ready for analysis.

IMPORTANT

Do not load the sample tube onto the analysis port until instructed to do so by a message on the touch screen. Doing so may compromise analysis results.

Selecting The Sample From The Outgassed Samples List

1. Press the Analysis key to move to the **Analysis** screen.

2. Press the Next Analysis key. The **Outgassed Samples** screen is displayed, see Figure 5.9.

3. Select the control sample by pressing the key marked with the Sample ID you entered, then press OK. The **Sample Information** screen is displayed.

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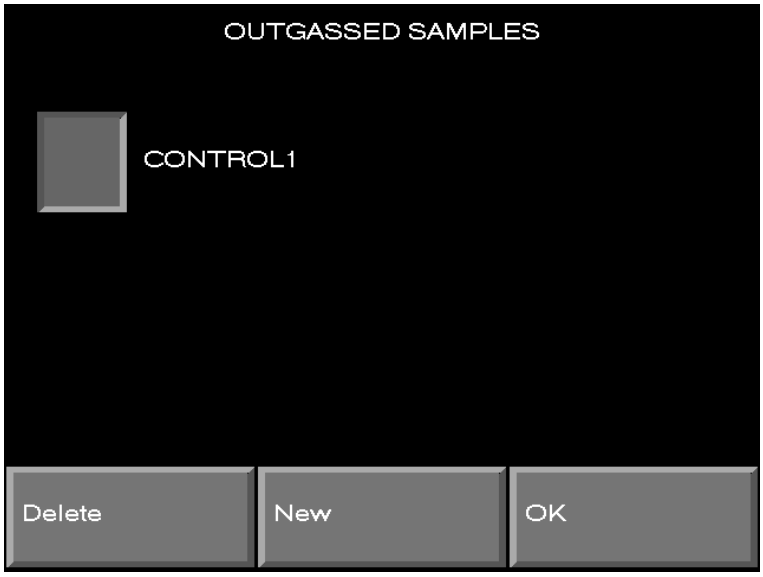


Figure 5.9 Outgassed Samples Screen

Entering the Outgassed Sample Weight

1. Enter the exact sample weight in the sample weight field (from step 3 in Weighing the Outgassed Sample, above):

   a. Press the Sample Weight key.

   b. Enter the sample weight on the numeric keypad and press Enter.

2. Press OK on the **Sample Information** screen to confirm entries and return to the main **Analysis** screen. The following message is displayed on the screen:

   LOAD SAMPLE
   Press OK when loaded

Loading the Sample

1. Attach the sample tube to the analysis port:

   a. Refer to Figure 5.10. Loosen the knurled nut on the analysis port.

   b. Remove the cap from the sample tube, then promptly insert the tube into the port until it stops against the manifold.

   c. Tighten the knurled nut until finger-tight.

      The tube does not have to feel rigid in the fitting to be sealed. If a gentle downward pull on the tube does not pull it out of the fitting, it is installed properly.

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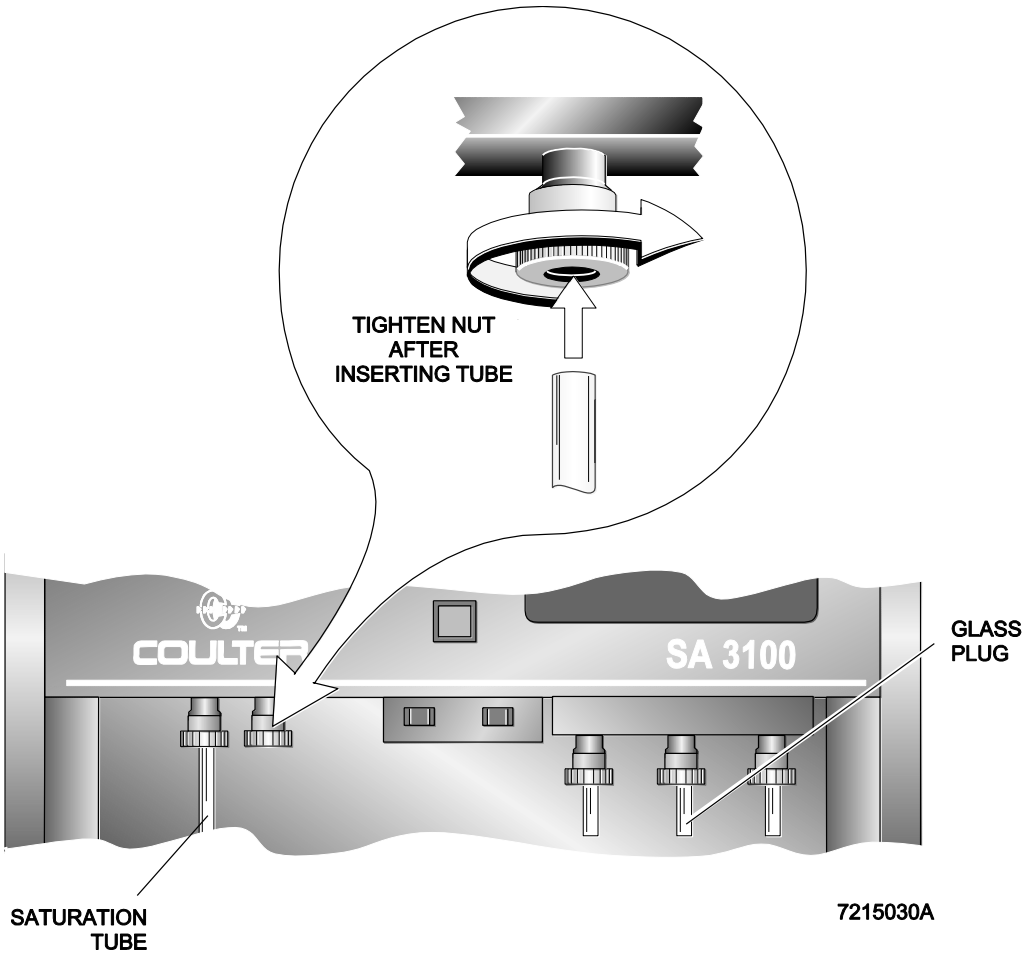


Figure 5.10 Attaching Sarnple Tube to Analysis Port

2. Press **Start**. The Status message on the screen reads

   WAIT
   Sample Evacuating.

Filling and Loading Liquid Nitrogen Dewar

WARNING

Use proper safety equipment when handling liquid nitrogen, including, but not limited to, safety glasses and low temperature thermal gloves

WARNING

Always temper new dewars by adding a small quantity of cryogen and swirling the liquid in the dewar before filling. This reduces the risk of implosion and injury.

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Figure 5.1 1 Adding Liquid Nitrogen to Dewar

1. If the dewar is new, temper the dewar by swirling a small quantity of liquid nitrogen in the dewar.

2. Add liquid nitrogen to the dewar until it is half-full.

IMPORTANT

Do not load dewar into the dewer rack until instructed to do so by a message on the touchscreen. Doing so will condense nitrogen onto the sample, causing low surface area results.

---

3. When the message

   LOAD DEWAR
   Press Start Analysis when loaded

   appears, load the half-full dewar into the dewar rack, see Figure 5.12.

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CAUTION

To avoid breaking glassware, do not force a dewar into the dewar rack if it is difficult to install. Make sure the top of the dewar is free of ice and try again.

a. Slide the dewar up over the sample and saturation tubes, inserting the top of the rear dewar wall behind the upper tab in the dewar rack.

b. Move the bottom of the dewar back into the rack, then slide the dewar down so that the bottom fits over the lower tab in the dewar rack.

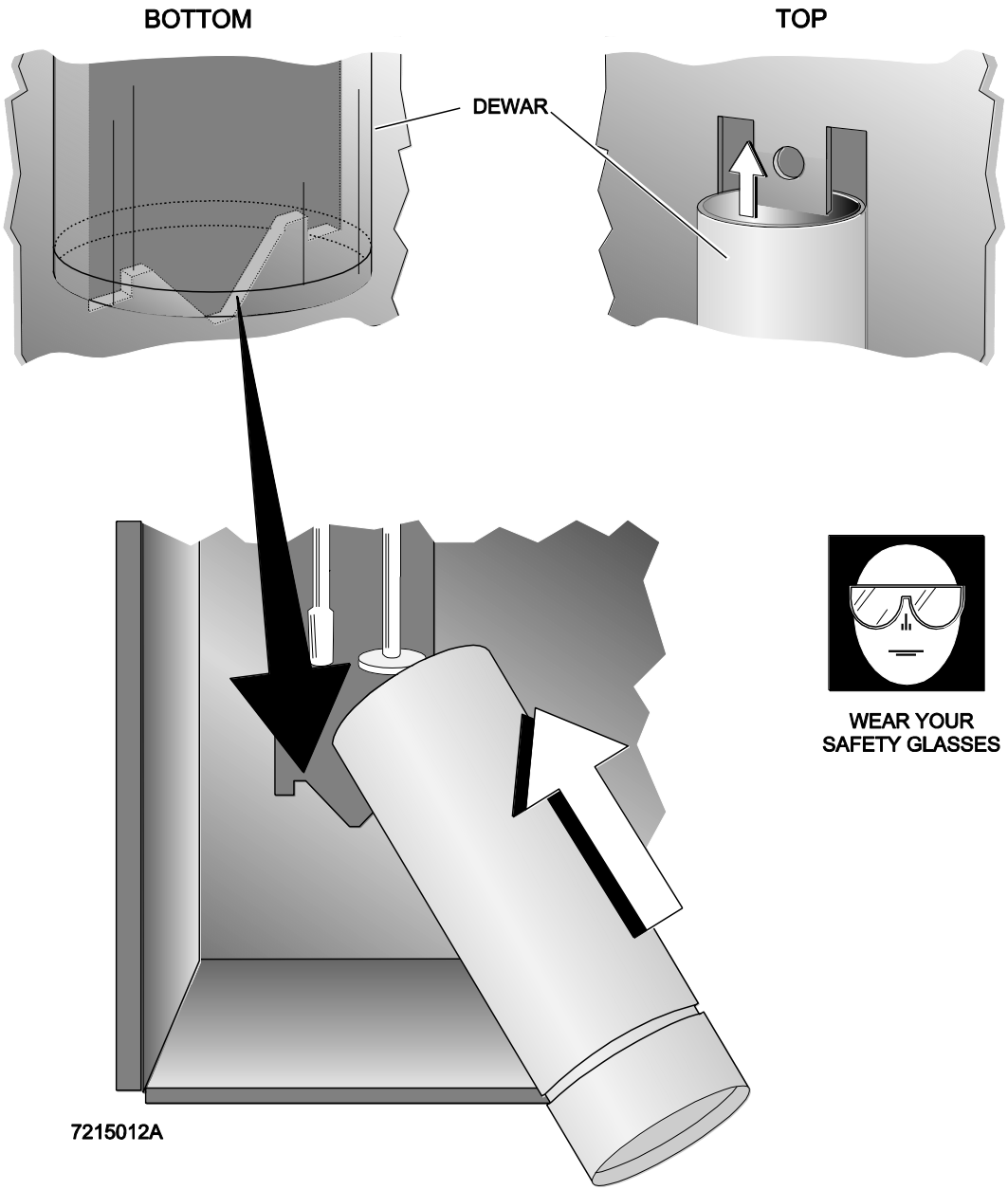


Figure 5.12 Installing the Dewar.

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IMPORTANT

Do not fill the dewar above 2 cm from the top. Doing so will cause temperature changes due to rapid evaporation and may cause inaccurate results.

4. Using the dewar funnel fill the dewar to within 2-3 cm of the top with liquid nitrogen, see Figure 5.13.

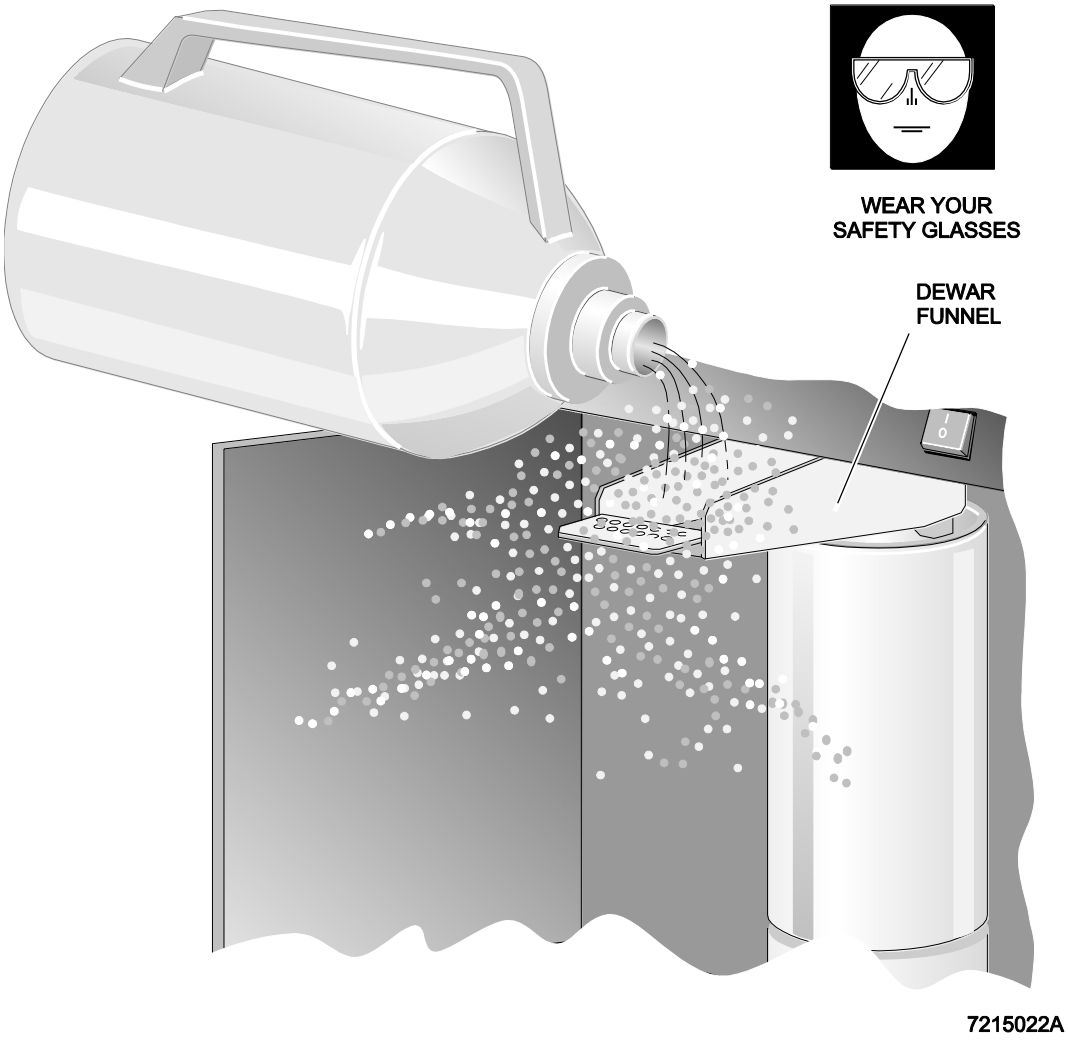


Figure 5.13 Filling the Dewar Using the Dewar Funnel

5. Install the insulating dewar lid, see Figure 5.14.

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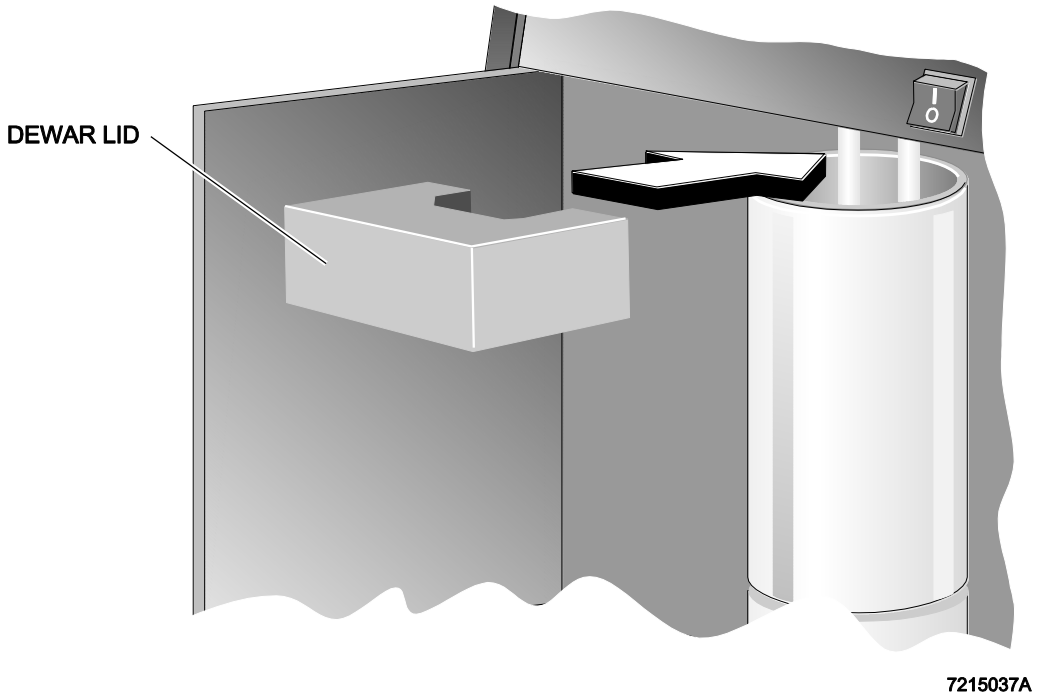


Figure 5.14 Installing Dewar Lid

6. Press **Start Analysis**. The sample analysis proceeds automatically.

During Analysis

Sample analysis takes approximately 20 min. The instrument automatically completes the following steps during analysis.

- Freespace measurement. After you press **Start Analysis**, the SA 3100 analyzer performs the freespace measurement, to account for the volume in the tube and accurately measure the quantity of gas adsorbed. The freespace data is displayed graphically on the screen during the procedure.

- Isotherm measurement. When the freespace measurement is complete, the instrument proceeds to the isotherm measurement, from which the specific surface area of the sample is calculated.

At the completion of analysis, results are displayed on the screen for approximately 2 min. Results are then temporarily removed in order to complete the necessary procedure for sample tube removal.

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After Analysis

WARNING

Do not remove the dewar until instructed to do so by a message on the touch screen. Doing so may cause explosive pressures to develop in the sample tube.

The dewar and sample tube must be removed after analysis using the following procedure. After the tube is removed, final results are displayed on the screen and printed if a printer is configured and attached.

1. The instrument automatically evacuates the sample tube in preparation for removing the dewar. When this evacuation is complete, the following message appears

   REMOVE DEWAR
   Press OK when removed.

2. To remove the dewar:

   a. Remove the insulating dewar lid.

   b. Lift the dewar up to release it from the lower tab of the dewar rack.

   c. Slide the dewar down and out.

CAUTION

Do not remove the sample tube until instructed to do so by a message on the touch screen. Doing so may reduce the lifetime of the vacuum system.

3. Press OK. The sample tube will now be automatically backfilled with gas. The following message appears

   WAIT
   Backfilling sample

   Backfilling takes approximately 2 minutes.

4. When backfilling is complete, the following message appears

   REMOVE SAMPLE
   Press OK when removed

   Remove the sample tube (simply loosen the knurled nut while holding the tube - then pull out the tube) and then press OK.

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5. The following message appears

   IS SAMPLE WEIGHT OK?
   [entered weight] g,

   YES and NO keys are displayed to allow you to correct any clerical errors made in entering the sample weight, or enter the outgassed weight if you neglected to enter it prior to analysis.

   - If the sample weight displayed is correct, press YES.
   - If the sample weight displayed is incorrect, press NO. A numeric keypad appears allowing you to enter the correct sample weight.

   Results are printed if a printer is installed and configured (see heading 3.5, Printer Installation).

5.3 REVIEWING THE RESULTS

When the result screen appears, see Figure 5.15, check that the reported specific surface area value is within the specified limits on the assay sheet for the control sample. If your specific surface area is outside the limits in the assay sheet, contact your Beckman Coulter representative.

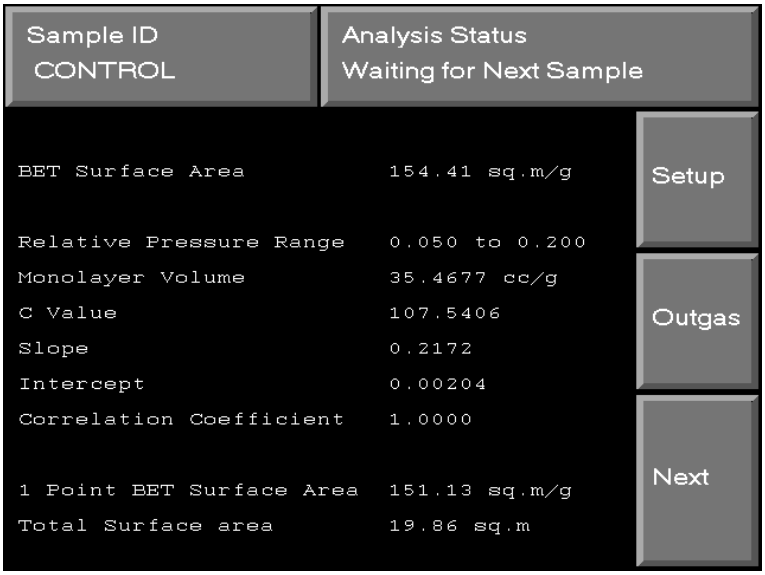


Figure 5.1 5 Specific Surface Area Result Screen

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6.1 BASIC CONCEPTS

Introduction

The measurement of surface area and pore size distributions may be performed in several different ways. Gas sorption is the most widely used and accurate method for total surface area measurements and pore sizes within the approximate range of 0.4 to 200 nm diameter. This method provides very high resolution data and has very wide applicability

The Gas Sorption method is used by the Coulter SA 3100 analyzer. Nitrogen and other inert gas adsorbates may be used. The method may be defined simply as the physical characterization of material structures using a process where gas molecules of known size are condensed (or adsorbed) on unknown sample surfaces. The quantity of gas condensed and the resultant sample pressure are recorded and used for subsequent calculation. This data, when measured at a constant temperature, allows an isotherm to be constructed. Isotherm data is then subjected to a variety of calculation models to obtain surface area and pore size distribution results. The BET calculation, for example, is one mode1 used for the determination of the sample specific surface area; the BJH calculation yields the pore size distribution.

The following sections introduce the concepts and terminology used in the Measurement of physisorption isotherms.

Adsorption or Absorption

Materials such as powders and solid pieces of inorganic and organic compounds and mixtures are capable of retaining amounts of gases and vapors within their structure. This may occur via a process of absorption or by a process of adsorption. Absorption may be understood by visualizing the material retaining the gas, vapor or liquid within the body of the material, such as when a fabric is dyed. In contrast, the process of adsorption is one of attachment to the surface of a material. Activated carbon adsorbs cooking fumes. Adsorption is often a totally reversible process.

Physisorption or Chemisorption

If we consider the process of adsorption as one where adsorbate molecules are attached to the surface of a material, these molecules may be retained by either physisorption or chemisorption forces. The difference between these two mechanisms may be summarized in the following ways:

- Physisorption is a readily reversible process at the temperature of adsorption whereas chemisorption is irreversible at the adsorption temperature.

- Chemisorption is a chemical bonding process whereas hysisorption is resulting from a physical attraction.

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- Chemisorption involves much higher energies of adsorption than does physisorption. Chemisorption energies are generally due to covalent bonding, whereas physisorption energies are generally smaller in value and are due to a process of condensation.

The adsorption of nitrogen and other gases such as argon and krypton used in the SA 3100 analyzer may be characterized as physisorption processes. The Coulter SA 3100 analyzer assumes that all adsorption detected is due to physically adsorbed gas. No calculation models are provided to account for adsorption due to a chemisorption processes.

The Adsorption and Desorption Isotherms

The surface area and pore size distribution of a sample is calculated from all or part of the adsorption and desorption isotherms. The SA 3100 Plus analyzer will measure both of these isotherm branches. The adsorption isotherm is an incremental set of data which describes how much adsorbate gas condenses on to a material at a given pressure and at a constant temperature. The volume of gas adsorbed is reported at STP conditions (Standard Temperature and Pressure). The quantity is reported by the SA 3100 analyzer as volume in units of cc/g. Desorption is the reverse process and is a decremental set of data reported in the same units.

Isotherm data is unique to a given analysis temperature because adsorptive pressures Vary with temperature. Data measured at different temperatures is easier to compare if the quantity adsorbed (Y-axis) is plotted against the sample relative pressure (X-axis) rather than the sample pressure. If data obtained at different temperatures is plotted against the relative pressure the resultant isotherms will be very similar, but not identical due to the density difference of nitrogen packed within the pores of the material.

Relative Pressure

As gas is being adsorbed on the sample surface not all the molecules are adsorbed. Those that are not adsorbed form a residual pressure in the sample chamber. This value of sample pressure divided by the saturation vapor pressure gives the relative pressure. This value may be considered to be the degree to which the sample is saturated with semi-liquid adsorbate. The range of relative pressure values measurable with the SA 3100 analyzer Vary within the range from O to 0.995.

Saturation Vapor Pressure

For accurate isotherms it is necessary to know precisely what the saturation vapor pressure of the adsorbate gas is during the analysis. The saturation vapor pressure may be considered to be the boiling pressure of the liquid gas. It varies with temperature. In the typical analysis setup liquid nitrogen is used for the sample coolant bath. The bath temperature will change with the atmospheric pressure and the purity of the nitrogen used. In an open container atmospheric gases will continually condense in the liquid nitrogen and so contaminate the liquid. The level of contamination is unknown unless the temperature or saturation pressure

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is monitored. It is therefore important for the most accurate results, that the saturation vapor pressure is measured throughout the duration of sample analysis.

Saturation Vapor Pressure

These three similar words may be defined in the following way. Adsorptive is the gas phase in the sample tube and elsewhere, Adsorbate is the condensed gas phase on the sample surface, and Adsorbent is the sample.

Measurement Method for Adsorption and Desorption Isotherms

The SA 3100 analyzer uses the static fully equilibrated volumetric method. In this method discrete data points are obtained and gas pressures are equilibrated before readings are recorded. The volume of adsorbate gas retained by the sample is calculated from the measured pressures at each data point. The volume of each dose of gas is constant and has been calibrated at the factory. The volume of the sample tube not occupied with the sample is an unknown volume at the start of the analysis. Helium gas is used to measure this volume, which is called the free space. Specific details about the calculation of the free space and the volume of gas adsorbed is given in Section 6.2.

This is considered the classic method for gas adsorption. When first developed it was manual in operation, slow and operator intensive. The SA 3100 analyzer uses an automated version of this method. It has been optimized for speed and accuracy. With this Static Equilibrated Method, discreet data points are taken and each is equilibrated to pre-defined limits to ensure the best results are obtained. High resolution results may be obtained with a large number of data points, or for rapid surface area results as few as three data points may be measured in just a few minutes.

6.2 THEORY

This section describes the basic theory of the measurement of adsorption and desorption isotherms. It also outlines the calculation models which are used in the SA 3100 analyzer. A fully detailed treatment of these calculations is not given. The interested reader is encouraged to refer to the original publications in the References section. Further background can be obtained from any one of the excellent text books which are available. For two of the best examples, see the heading For Further Reading, at the end of this chapter.

Measurement and Calculation of Isotherm Data

The adsorption process is measured volumetrically with a static fully equilibrated procedure. From just a few to more than a hundred individual data points form all or part of the adsorption and desorption isotherms. The isotherm volume data (Y-axis or ordinate) is calculated by subtracting the freespace of the sample tube, which is that volume of the sample tube not occupied by the sample, from the total volume of gas dosed to the sample. An incremental data set is formed. Each data point is processed by calculating the volume adsorbed and measuring the

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sample pressure which is then divided by the saturation vapor pressure. This relative pressure value is recorded as the X-axis or abscissa values.

The ideal gas law is used for the calculation of both the freespace and the volume of adsorptive dosed. The quantity of a gas in moles n, dosed into the measurement manifold is given by Equation 6.1, where P_M, V_M and T_M are pressure, volume and temperature of the dose manifold, and R is the Gas Constant.

$$P_M V_M = nRT_M \qquad (6.1)$$

Freespace determination

Helium gas is used to measure the freespace of the sample tube. The freespace is the volume in the sample tube and therefore it vanes with the volume occupied by the sample itself. It is an important measurement if accurate volumetnc data is required.

The volume of helium dosed to the sample tube is calculated after opening and closing the sample valve. The volume of the sample tube, or freespace, may be found from the resultant pressure drop.

The dose of gas may be described with the following material balance equation:

$$\frac{P_{M1}V_M}{T_M} = \frac{P_{M2}V_M}{T_M} + \frac{P_{M2}V_S}{T_S} \qquad (6.2)$$

where V_S and T_S are the volume and temperature of the sample and sample tube, V_M and T_M are the volume and temperature of the manifold, and P_{M1}, and P_{M2}, are the pressure of the manifold before and after the dose of gas, respectively

In the above example, no consideration is given to the temperature or the volume of the intermediate space above the level of the liquid nitrogen and below the manifold volume. This intermediate volume varies with the sample tube, room temperature and also the level of the liquid nitrogen. The freespace value will change, (or at least appear * to) over the course of a long measurement as the liquid nitrogen level drops due to its evaporation. The SA 3100 analyzer minimizes inaccuracies caused by depletion of the liquid nitrogen during a long analysis.

$$Vd_n = \frac{(P_{M1} - P_{M2}) \times V_M}{T_M} \times \frac{273.15}{760} + Vd_{n-1} \qquad (6.3)$$

where:
- Vd_n = Volume dosed
- P_{M1} = Initial manifold pressure
- P_{M2} = Final manifold pressure
- V_M = Manifold volume
- T_M = Manifold temperature
- Vd_{n-1} = Volume dosed from previous data point
- 273.15/760 = Standard temperature and pressure conversion

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Three helium data points are measured to ensure accuracy These are measured at incremental pressures. The helium freespace correction is obtained from the linear plot of volume dosed vs. sample pressure. The slope of this line is equivalent to the volume of the sample tube per unit of sample tube pressure. The helium freespace determination is used in the calculation of the volume adsorbed (see the next section).

Determination of Volume Adsorbed

After the helium freespace measurement has been completed, the adsorption isotherm is measured. The volume of adsorptive dosed to the sample is calculated in the same way as for the helium freespace dose (see equation 6.4). The volume of gas adsorbed by the sample is calculated for each measured data point by subtracting the volume of gas present in the freespace from the volume of the adsorbate dosed to the sample:

$$Vads_n = Vd_n - (P_{Sn} \times slope + intercept) \qquad (6.4)$$

where:

Vads,	=	Volume adsorbed
Vd_n	=	Volume dosed
P_{Sn}	=	Sample pressure
slope	=	Freespace measurement slope
intercept	=	Freespace measurement intercept

Explanation of Isotherm Pressure Tables

The isotherm can comprise a varied number of data points. All or part of the adsorption and desorption branches can be measured according to requirements. Selection of one or more calculations such as BET, Langmuir and BJH determine how many data points are needed. A master relative-pressure table contains a detailed set of both adsorption and desorption data points. Each specified calculation has certain data points associated with it which are determined from the profile settings. A target pressure table is built-up from the calculations specified. For example, if just BET is chosen only the first part of the isotherm up to a relative pressure of 0.2 is needed. The target pressure table then comprises data points from vacuum to 0.2 Ps/P, Similarly, if BET surface area and BJH desorption calculations are chosen a complete adsorption and desorption isotherm must be performed. The number of adsorption data points performed is determined from other calculations chosen (such as the requirement for T-Plot data). If only BET data is required then the minimum number of adsorption data points are obtained.

BET Surface Area

The BET (Brunauer, Emmett and Teller) calculation was first introduced in 1938' and has continued to gain popularity ever since. It is the most commonly used calculation method for the characterization of Specific Surface Area. The BET surface area (which includes all internal structure) is calculated from a multilayer adsorption theory. The theory is not detailed here but is based upon the assumption that the first layer of molecules adsorbed on the surface involves adsorbate-adsorbent energies, and subsequent layers of molecules adsorbed involve the energies of vaporization (condensation) of the adsorbate-adsorbate interaction.

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The BET equation should produce a straight line plot, the linear form of which is most often represented as:

$$\frac{P_S}{V_A(P_0-P_S)} = \frac{1}{V_M C} + \left[\frac{C-1}{V_M C}\right] \times \frac{P_S}{P_0} \qquad (6.5)$$

where:
- V_M = volume of monolayer
- V_A = volume adsorbed
- P_S = sample pressure
- P_O = saturation pressure
- C = constant related to the enthalpy of adsorption

A specified relative pressure range (usually 0.05 to 0.2) is chosen and the isotherm data is used to calculate the BET function, which is plotted against relative pressure (P_S/P_O), to give a straight line having a slope $(C-1)/V_m C$ and intercept $1/V_m C$.

The BET surface area in (m^2/g) is then determined from the following expression:

$$S_{BET} = \frac{V_M \times N_A \times A_M}{M_V} \qquad (6.6)$$

where S_{BET} is the BET surface area, N_A is Avogadros number A_M is the cross sectional area occupied by each adsorbate molecule, and M_V is the gram molecular volume (22414 mL). For nitrogen BET determinations, the cross-sectional area of the adsorbate molecule is assumed to be 0.162 nm^2.

Choosing BET Parameters

The operator chooses the number of BET data points, specified in the Profile setup for the calculation. The standard choices are 3,5,5 LOW, 10, and 10 LOW Choosing a 'LOW option acquires data and calculates BET surface area at a low relative pressure range (0.005 - 0.05). These options should be used for microporous materials. If a microporous material is measured with the non-low options, negative intercept and C-values are obtained, the correlation coefficients may be low, and results may be incorrect.

When measured using the UNKNOWN Sample Category (see heading 7.4, Profiles, for a discussion of Sample Categories), the 3-point, 5-point and 10-point options may produce similar numbers of measured data points, and usually a larger number of points than required for the calculation, but the number of interpolated data points used for the calculation will correspond to the chosen option. If, however, a standard isotherm has been measured for the material, and that standard is selected as the Sample Category, the instrument makes measurements at approximately the specified number of data points. The measured data is listed in the Analysis Data section of the Surface Area Report, and the specified number and location of the BET points are listed in the Interpolated Data section.

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Langmuir Surface Area

The Langmuir surface area calculations are applicable to adsorption isotherms data which have Type 1 characteristics. A Type 1 isotherm is obtained if the sample is a microporous material and is therefore characterized by a rapid rise in the volume adsorbed at very low relative pressures values. The main assumption with the Langmuir calculation is that only a monolayer is formed. The expression used is:

$$\frac{P_S}{V_A} = \frac{1}{bV_M} + \frac{P_S}{V_M} \qquad (6.7)$$

where V_M is the volume of the monolayer, P_S is the sample pressure, b is the Langmuir constant, and V_A is the volume adsorbed.

A linear plot of P_S/V_A vs. P_S or P_S/Po produces the monolayer volume V_M. The surface area may then be calculated from:

$$S_{Langmuir} = \frac{N_A \times A_M \times V_M}{M_V} \qquad (6.8)$$

where $S_{Langmuir}$ is the Langmuir surface area and M_V is the gram molecular volume (22,414 ml).

Choosing Langmuir Parameters

The operator chooses the number of Langmuir data points, specified in the Profile setup for the calculation. 5-point and 10-point options are available. As with the BET calculation, when measured using the UNKNOWN Sample Category (see heading 7.4, Profiles, for a discussion of Sample Categories), the 5-point and 10-point options may produce similar numbers of measured data points, and usually a larger number of points than required for the calculation, but the number of interpolated data points used for the calculation will correspond to the chosen option. If, however, a standard isotherm has been measured for the material, and that standard is selected as the Sample Category, the instrument makes measurements at approximately the specified number of data points. The measured data is listed in the Analysis Data section of the Surface Area Report, and the specified number and location of the Langmuir points are listed in the Interpolated Data section.

t-Plot Method

The t-Plot method is used for the estimation of micropore volume and micropore area. It provides a way of characterizing and sizing the pore structure of a material.

Data directly available from the t-Plot calculation are the surface area of the meso and macropores, and the micropore volume. It may be used in conjunction with the BET surface area to calculate the micropore surface area.

The generally accepted definitions of the pore size regions are:

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Micropores: <2 nm
Mesopores: 2 to 50 nm
Macropores: >50nm

Calculation Method

The volume of gas adsorbed is plotted against the adsorbate molecular film thickness. This produces the t-Plot (thickness-plot) which may be used for qualitative as well as quantitative estimation of sample porosity The thickness of the film is calculated from either the Halsey calculation, Equation 6.9, or from the Harkins and Jura calculation, Equation 6.10.

$$t = \left| \frac{13.99}{\log \frac{P_0}{P_s} + 0.034} \right|^{1/2} \quad (6.9)$$

$$t = 3.54 \times \left| \frac{5}{2.303 \times \log \frac{P_0}{P_s}} \right|^{1/3} \quad (6.10)$$

Volume adsorbed data is plotted against the calculated film thickness. This "t-plot" data is then used to calculate the Y-intercept value which when converted from a gas volume to a liquid volume gives the micropore volume. The slope of the linear section is used to calculate the meso / macro pore surface area. The equations used for the micropore volume and the mesopore surface area are:

$$\text{VOLUME}_{\text{MICROPORE}} = (0.001547)(\text{t-Plot intercept}) \quad (6.11)$$

$$\text{SURFACE AREA}_{\text{MESOPORE}} = 1547(\text{t-Plot slope}) \quad (6.12)$$

The t-Plot calculation may also be used to provide the Micropore Surface Area value. This is determined from the BET surface area and the mesopore area (6.12) as follows:

$$\text{SURFACE AREA}_{\text{MICROPORE}} = \text{SURFACE AREA}_{\text{BET}} - \text{SURFACE AREA}_{\text{MESOPORE}} \quad (6.13)$$

Interpretation of data

The t-Plot graph gives information about where the pores (if any) are. It can also be used to assess whether pores are micropore or mesopore sized.

If the sample is non-porous then the thickness of the adsorbate film will be proportional to the volume adsorbed, producing a straight line which will pass through the origin of the t-Plot, as in Figure 6.1 A.

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If the t-Plot does not pass through the origin, the presence of micropores is indicated, as in Figure 6.1 B. The magnitude of the Y-axis intercept yields the micropore volume.

If the T-Plot deviates from a straight line at higher thickness values this indicates the presence of mesoporosity, as in Figure 6.1 C. Conversely, if the plot is straight no mesopores are indicated.

A mixture of micropores with mesopores gives a plot like Figure 6.1 D, which is a combination of the plots in Figures 6.1 B and 6.1 C.

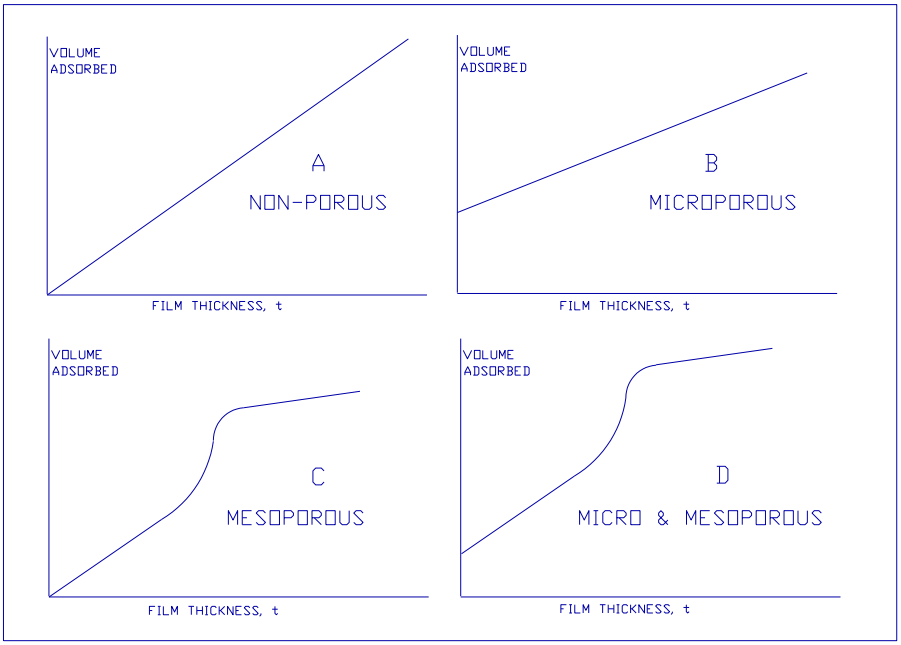


Figure 6.1 Idealized V-T Plots .

Selection of t-Plot Resolution, Range and Other Parameters

Resolution
The resolution option allows the operator to determine how many t-plot data points are presented. Approximately 10, 20 and 30 data points are obtained with the settings Low, Normal and High respectively This option does not always Vary the number of data points measured, but affects the number of interpolated data points output. Choose the resolution based upon your needs: do you which to compare specific data points? If not, then a setting of Normal is satisfactory for most material. All settings should give the same t-plot data, although it is possible to miss come of the detail if low resolution is chosen. This is because the calculation is performed over the specified t-plot range rather than from the raw data. There may or may not be the same number of raw data points, depending upon the isotherm characteristics.

T-Plot range
The t-plot range specifies the range over which the calculation is performed. This calculation should be performed over a linear section of the data set, which is volume adsorbed in cc/g at STP on the y-axis and Film Thickness in nm on the x-

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axis. Choose the higher thickness range for carbon or other samples which have a wide range of pore sizes from the microporous through the meçoporous range. The default selection is a film thickness setting of 0.35 to 0.5 nm.

Calc BET range
The BET calculation is performed as a subroutine of the t-Plot calculation. The BET surface area value may be considered to be the total surface area of the sample. If this value is not obtained, the micropore area cannot be calculated. The operator must choose the range for the BET calculation. This choice should be made based upon the amount of micropores present in the sample. If the sample has a significant quantity of micropores, use the Low BET range setting of 0.005-0.05 Otherwise use a normal setting of 0.05-0.2. If you also request a BET calculation, then use the same value, although they are marked with LOW (or not), instead of with the numeric ranges.

Diagnosing Problems with T-Plot Data

Most problems with t-plot data are encountered with the selection of the range for the t-Plot calculation. To obtain good results, select a range over which the t-Plot is linear. The correlation coefficient should be very close to the value 1 if the correct range is chosen. If the t-Plot is non-linear, as indicated by a correlation coefficient of less than approximately 0.995, use a higher calculation range.

Most problems with t-plot data are encountered with the selection of the range for the t-Plot calculation. To obtain good results, select a range over which the t-Plot is linear. The correlation coefficient should be very close to the value 1 if the correct range is chosen. If the t-Plot is non-linear, as indicated by a correlation coefficient of less than approximately 0.995, use a higher calculation range.
It is also possible to obtain slightly negative values for the micropore volume or micropore area for samples which have no micropores. This results from the selection of a non-optimum calculation range. If this is the case, select a higher calculation range. Sometimes it is difficult with some samples which have a wide pore size range, (for example, carbon) to find a linear section of the t-Plot for the calculation. If the standard options in the t-Plot calculation do not suffice, contact your Beckman Coulter Representative and ask for the 'SA 3100 Advanced Configuration Kit'.

Pore Size Distributions

This section covers the calculation of pore size distributions from nitrogen isotherms. A cylindrical pore model is assumed, which further assumes open-ended pores and the absence of pore networks.

The pore size distribution is obtained by the analysis of either the adsorption or desorption isotherm branches

The method used is based on the BJH (Barrett, Joyner, and Halenda) method which 4 involves the area of the pore walls, and uses the Kelvin equation to correlate the relative pressure of nitrogen in equilibrium with the porous solid, to the size of the pores where capillary condensation takes place. A detailed theoretical study of this and other aspects of gas sorption may be found in

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Adsorption, Surface Area and Porosity, 2nd Ed. Gregg, S.J. and Sing, K.S.W., Academic Press, 1982. This is highly recommended reading material.

Mesopores within a solid adsorbent are filled progressively with adsorbate by the process of capillary condensation. Increasing numbers of molecular layers condense within increasing size pores until at P/P of near unity, all mesopores and macropores are full of liquid adsorbate.

The desorption isotherm is constructed by plotting the volume desorbed per gram of sample against the relative pressure, P/P. The procedures used to calculate the pore size distribution are described below.

The volume desorbed from the sample at different decreasing relative pressure values are converted to equivalent liquid volumes, because it is assumed that capillary condensation has taken place and the pores are filled with liquid rather than gas. The liquid volume is obtained by multiplying the gas volume by 0.00156 (a conversion from gas at STP to liquid).

The Kelvin equation is used to calculate the core radius of the liquid in the capillary:

$$RT \ln\frac{P_S}{P_0} = -2\gamma\frac{V_M}{R_K} \qquad (14)$$

Where:

R_κ = Kelvin radius
γ = adsorbate surface tension at T, in mN/m,
R = the gas constant
T = boiling point of nitrogen
V_M is the molar volume of nitrogen.

Substituting for the values of the different constants and solving for Rk, we obtain:

$$R_K(\text{angstroms}) = 4.14 \log\frac{P_S}{P_0} \qquad (15)$$

The thickness of the nitrogen film adsorbed on the walls of the pore at a given relative pressure is calculated with the modified Halsey equation:

$$t\ (\text{angstroms}) = 3.54 \times \left[\frac{5}{2.303 \times \log\frac{P_0}{P_s}}\right]^{1/3} \qquad (16)$$

The actual radius of the pore, R_p, is found by the addition of the film thickness, t to the P Kelvin radius, R_K.

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The values of the different constants needed to calculate the pore size distribution parameters are given below:

Surface tension of nitrogen	=	8.855 mN/m
Molar Volume, V_M	=	34.6 cm^3 mole
Normal boiling point of N_2 T	=	77.3 K
Ideal gas constant, R	=	8.31 * 10^7 erg/ moleK
		Conversion factor for gas
STP to liquid volume, C	=	0.00156

Choosing BJH Parameters

Selection of Resolution
The number of BJH pore size data points is selected with the Resolution option. The choices, LOW, MED and HIGH correspond to approximately 25, 45 and 65 data points. The number of measured data points is not necessarily the same as the number of points in the pore size distribution.

Plot Range
Choose Autoscaling unless you require specific plots for comparison of different data sets using the same X-axis scale. Values are in nm.

Equation
Choose between Harkins-Jura and Halsey for the film thickness equation. The standard selection is Harkins-Jura

Pore Volume Calculation

The Pore Volume calculation is performed at a pre-determined relative pressure on either the adsorption or desorption isotherm branch (both may be selected). The standard choices are 0.9814 or 0.9907. To change these contact your Beckman Coulter Representative and ask for the 'SA 3100 Advanced Configuration Kit'. The data is presented as the total pore volume of all pores less than the pore size equivalent to the chosen relative pressure.

6.3 FOR FURTHER READING

The following are excellent sources for more detailed treatments of gas sorption theory:

1. Gregg SJ, Sing KSW. Adsorption, Surface Area and Porosity, 2nd Edition. Harcourt Brace Jovanovich, London, England, 1982.

2. Rudzinski W, Everett DH. Adsorption of Gases on Heterogeneous Surfaces. Harcourt Brace Jovanovich, London, England, 1992.

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7.1 OVERVIEW

The instructions in this chapter assume that you have read Chapter 4, Introducing the SA 3100 Analyzer, which discusses the basic concepts and components of the system, and describes the structure of the user interface.

Heading 7.2, SA 3100 Analyzer Procedures, gives a brief description of all the procedures that you may need in the course of standard operation of the instrument (with the exception of cleaning and maintenance procedures, which are covered in Chapter 8, Cleaning and Maintenance). The sections that follow explain each of the procedures in detail. These procedures are organized in the order of the list below, which is the typical order in which you will use them when operating the SA 3100 analyzer.

Each section begins with general information, followed by step-by-step instructions for completing the procedure.

7.2 SA 3100 ANALYZER PROCEDURES

General

Using the SA 3100 analyzer consists of performing all of the following procedures, with varying frequency. Your first sample analyses will probably involve all of these steps. Depending on the variety of your applications, however, you may need the first four listed steps only infrequently after you familiarize yourself with the instrument.

The six steps marked with an asterisk (beginning with Weigh the Sample Tube Assembly and ending with Analyze a Sample) must be completed to obtain analysis results.

Note that outgassing and analysis can proceed simultaneously and independently of one another (outgassing of a *particular* sample, however, must precede analysis).

Procedures

Configuring the Instrument

Configuring the SA 3100 analyzer involves setting the screen brightness, enabling or disabling the audible click produced when a key is pressed, and choosing the printer, if any.

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Defining or Modifying a Profile

The profile determines the types of analyses performed on a sample. If a profile does not exist for the type of analysis you wish to perform, you must modify an existing profile or create a new one. Two profiles are provided with the instrument.

Determining the Optimal Sample Quantity

Various factors determine the optimal quantity of sample for a measurement. We provide a table correlating the estimated surface area of your sample with the quantity of sample to use.

Choosing a Sample Tube

There are three types of sample tubes available for use with the SA 3100 analyzer, two of which are available in two sizes. You must decide which is appropriate for the sample you are going to analyze and the type of analysis you require.

Weighing the Sample Tube Assembly*

You must weigh a clean and dry sample tube along with the sample tube insert and make a note of the weight. The empty sample tube weight is necessary to accurately weigh the outgassed sample later.

Adding Sample to the Sample Tube*

Add the sample to the sample tube in the amount determined above.

Logging-in Samples*

You must enter the sample information, including the profile to be used for analysis, using the touch screen. If you are outgassing samples on the SA 3100 analyzer, it is most convenient to log in samples prior to outgassing, allowing the instrument to keep track of sample status throughout the analysis process. If you are using the COULTER SA-PREP six-station sample outgasser, sample information can be entered before analysis.

Outgassing Samples*

Once sample tubes have been attached to outgassing ports on the SA 3100 analyzer, the automated outgassing procedure may be initiated. For increased throughput the COULTER SA-PREP six-station sample outgasser is also available. Contact your Beckman Coulter representative for details.

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Weighing Outgassed Samples*

The sample tubes, inserts, and samples must be accurately weighed after outgassing to determine the exact sample weight.

Analyzing a Sample*

After completing the above steps, you may proceed with sample analysis. Other samples may be logged-in and outgassed while sample analysis is in progress. An analysis report in ASCII format is automatically written to the floppy disk at the completion of analysis. The file can then be read using the SA-VIEW program. See 7.14 Accessing Data and Report Files, and Appendix A, COULTER SA-VIEW Software.

*Must be completed to obtain analysis results.

Printing a Report

If a printer is installed and configured, an analysis report can be automatically printed at the completion of each analysis if you specify this option in the profile. You can print the last results obtained at any time until the unit is shutdown or another analysis has been initiated. A printout can also be obtained at any point during an analysis, consisting of the data accumulated to that point. Using SA-VIEW software, you can print reports or sections of reports for any completed analyses.

Accessing Data or Report Files

Data on the diskette can be read by a text editor in any IBM-compatible PC, or by using Microsoft Windows-based SA-VIEW software. An analysis log file is also saved onto the diskette allowing easy location of particular files.

7.3 SA 3100 ANALYZER CONFIGURATION

General

There are three settings in the SA 3100 analyzer configuration: screen brightness, key click enable/disable, and printer selection. See heading 3.5, Printer Installation, to install a printer.

The default settings are:

- Screen brightness = 75%
- Key click = on
- Printer selection = None

Changing the Configuration

To change any of the default settings:

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1. Press Setup in either the **Analysis** or **Outgas** screen, then press Configuration when the **Setup and Print** screen is displayed, see Figure 7.1.

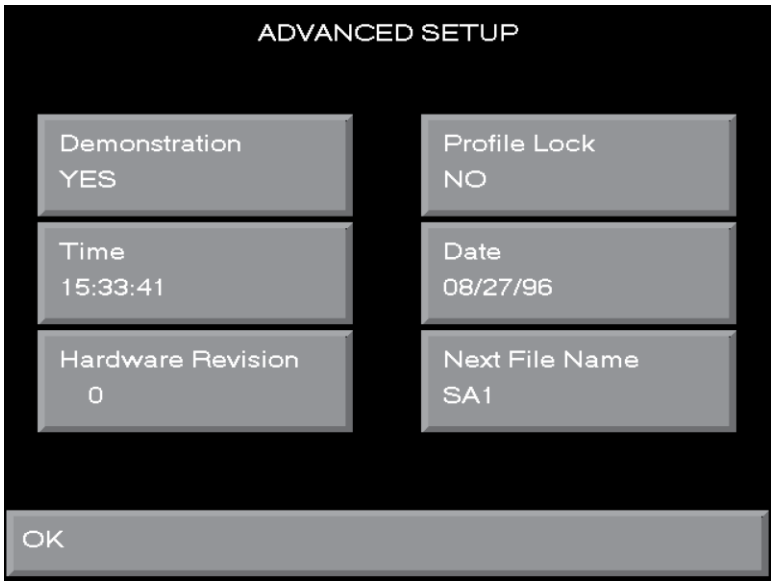


Figure 7.1 Setup and Print Screen

The **Configuration** screen appears, see Figure 7.2. Each key displays the current setting associated with that key.



Figure 7.2 Configuration Screen

2. Press the key for the setting you want to change.

Screen Brightness displays a numeric keypad allowing you to enter the new brightness setting. Settings from 50 (%) to 100 (%) are accepted.

Key Click displays the **Key Click** screen, which allows you to choose Yes or No.

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Printer displays the **Printer** screen, on which you may select the installed printer. Whether or not a report is printed automatically at the conclusion of analysis is determined by the profile.

Press OK to confirm your selection and return to the **Setup and Print** screen. Press OK again to return to the **Analysis** screen.

7.4 PROFILES

General

The profile is the most powerful tool in the user interface of the SA 3100 analyzer. Profiles define all the parameters for sample analyses, and are saved under Profile ID's in the profile list. When a sample is logged into the system prior to outgassing, the profile is one of the choices that must be made. Most users will soon build a list of profiles and will simply pick the appropriate profile each time an analysis is required. Profiles may be created or edited at any time.

Components of a Profile, below, describes the components of a profile. The procedures for editing, creating, and deleting profiles follow. For creating and editing profiles, refer to Profile Editing Keys at the end of this section which explains each key in the **Profile** and **New Profile** screens.

Fast BET Profile

The Fast BET profile is a special profile which provides up to a 40% reduction in analysis time over a normal BET measurement. Additional time saving can be achieved by using a standard isotherm for the measurement, see the Category heading under Components of a Profile, below. Some accuracy and reproducibility are sacrificed in order to accelerate the measurement using the Fast BET profile; <2% CV can be expected for normal BET measurements, but Fast BET measurements may show up to 3% CV. For details on creating a Fast BET profile, see heading Creating a Fast BET Profile, at the end of the Profiles section.

Components of a Profile

The components of a profile are as follows.

Profile ID

The Profile ID identifies the profile in the profile list. Eight characters, including spaces, can be used for the Profile ID.

Adsorbate

The adsorbate gas to be used must be selected in order to determine the correct physical constants. Nitrogen is the default adsorbate. The instrument can be

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configured to use Argon and Krypton with the 'SA 3100 Advanced Configuration Kit', contact your Beckman Coulter representative for details.

Note. Selecting the adsorbate in the profile does not physically switch the gas input. The gas line must be manually switched.

Sensitivity

The sensitivity of measurement is related to the criteria used to determine whether the sample has equilibrated after a dose of adsorbate gas. These criteria also determine the speed of analysis. Four choices of sensitivity are available: LOW, LOW (FAST), NORMAL, and HIGH.

- For high surface area materials, >200m^2/g, use LOW sensitivity.
- For Fast BET analyses, use LOW (FAST) sensitivity.
- For materials with surface areas in the range 10m^2/g to 200m^2/g, or for unknown materials, use NORMAL sensitivity.
- For materials with surface areas <10m^2/g, use HIGH sensitivity.

Sample Category

The SA 3100 analyzer can accelerate the analysis of a sample when a typical isotherm has been measured and stored for that material. The typical, or 'standard' isotherm allows the instrument to more intelligently determine the adsorbate dose sizes required to reach the P/P_0 points for a given calculation.

You can create and name one or more Sample Categories, one for each material that you intend to run with some frequency. Each category has a standard isotherm associated with it. When one of these categories is selected in the profile, the adsorbate doses during analysis will be based on the associated standard isotherm.

To create a new category, choose NEW ADS STD or NEW FULL STD (SA 3100 Plus only). NEW ADS STD measures an adsorption isotherm, and NEW FULL STD measures a complete adsorption/desorption isotherm. At the beginning of the analysis the instrument will ask you to name the category. The isotherm will then be measured and stored as the standard isotherm associated with the new category.

On subsequent runs of the material, select the category you named as the Category selection in the profile.

The sample category selections are:

- UNKNOWN: For unknown samples with no existing standard isotherm, and for which you foresee no need for a standard isotherm.
- NEW ADS STD: To create a standard adsorption isotherm. You will be asked by the SA 3100 analyzer to enter a name for the standard before the measurement begins. The standard will then enter the Category list under the name you assign,

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and can be selected for the profile that you will use to regularly analyze the material.

- NEW FULL STD (SA 3100 Plus only): To create a standard adsorption/desorption isotherm, for use if you intend to make desorption pore parameter measurements on the material. You will be asked by the SA 3100 analyzer to enter a name for the standard before the measurement begins. The standard will then enter the Category list under the name you assign, and can be selected for the profile that you will use to regularly analyze the material.

- [USER-NAMED STD]: To run an accelerated analysis using a standard isotherm for the given material which was created using one of the NEW Category selections above, and named by you.

Printer Output

The Printer Output determines whether results are automatically printed at the end of an analysis, and if so, whether a full report or a summary is printed. A full report consists of all data and calculation results from an analysis, in tabular and graphic form where applicable. A full report also begins with a report summary. A report summary consists only of the results of the calculations selected for an analysis. A report summary does not include the freespace data, the isotherm, or any graphs. For more information on printing, see heading 7.13, Printing Results.

Printer Output selections are:

- None
- Full Report
- Summary

Calculation

The calculation determines the type of analysis to be performed. Up to eight calculations can be performed on a sample at a time. There are six calculation types:

BET: BET is a surface area measurement, and requires a choice of the number of isotherm points to be used for the calculation. 3-point, 5-point, and 10-point BET calculations are available using a P/P_0 range of 0.05 to 0.2. 5-point and 10-point BET calculations are available with a LOW option. The LOW options use a P/P_0 range of 0.005 to 0.05, and are suitable for samples which exhibit Type 1 isotherms, such as microporous materials.

BJH ADSORB: BJH ADSORB is a measurement of the pore size distribution of the samples using an adsorption isotherm. Three selections are required:

- Resolution. LOW, MEDIUM, HIGH. Higher resolution increases the analysis time.

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- Equation. Harkins-Jura or Halsey, alternative equations for calculating the monolayer thickness used for pore size calculations.

- Size range. Autoscaling, 3 to 20 nm, 5 to 50 nm, 10 to 100 nm, 3 to 200 nm, in pore diameter.

BJH DESORB: BJH DESORB is a measurement of the pore size distribution of the samples using a desorption isotherm (SA 3100 Plus only). Three selections are required:

- Resolution. LOW, MEDIUM, HIGH. Higher resolution increases the analysis time.

- Equation. Harkins-Jura or Halsey, alternative equations for calculating the monolayer thickness used for pore size calculations.

- Size range. Autoscaling, 3 to 20 nm, 5 to 50 nm, 10 to 100 nm, 3 to 200 nm, in pore diameter.

LANGMUIR: The LANGMUIR calculation is an alternative method for calculating surface area suitable for samples which exhibit Type 1 isotherms, such as microporous materials. 5-point and 10-point LANGMUIR calculations are available. LANGMUIR calculations use a P/P_0 range of 0.005 to 0.5.

t-PLOT: The t-PLOT calculation is a method for estimating micropore volume and micropore area. This calculation requires four choices:

- Resolution. Low, Normal, and High.

- Calc. BET Range. The t-PLOT uses the specific surface area, so the P/P_0 range used to calculate the surface area must be chosen. Choices are: 0.05 - 0.2, and 0.005 - 0.05.

- Calc. t-PLOT Range. The range for calculation of the t-PLOT depends on the type of material: microporous, mesoporous, or a mixture. Choices are 0.03 - 0.45, 0.35 - 0.5, 0.5 - 0.7.

PORE VOLUME: The PORE VOLUME calculation is a measure of the total volume of pores on the sample with diameters less than or equal to a given diameter, expressed in cc/g. The calculation requires one choice:

Relative Pressure. 0.9814 ADS, 0.9907 ADS, 0.9814 DES (SA3100 Plus only), or 0.9907 DES (SA 3100 Plus only). These options represent the relative pressure at which the pore volume is calculated, using either an adsorption or desorption (SA 3100 Plus only) isotherm. A relative pressure of 0.9814 includes pores

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of diameters less than or equal to 100 nm, and a relative pressure of 0.9907 includes pores of diameters less than or equal to 200 nm.

See Chapter 6, Principles of Operation, for a more detailed discussion of these calculations and the underlying theory.

Defining, Editing and Deleting Profiles

To edit an existing profile, create a new profile, or delete a profile from the profile list you must access the **Profile** screen:

Press Setup in either the **Analysis** or **Outgas** screen, then press Edit Profiles when the **Setup and Print** screen is displayed. The **Profile** screen appears displaying the profile for the current sample, see Figure 7.3. Editing the displayed profile, editing another profile, creating a new profile, and deleting profiles are covered in sections with their own headings below.

Editing the Displayed Profile

To edit the displayed profile:

1. Change information or settings in each field (except for Profile ID) as necessary by selecting the associated key on the screen. Refer to the Profile Keys section below.

2. Press OK to save the profile.

3. If you do not have any other profile-related functions to perform, press OK again to return to the **Outgas** or **Analysis** screen.

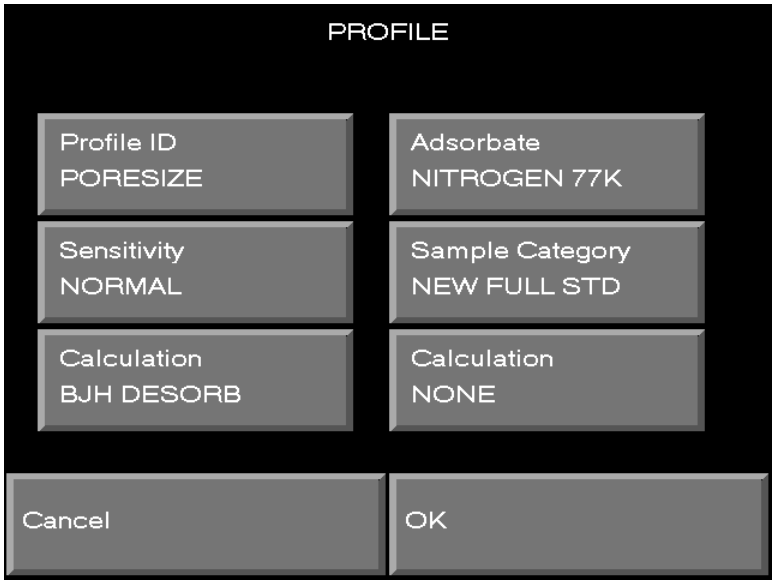


Figure 7.3 Profile Screen

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Editing a Profile Other Than the Displayed Profile

To edit a profile other than the displayed profile:

1. Press Profile ID, the **Profiles** screen, Figure 7.4, appears.

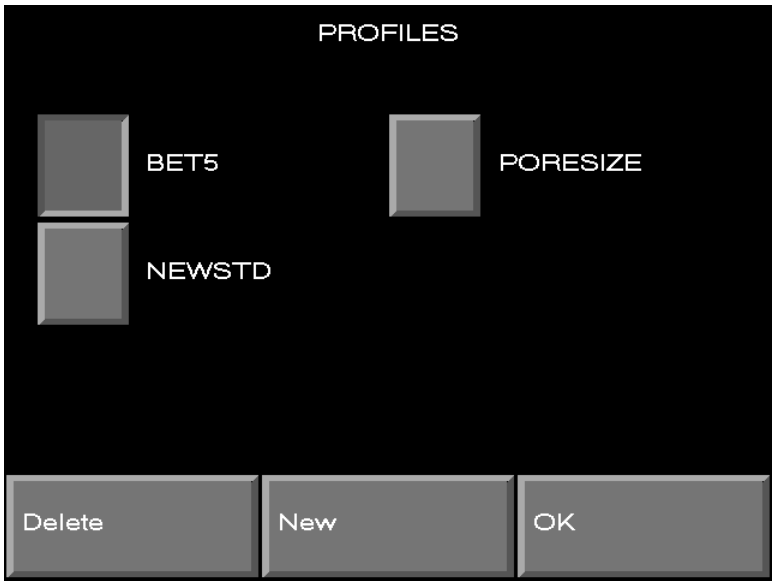


Figure 7.4 Profiles Screen

2. Select the desired profile.
3. Press OK to return to the **Profile** screen.
4. Change information or settings in each field (except for Profile ID) as necessary by selecting the associated key on the screen. Refer to the Profile Keys section below.
5. Press OK to save the profile.
6. If you do not have any other profile-related functions to perform, press OK again to return to the **Outgas** or **Analysis** screen

Defining a New Profile

To define a new profile:

1. Press Profile ID, the **Profiles** screen, Figure 7.4, appears.
2. If a profile already exists which contains common elements with the new profile you wish to create, select the profile in the **Profiles** screen.
2. Press New in the **Profiles** screen. The **New Profile** screen appears, Figure 7.5.

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Figure 7.5 New Profile Screen

3. Change information or settings in each field as necessary by selecting the associated key on the screen. Refer to the Profile Keys section below.

4. Press OK to save the profile and return to the **Profiles** screen. If you have not assigned a new Profile ID, you are asked to confirm that you want to replace the original profile with that Profile ID.

   From the **Profiles** screen, you can perform other profile-related functions or exit to the **Setup and Print** screen by pressing OK twice. Press OK again to return to the **Outgas** or **Analysis** screen.

Deleting Profiles

To delete a profile:

1. Press Profile ID, the **Profiles** screen, Figure 7.4, appears.

2. Select the profile you wish to delete.

3. Press Delete to delete the profile. You are asked to confirm the deletion of the profile.

4. If you do not have any other profile-related functions to perform, press OK twice to return to the **Setup** screen. Press OK again to return to the **Outgas** or **Analysis** screen.

Profile Editing Keys

Pressing each key on the **Profile** or **New Profile** screen allows you to exercise options associated with that key as follows (press OK to confirm a selection and return to the previous screen or Cancel to cancel any changes made and return to the previous screen).

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Profile ID Key

Profile ID

Options depend on whether you are in the **Profile** screen or creating a new profile in the **New Profile** screen.

- **Profile** screen - Displays the **Profiles** screen, see Figure 7.4. From this screen you may delete unwanted profiles from the list, select another profile to edit, or display a template to create a new profile. If you are editing the currently displayed profile, do not use the Profile ID key.
- **New Profile** screen - Displays an alphanumeric keypad allowing you to enter a new Profile ID when creating a new profile.

Adsorbate Key

Adsorbate

Displays the **Adsorbate** screen, allowing you to select from the currently available adsorbate gases.

Sensitivity Key

Sensitivity

Displays the **Sensitivity** screen, allowing you to choose one of three levels of sensitivity:

- HIGH
- NORMAL
- LOW
- LOW (FAST)

Category Key

Category

Displays the **Category** screen, see Figure 7.6, allowing you to:

Choose from the following using the square keys:

- UNKNOWN - for a sample with no existing standard isotherm, and for which no standard isotherm is desired.
- NEW ADS STD - to create an adsorption standard isotherm for a new sample.
- NEW FULL STD - (SA 3100 Plus only) to create a full adsorption and desorption standard isotherm for a new sample.
- [USER-NAMED STANDARDS] Any previously created standards. This choice instructs the SA 3100 analyzer to use the standard to accelerate the analysis of the sample.

Note. When the Sample Category in a profile is NEW ADS STD or NEW FULL STD, you will be asked to enter a name for the standard after the cryogen dewar is loaded and before the measurement begins.

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- or -

Delete any previously created standard:

To delete a standard, select the standard using the associated square key, then press Delete.

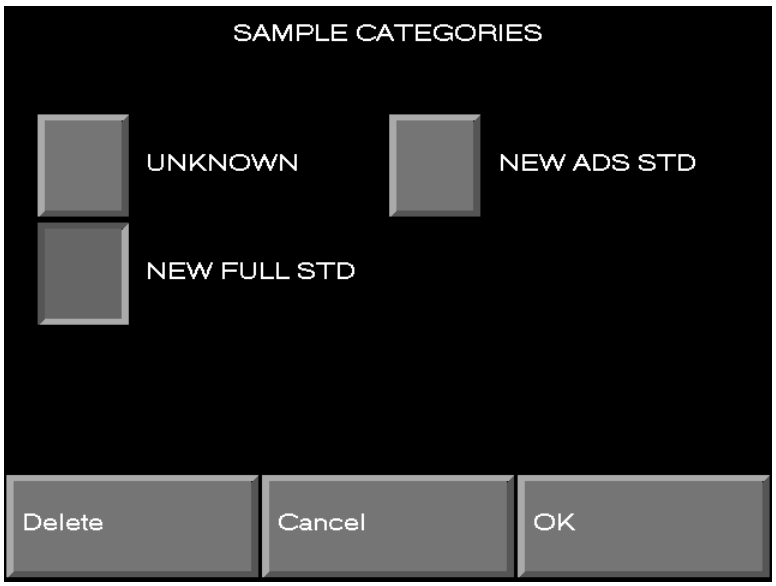


Figure 7.6 Category Screen

Printer Output Key

Printer Output — Displays the **Printer Output** screen, allowing you to choose from:

- None
- Full Report
- Summary

Calculation Key

Calculation — Options depend on what is currently displayed in the key. A new Calculation key is created each time one is used. To add a calculation, use a Calculation key on which 'None' is displayed. To change a calculation, press the key displaying the calculation to be changed. Each possibility for Calculation keys is explained under its own heading below.

Calculation Key Displaying 'None'

The **Calculations** screen appears, Figure 7.7, allowing you to select BET, LANGMUIR, t-PLOT, BJH ADSORB, BJH DESORB (SA 3100 Plus only) or PORE VOLUME. If you select one of the calculations, the associated **Calculation** screen appears as described below under Calculation Key displaying 'BET', or Calculation Key Displaying 'BJH ADSORB', etc.

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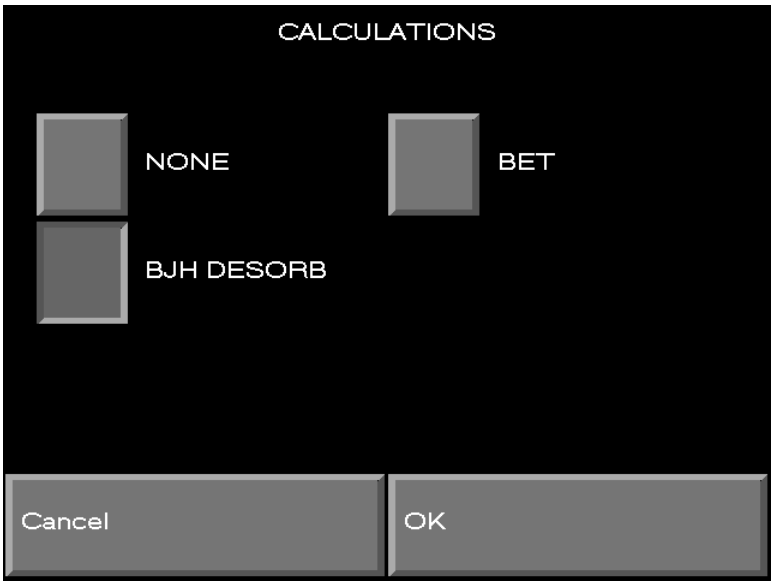


Figure 7.7 Calculations Screen

Calculation Key Displaying 'BET'

The BET **Calculation** screen appears, Figure 7.8, allowing you to select:

Calculation — to access the **Calculations** screen and change the calculation. If you change the calculation the **Calculation** screen for the chosen calculation appears, see below.

Points — to change the number of isotherm points used.

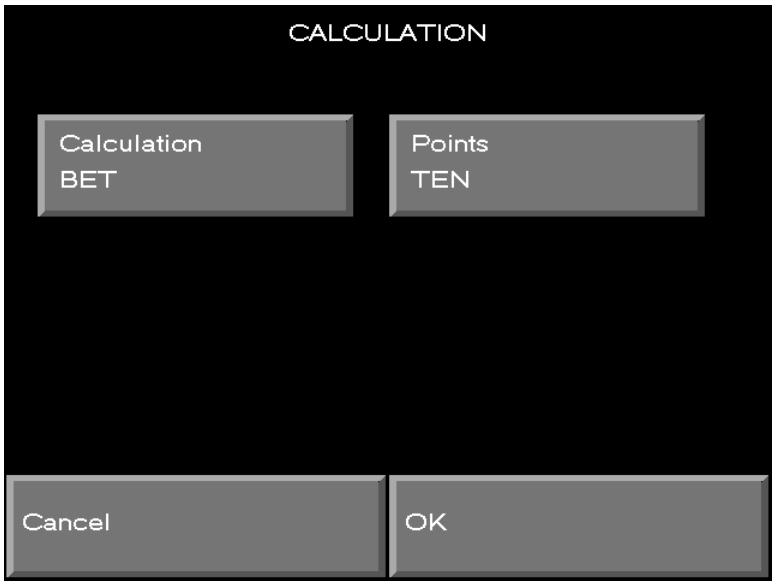


Figure 7.8 BET Calculation Screen

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Calculation Key Displaying 'BJH Adsorb' or 'BJH Desorb'

The BJH ADSORB or BJH DESORB (SA 3100 Plus only) **Calculation** screen appears allowing you to select:

Calculation — to access the **Calculations** screen and change the calculation. If you change the calculation to BET, another **Calculation** screen appears, see above for BET, or below for others.

Resolution — to change the number of isotherm points used.

Size Range — to change the size range of the pore size distribution.

Equation — to change the equation used to calculate the pore size distribution.



Figure 7.9 BJH Calculations Screen

Calculation Key Displaying 'LANGMUIR'

The LANGMUIR **Calculation** screen appears allowing you to select:

Calculation — to access the **Calculations** screen and change the calculation. If you change the calculation the **Calculation** screen for the chosen calculation appears, see below for t-PLOT and PORE VOLUME, or above for others.

Points — to change the number of isotherm points used.

Calculation Key Displaying 't-Plot'

The t-PLOT **Calculation** screen appears allowing you to select:

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Calculation	to access the **Calculations** screen and change the calculation. If you change the calculation the **Calculation** screen for the chosen calculation appears, see below for PORE VOLUME, or above for others.
Resolution	to change the number of isotherm points used.
Calc. BET Range	to change the P/P_0 range used for the BET surface area calculation.
Calc. t-PLOT Range	to change the t-PLOT range used for calculation.
Equation	to change the equation used.

Calculation Key Displaying 'Pore Volume'

The Pore Volume **Calculation** screen appears allowing you to select:

Calculation	to access the **Calculations** screen and change the calculation. If you change the calculation the **Calculation** screen for the chosen calculation appears, see above.
Relative Pressure	To select the relative pressure at which to calculate the total pore volume.

Creating a Fast BET Profile

Fast BET Profile Components

A profile to perform a Fast BET analysis must meet the following criteria:

- It must have <u>one</u> BET calculation (3-point, 5-point, or 10-point), and no other calculations.
- The Sensitivity selection <u>must</u> be LOW (FAST).

Optimizing Speed

To achieve optimum speed for your Fast BET analysis, use standard isotherms for the materials you will regularly analyze in this manner. To do this, use NEW ADS STD as the Category selection the first time you measure a material. This measurement will not be fast because of the time it takes to measure a standard isotherm. The next time, however, change the Category selection to the USER-NAMED STANDARD you have created. If you have more than one material that you will be performing Fast BET measurements on, create a different profile for each one, using the standard isotherm for the given material as the Category selection in each profile.

Note. Best results for Fast BET analyses will be obtained using a 3cc RapiTube sample tube.

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7.5 DETERMINING THE OPTIMAL SAMPLE QUANTITY

General

The SA 3100 analyzer produces accurate results for sample quantities with a total surface area as low as 1 m^2. Optimal results, however, are obtained when the total surface of the sample is between 3 m^2 and 30 m^2. Also, the mass of sample should be more than 100 mg to ensure weighing accuracy. The table below shows the recommended sample quantity as a function of estimated specific surface area for samples with a specific surface area of greater than 1 m^2/g, using nitrogen as the adsorbate. Refer to the appropriate range in the table to determine how much sample to add. For samples with a specific surface area of less than 1 m^2 see Low Surface Area samples, below. After you have analyzed your sample, you will know the specific surface area and you will be able to more accurately determine the optimal sample quantity.

Samples with Specific Surface Area > 1m^2

Use Table 7.1 to determine how much sample to use for samples with a specific surface area greater than 1m^2/g.

The mass of sample you need and the material's bulk density will determine the size of sample tube you need, see heading 7.6, Choosing a Sample Tube.

Table 7.1 Optimal Sample Quantity

Estimated Specific Surface Area	Mass of Sample for Analysis
>30 m^2/g	0.1 g - 0.2 g
10 m^2/g - 30 m^2/g	0.3 g
3 m^2/g - 9.9 m^2/g	1 g
2 m^2/g - 2.9 m^2/g	1.5 g
1.5 m^2/g - 1.9 m^2/g	2 g
1 m^2/g - 1.4 m^2/g	3 g

Low Surface Area Samples

For samples with specific surface areas less than 1m^2/g, calculate the quantity required based on a minimum total surface of 3m^2. If the resulting volume is too large to fit into a 12cc sample tube (see heading 7.6, below) the minimum total surface area can be reduced to as low as 1m^2, although some accuracy will be sacrificed. If the resulting volume is still too large, you will have to consider adsorbate gases other than nitrogen.

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7.6 CHOOSING A SAMPLE TUBE

General

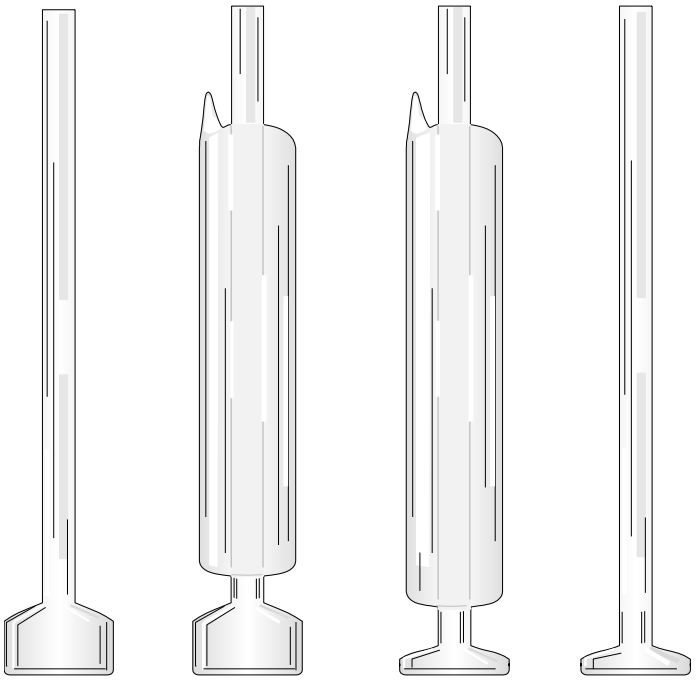
See Figure 7.10 illustrating available sample tubes. Sample tubes are available in three standard styles: Beckman Coulter RapiTube sample tubes, Beckman Coulter VacJak sample tubes, which are vacuum jacketed, and the object tube. RapiTube sample tubes are available in three sizes, 3cc, 9cc and 12cc; VacJak sample tubes are available in 3cc and 9cc sizes. The object tube is available in only one size and is not vacuum jacketed. The tube you need is determined by the type of analysis you wish to perform and the properties of the material you are analyzing.

Sample Tube Inserts

All tubes have inserts to decrease the dead volume inside the tube during analysis. These inserts increase sensitivity and accelerate the measurement.

Sample Tube Size

The size of sample tube depends on the volume of sample required, see heading 7.5 Determining the Optimal Quantity of Sample. The sample must fit into the bulb with at least 1mm clearance between sample and the top of the bulb.



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Beckman Coulter RapiTube Sample Tubes vs. Beckman Coulter VacJak Sample Tubes

RapiTube sample tubes can be used for any measurement, and should be used for all surface area measurements except for solid piece samples that will not fit in the tubes. VacJak tubes minimize the effects of changing liquid cryogen level as

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cryogen boils away during long pore parameter analyses, and are recommended under the following conditions:

- When highest accuracy is required for pore parameter measurements
- When the pore parameter measurement takes longer than 8 hours
- Or, when a full adsorption and desorption isotherm is measured for pore size determination, the desorption isotherm crosses below the adsorption isotherm. In this case, repeat the measurement with a VacJak tube, and use the VacJak tube for future runs of this material when using the selected profile.

Object Tube

The object tube is intended for the surface area analysis of solid piece samples that will not fit in standard tubes. The object tube is not intended for pore parameter measurements.

7.7 WEIGHING THE SAMPLE TUBE ASSEMBLY

General

The sample tube assembly consists of the sample tube and the sample tube insert, see Figure 7.11. The assembly must be accurately weighed using an analytical balance before adding sample. Although you add a known amount of sample to the tube, weight is lost during outgassing. Therefore, the sample tube weight must be known to accurately measure the sample weight after outgassing.

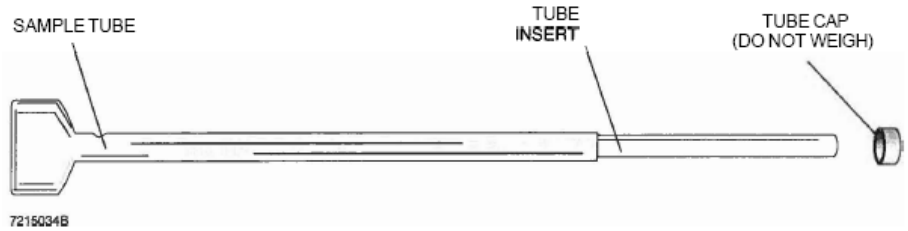


Figure 7.1 1 Sample Tube Assembly

Labeling Parts

The sample tube and sample tube insert must stay together to avoid weight inaccuracies resulting from interchanged parts. You should therefore label sample tubes on the provided labeling area BEFORE weighing. Indelible lab markers can be used to place identifying marks on inserts. Allow ink to dry completely before weighing.

Weighing Procedure

1. Ensure that the sample tube is clean and dry.

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2. Refer to Figure 7.11. Insert the sample tube insert into the sample tube. The insert decreases the free volume inside the tube, increasing sensitivity and accuracy.

3. Weigh the tube (including the tube insert) on the analytical balance to at least 0.001g precision. Make sure the following conditions are met before weighing the sample tube assembly:

   - The sample tube is at room temperature.
   - The sample tube is clean, dry, and without fingerprints. Use a lint-free laboratory tissue to remove fingerprints.
   - The weighing pan on the analytical balance is clean.
   - The sample tube is not charged with static electricity. Use an anti-static device to discharge the tube.

Ensure that the insert used for this weighing stays with the sample tube. Interchanging parts after weighing introduces significant weight errors. Make a note of the weight - you will need it later. Label the parts uniquely to avoid confusion.

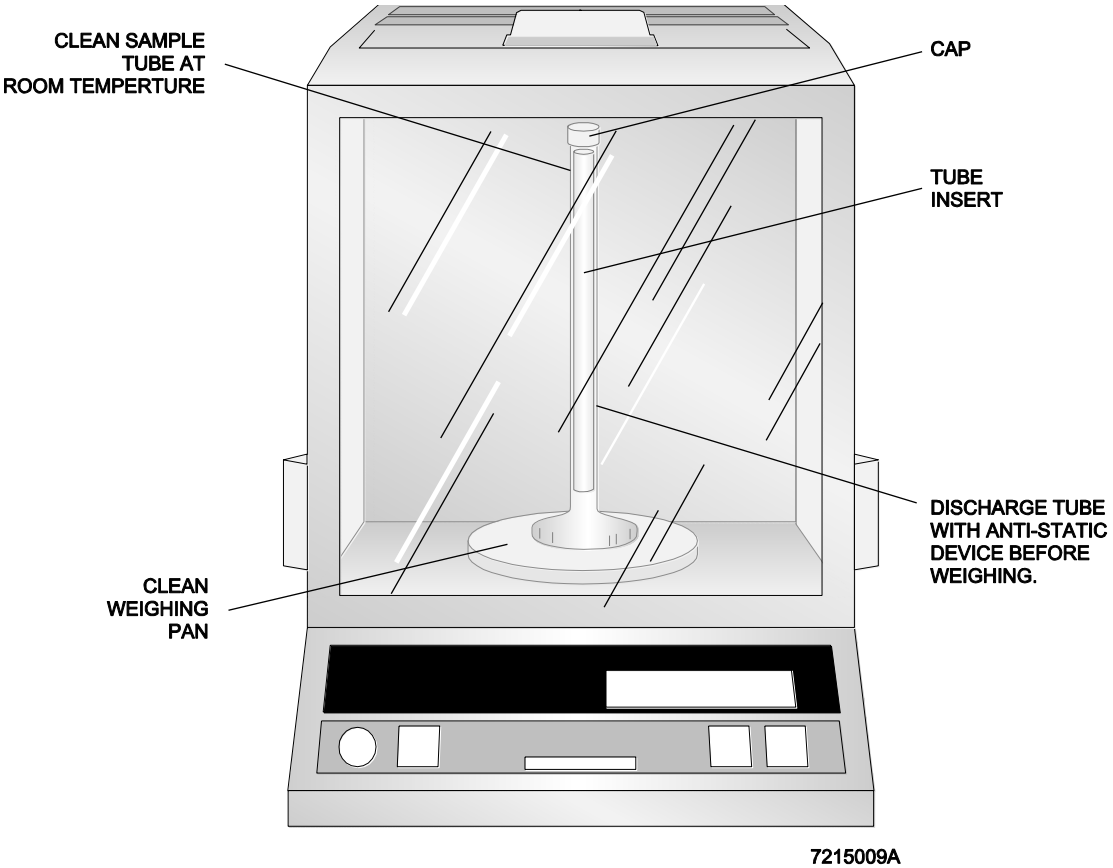


Figure 7.12 Weighing the Sample Tube Assembly Using an Analytical Balance

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7.8 ADDING SAMPLE TO THE SAMPLE TUBE

General

The amount of sample added to the sample tube must be within certain limits to ensure accurate analysis. See heading 7.5 Determining the Optimal Sample Quantity.

Procedure

1. Remove the tube insert from the sample tube, then pour the weighed sample into the sample tube using the sample loading funnel, Figure 7.13.

2. Replace the tube insert in the sample tube.

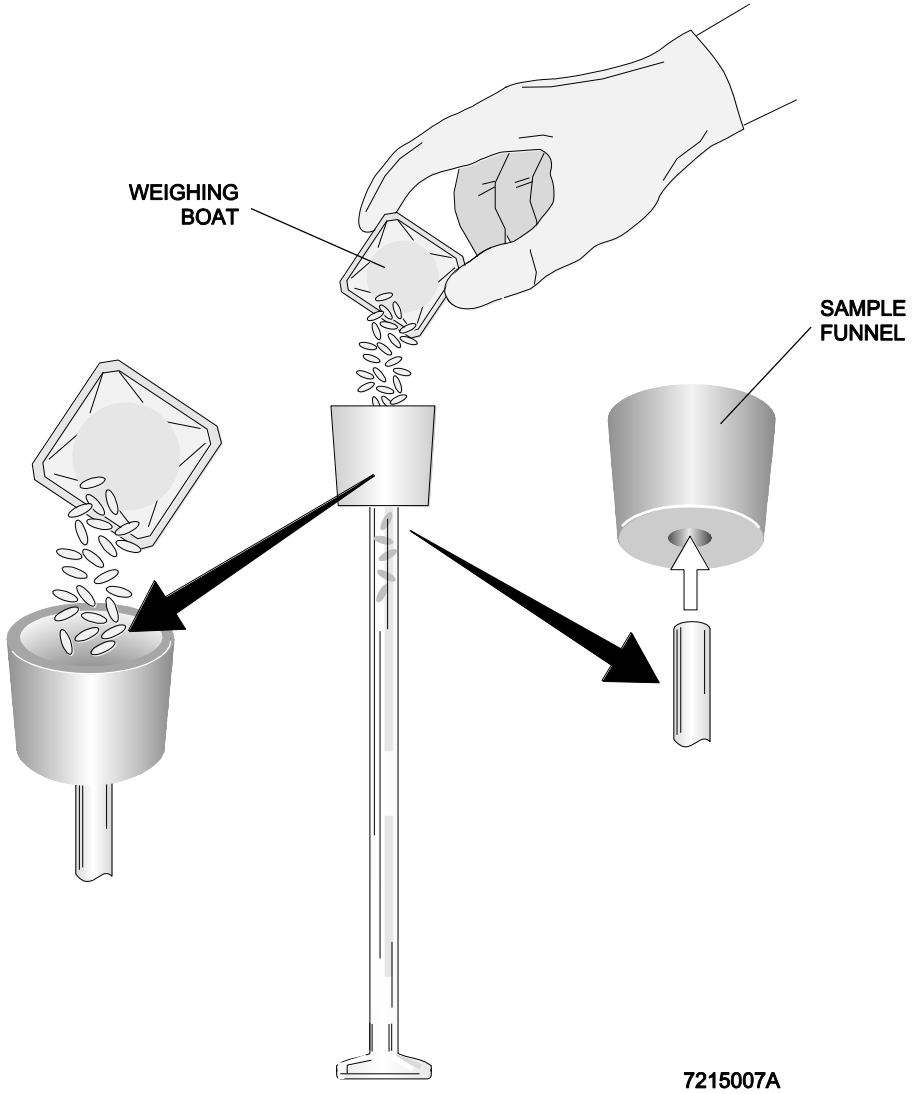


Figure 7.1 3 Adding Sample to the Sample Tube

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7.9 LOGGING-IN SAMPLES

General

Before analysis, samples must be logged into the SA 3100 analyzer.

If outgassing is to be done on the instrument, samples should be logged in prior to outgassing by entering information on the **Sample Information** screen, Figure 7.15. The Sample Information contains all the information necessary to identify the sample and define the analysis. The SA 3100 analyzer keeps track of the status of the samples as it proceeds through the outgassing and analysis process. When outgassing of the samples is complete, the sample information is added to the **Outgassed Samples** list.

If outgassing is to be done on the SA-PREP outgasser, samples can be logged in as new samples before initiating analysis, as described in section 7.12, Analyzing Samples. When you are ready to analyze the samples, refer back to this section for descriptions of the components of the **Sample Information** screen.

Components of Sample Information Screen

The Sample Information consists of the following items.

Sample ID

An alphanumeric identifier for the sample.

Customer

The party requiring the analysis.

Note. This field is stored as an independent string of characters, and can therefore be used for any information. The name of this field can be changed using the 'SA 3100 Advanced Configuration Kit'.

Operator

You.

Note. This field is stored as an independent string of characters, and can therefore be used for any information. The name of this field can be changed using the 'SA 3100 Advanced Configuration Kit'.

Sample weight

The mass, in grams, of the sample added to the sample tube. The sample weight must be accurately measured after outgassing and re-entered; the instrument will also prompt you for the correct weight at the completion of the measurement and before calculations are made.

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Profile

The analysis protocol for the sample chosen from the **Profile List**, see heading 7.3 Profiles.

Max temperature

The maximum temperature the sample material can endure without degrading. This temperature limits the outgassing furnace temperature. The actual temperature used for outgassing is set on the **Outgas** screen, but if the set temperature is greater than the Max temperature, the furnace will be limited to the Max temperature.

Log-in Procedure

To log a sample into the SA 3100 analyzer:

1. Move to the **Outgas** screen, Figure 7.14:

   If the **Analysis** screen is displayed press the Outgas key to move to the **Outgas** screen. If the **Outgas** screen is displayed, go to step 2.

2. If no samples are currently outgassing, the instrument is ready to outgas the sample, indicated by the following message at the bottom of the **Outgas** screen

   LOAD AND LOG SAMPLES
   Press Start Outgas when loaded

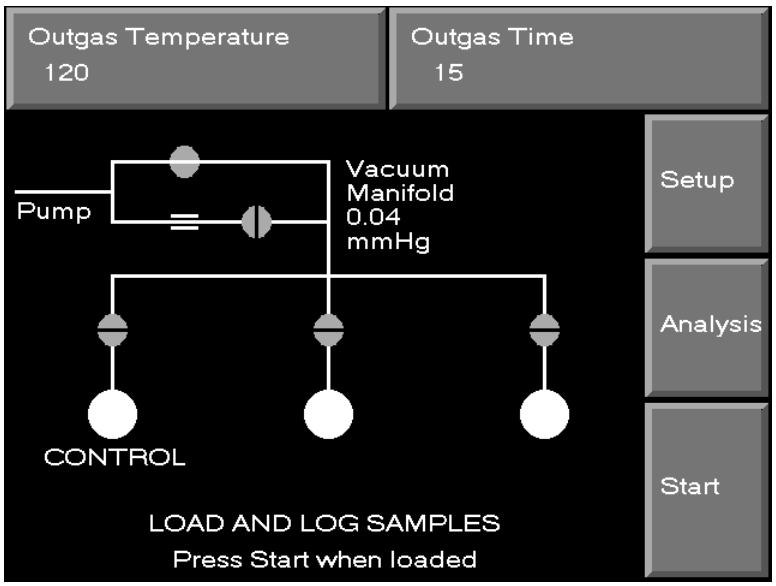


Figure 7.14 Outgas Screen

The circles in the central area of the **Outgas** screen correspond to the outgas ports. Press the circle corresponding to the outgas port to which you will attach the sample tube for outgassing, see heading 7.10 Outgassing Samples. The **Sample Information** screen appears, Figure 7.15.

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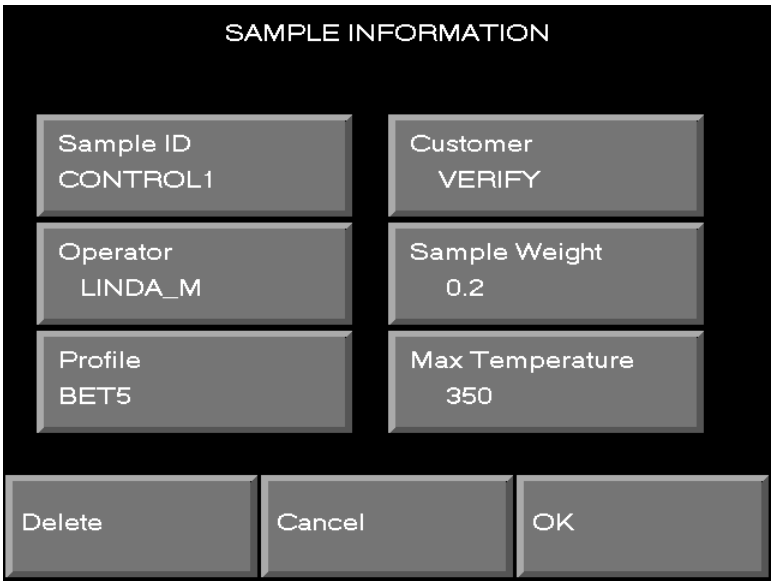


Figure 7.1 5 Sample Information Screen

3. Enter information or make selections by pressing each key on the **Sample Information** screen:

   - Sample ID, Customer and Operator display alphanumeric keypads allowing you to enter information for these fields.
   - Sample Weight and Max Temperature display numeric keypads allowing you to enter values. Sample weight is in grams and Max temperature is in degrees Centigrade.
   - Profile displays the **Profile List** screen, allowing you to select a profile. The profile determines the analysis to be performed.

Press OK to confirm your selections and return to the **Outgas** screen. If you wish to remove a sample you have logged in from the sample list, press the associated circle and press Delete. There are three outgassing ports on the instrument, allowing you to outgas up to three sample simultaneously. If you want to outgas more than one sample, repeat the log-in procedure for the other samples. See heading 7.10, Outgassing Samples, for attaching sample tubes to the outgassing ports and starting the outgas process.

7.10 OUTGASSING SAMPLES

General

This procedure assumes you have already logged-in the samples to be outgassed as described above in heading 7.8 Logging-in Samples. Once samples have been logged-in, outgassing consists of attaching the sample tubes to the outgas ports, editing the outgas parameters if necessary, and initiating the automated procedure.

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To outgas a sample, you should have a sample tube with a known empty weight, loaded with the correct quantity of sample and containing a sample tube insert.

Outgassing Parameters

The outgassing parameters are the outgas temperature and the outgas time. The outgas time is the elapsed time after the sample reaches the outgas temperature. The maximum outgas temperature is 350 degrees Centigrade. We recommend a minimum outgas time of 5 minutes at the highest temperature your sample can withstand. If you do not have a standard outgas protocol, or are unsure of what values to use, see Appendix B, Outgassing Notes.

Outgassing Procedure

Attaching the Sample Tube to an Outgas Port

Before attaching sample tubes to outgas ports, note that port O-rings should be checked for cleanliness daily, and greased weekly. See heading 8.3, Cleaning and Greasing O-rings.

CAUTION

Do not use any tools to remove or install port fittings. Fittings are designed to seal when finger-tight

---

Refer to Figure 7.16.

1. Loosen the knurled nut on the outgas port.
2. Insert the tube into the outgas port until it stops against the manifold.
3. Tighten the knurled nut until finger-tight.

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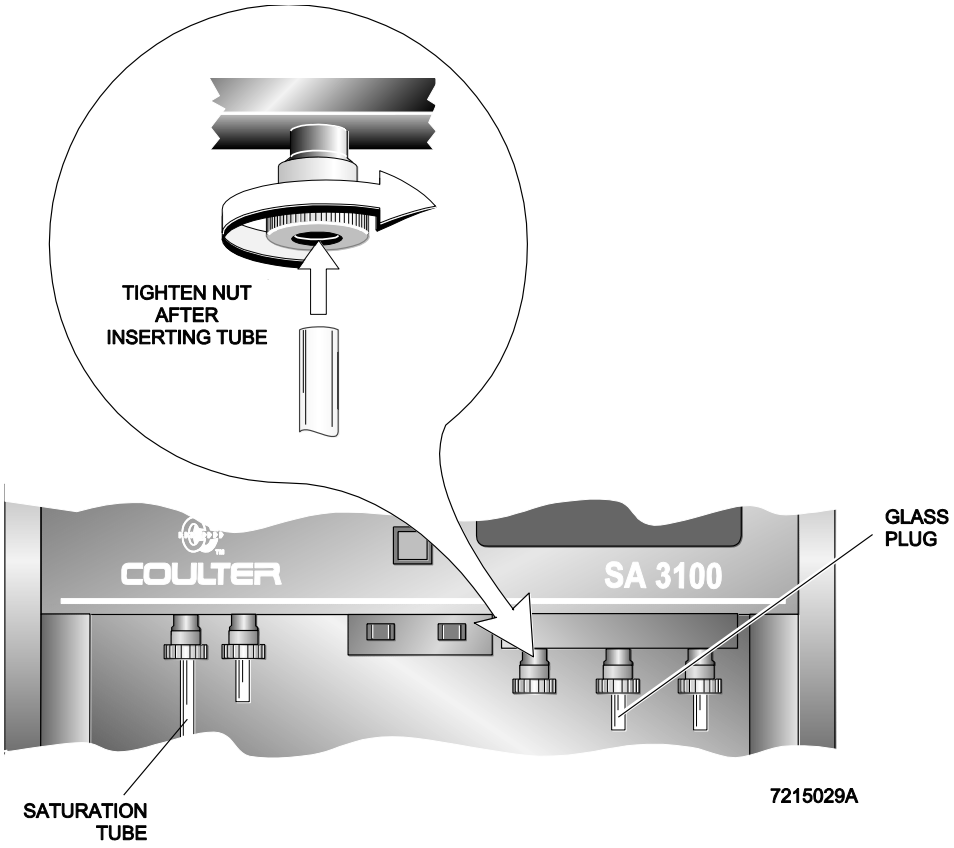


Figure 7.16 Connecting the Sample Tube to an Outgas Port

The sleeve compresses the O-ring against the glass tube, forming an air-tight seal. The tube does not have to feel rigid in the fitting to be sealed. If a gentle downward pull on the tube does not pull it out of the fitting, it is installed properly.

Editing the Outgassing Parameters

To edit an outgas parameter:

1. Move to the **Outgas** screen, Figure 7.14:

   If the **Analysis** screen is displayed press the Outgas key to move to the **Outgas** screen. If the **Outgas** screen is displayed, go to step 2.

2. Press Outgas Temperature or Outgas Time to change the value displayed on the key. Both keys provide you with a numeric keypad to enter a new value.

Note. The Maximum Temperature entered in the Sample Information for any of the samples to be outgassed overrides the Outgas Temperature if the Outgas Temperature is higher than the Maximum Temperature.

Initiating Outgassing

When sample tubes have been attached to the outgassing ports and correct outgassing parameters have been entered, you may initiate outgassing by pressing Start Outgas.

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When outgassing is complete, the message *Outgassed* is displayed below the graphic for the outgassing port(s) used. In addition, the following message is displayed at the bottom of the **Outgas** screen:

REMOVE SAMPLES
Press OK when removed

The Sample Information for outgassed samples enters the Outgassed Samples list. The sample(s) are now ready to be weighed and analyzed.

Removing Sample Tubes

To remove sample tubes from the outgas ports:

1. Have a sample tube cap ready.

2. Loosen the knurled nut while holding the tube and pull the tube out of the fitting.

3. Promptly place the sample tube cap on the sample tube (the tube is filled with nitrogen, so waiting too long will allow the gas to mix with air).

If you cannot weigh the outgassed samples immediately, make sure the caps are secure on the tubes and place the tubes in a clean desiccating cabinet.

7.11 WEIGHING OUTGASSED SAMPLES

General

After samples have been outgassed, the sample weight can be accurately determined. This outgassed weight is critical to obtaining correct analysis results. Therefore, extreme care should be taken when weighing the outgassed samples.

Weighing the Sample Tube Assembly and Sample

When you weighed the empty tube, you included the sample tube insert. The weighing with the outgassed sample should therefore also include the insert, but not the cap.

See Figure 7.17. To weigh the outgassed sample:

1. Weigh the tube (including the tube insert and sample) on the analytical balance to at least 0.001 g precision. Remove the cap immediately before weighing and promptly replace the cap after weighing. Make sure the following conditions are met before weighing the sample tube after outgassing:

   - The sample tube is at room temperature.

   - The sample tube is clean and without fingerprints. Use a lint-free laboratory tissue to remove fingerprints.

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- The weighing pan on the analytical balance is clean.
- The sample tube is not charged with static electricity. Use an anti-static device to discharge the tube.

2. Subtract the weight found earlier for the sample tube assembly without sample to determine the outgassed sample weight.

Make a note of the resulting sample weight. The instrument prompts you to enter this weight in the Sample Information at the completion of the measurement and before calculations are made.

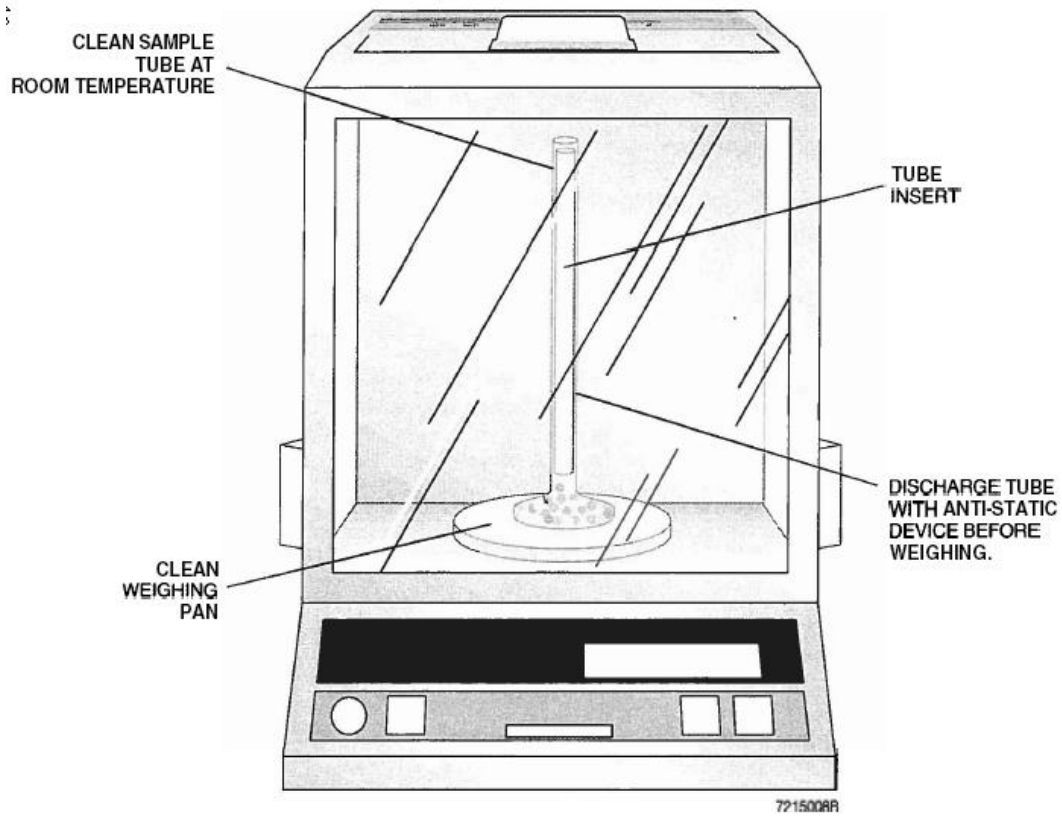


Figure 7.17 Weighing the Sample Tube Assembly and Sample after Outgassing

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7.12 ANALYZING SAMPLES

General

Before analyzing a sample it must be logged-in to the SA 3100 analyzer, outgassed, and weighed, see the appropriate procedures above. To initiate the analysis, you simply need to choose the sample from the **Outgassed Samples** screen, attach the sample tube to the sample port, and start the analysis. The analyzer prompts you at each step.

For samples outgassed on the SA-PREP outgasser, the procedure below directs you as required.

Analysis Procedure

IMPORTANT

Do not load the sample tube onto the analysis port until instructed to do so by a message on the touch screen. Doing so may compromise analysis results.

Selecting the Sample to Analyze

You must first select the sample to be analyzed from the **Outgassed Samples** screen, or log-in the sample if it was outgassed elsewhere:

1. If the **Outgas** screen is displayed, press Analysis to move to the **Analysis** screen.

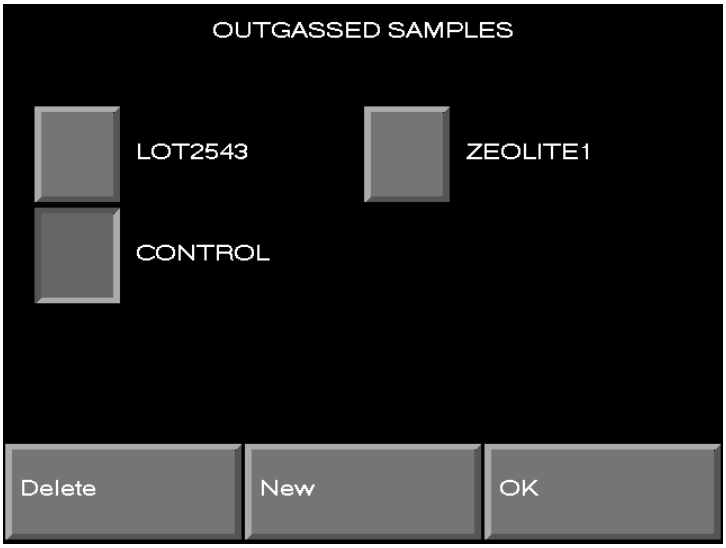


Figure 7.18 Outgassed Samples Screen

2. Press Next Analysis. The **Outgassed Samples** screen is displayed, see Figure 7.18.

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- If the sample to be analyzed was outgassed on the SA 3100 analyzer:
  Select the sample you want to analyze by pressing the key marked with the correct Sample ID, then press OK. The **Sample Information** screen is displayed.

- If you are analyzing a sample outgassed on the SA-PREP outgasser:

  Press New. The **New Sample** screen is displayed. Fill out this screen with the exception of the Sample Wt, referring to section 7.8 Logging-in Samples if necessary, but do not press OK. Proceed to Entering the Outgassed Sample Weight, and complete steps 1, 2 and 4.

  Note. If all samples outgassed on the SA 3100 analyzer have been analyzed, or if no samples have been outgassed on the instrument, the **New Sample** screen is displayed immediately after Next Analysis is pressed.

Entering the Outgassed Sample Weight

Enter the exact outgassed sample weight in the Sample Weight field (see heading 7.10 Weighing Outgassed Samples):

1. Press the Sample Wt key of the **Sample Information** screen.

2. Enter the sample weight on the numeric keypad and press Enter.

3. Edit any other fields as necessary, with the exception of Sample ID - pressing Sample ID will return you to the **Outgassed Samples** screen.

4. Press OK on the **Sample Information** screen to confirm entries and return to the **Analysis** screen (if you have entered information for a sample outgassed elsewhere, you may have to press OK more than once to return to the **Analysis** screen). The following message appears:

   LOAD SAMPLE
   Press OK when loaded

   Load the sample as described below.

Loading the Sample

Before attaching the sample tubes to the sample port, note that port O-rings should be checked for cleanliness daily, and greased weekly. See heading 8.3, Cleaning and Greasing O-rings.

CAUTION

Do not use any tools to remove or install port fittings. Fittings are designed to seal when finger-tight.

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See Figure 7.19. Attach the sample tube to the sample port:

1. Loosen the knurled nut on the sample port.

2. Remove the cap from the sample tube, then insert the tube into the port until it stops against the manifold.

3. Tighten the knurled nut until finger-tight.

   The sleeve compresses the O-ring against the glass tube, forming a gas-tight seal. The tube does not have to feel rigid in the fitting to be sealed. If a gentle downward pull on the tube does not pull it out of the fitting, it is installed properly.

4. Press OK. The Status message on the screen reads:

   'WAIT... SAMPLE EVACUATING'.

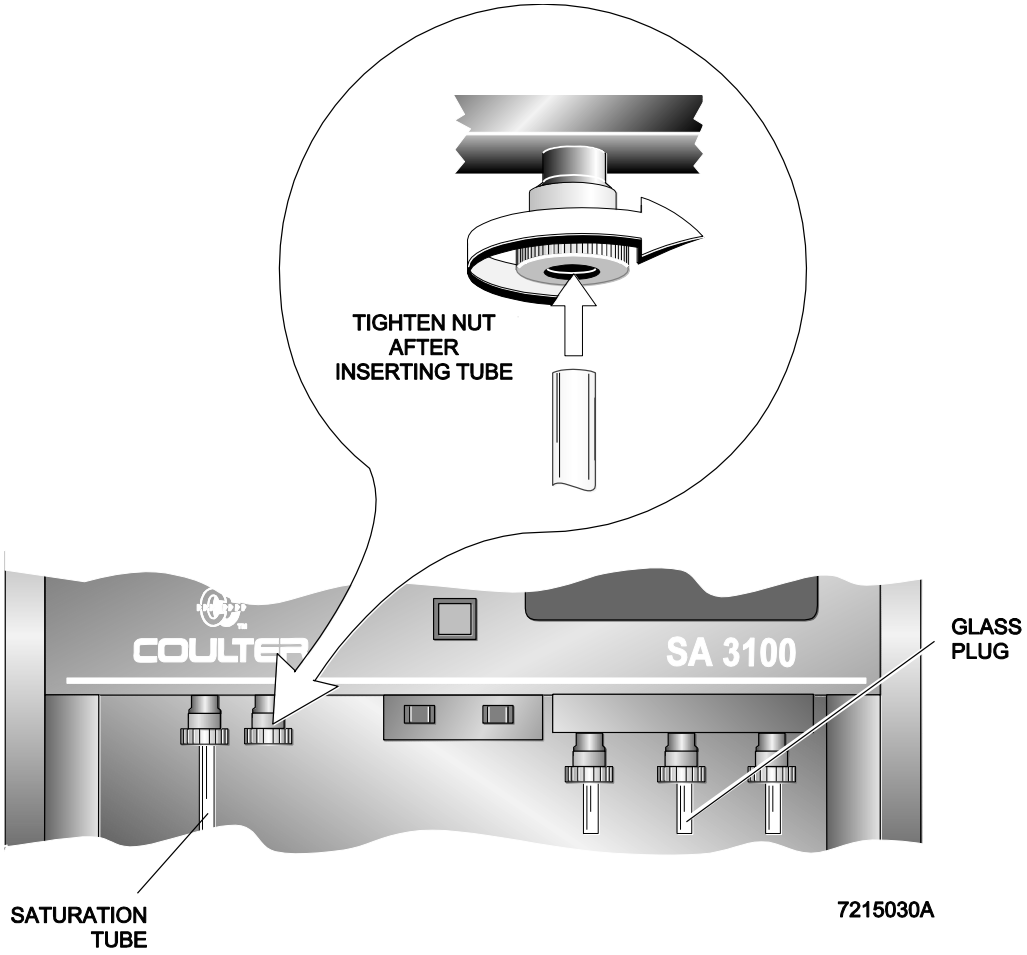


Figure 7.19 Connecting the Sample Tube to the Analysis Port

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Tempering, Filling and Loading Liquid Nitrogen Dewar

WARNING

Use proper safety equipment when handling liquid nitrogen, including, but not limited to, safety glasses and low temperature thermal gloves.

WARNING

Always temper new dewars by adding a small quantity of liquid nitrogen and swirling the liquid in the dewar before filling. This reduces the risk of implosion and injury.

1. If the dewar is new, temper the dewar by swirling a small quantity of liquid nitrogen in the dewar.

2. Add liquid nitrogen to the dewar until it is half-full.

IMPORTANT

Do not load the dewar onto the dewar rack until instructed to do so by a message on the touch screen. Doing so will condense nitrogen on the sample, causing low surface area results.



Figure 7.20 Adding Liquid Nitrogen to Dewar

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3. When the following message appears:

   LOAD DEWAR
   Press Start Analysis when loaded

   load the half-full dewar into the dewar rack, see Figure 7.21.

   a. Slide the dewar up over the sample and saturation tubes, inserting the top of the rear dewar wall behind the upper tab in the dewar rack.

   b. Move the bottom of the dewar back into the rack, then slide the dewar down so that the bottom fits over the lower tab in the dewar rack.

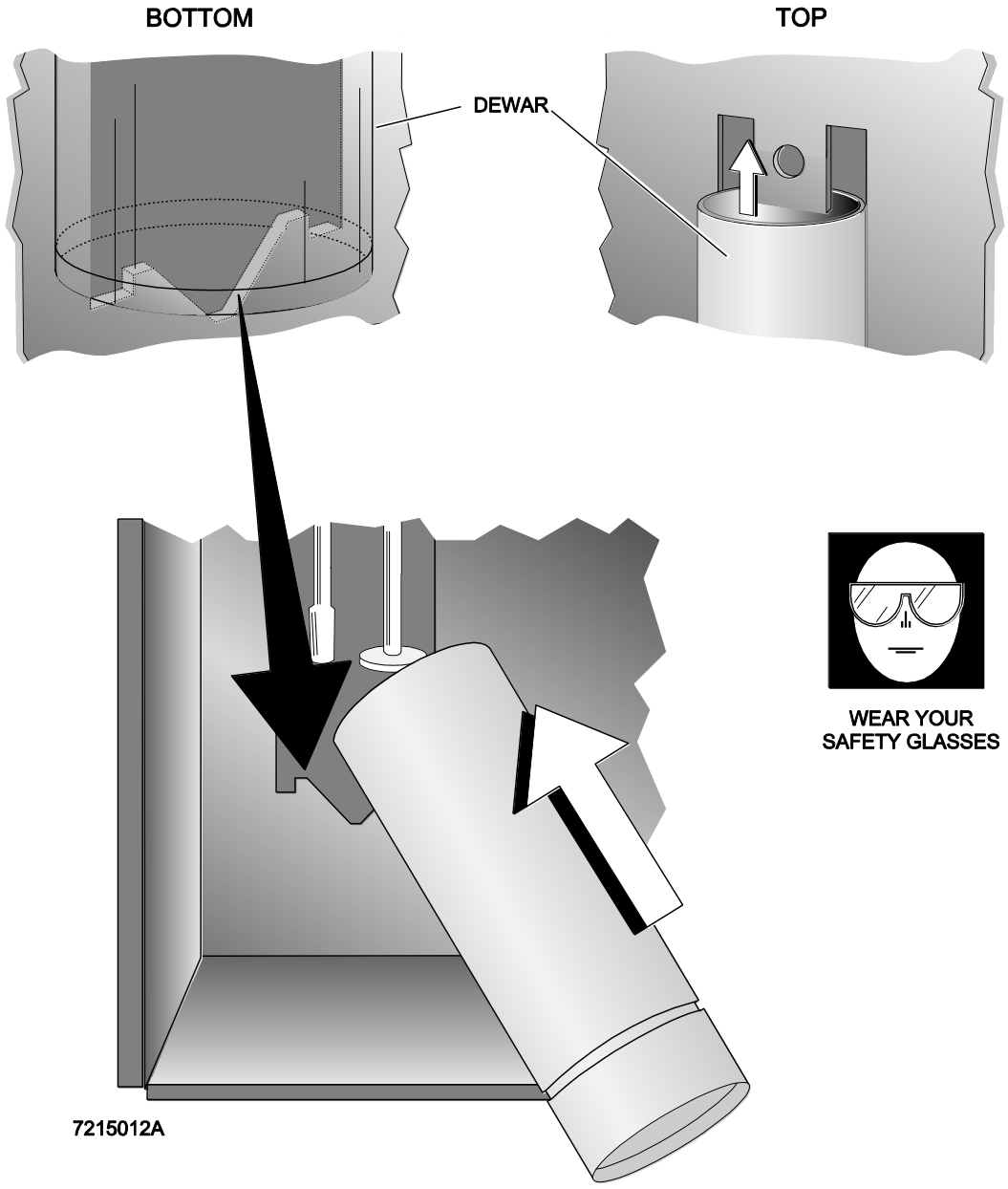


Figure 7.21 Loading the Dewar into Dewar Rack

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IMPORTANT

Do not fill the dewar above 2 cm from the top. Doing so will cause temperature changes due to rapid evaporation and may cause inaccurate results.

4. Using the dewar funnel fill the dewar with liquid nitrogen to 2 - 3 cm (1 inch) from the top, see Figure 7.22.

5. Install the insulating dewar lid, see Figure 7.23.

6. Press the Start Analysis key. The sample analysis proceeds automatically.

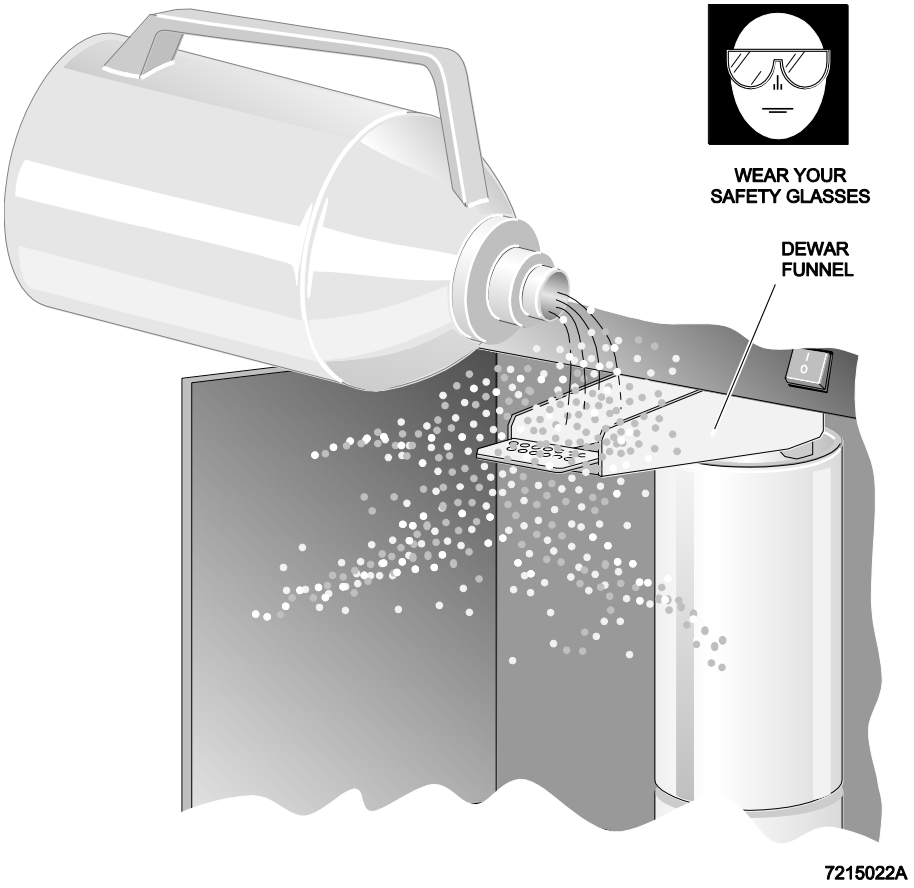


Figure 7.22 Using the Dewar Funnel to Fill the Dewar

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Figure 7.23 Installing Dewar Lid

During Analysis

While sample analysis is in progress, the isotherm is displayed in the central area of the **Analysis** screen, and the current status is displayed on the Analysis Status key.

Results for different calculations (if more than one is selected in the profile) appear on the **Analysis** screen when ready. A result on the screen does not mean the entire analysis is complete.

To view an analysis display other than the one on the screen, press anywhere within the central area of the analysis screen. The **Screen Outputs** screen is displayed, allowing you to choose a different display. If, for example, a BJH ADSORB calculation was chosen along with a BET calculation, the BET is displayed when ready. To restore the isotherm to the screen, press the center of the screen and then choose Isotherm from the available choices on the **Screen Outputs** screen, and press OK.

After Analysis

WARNING

Do not remove the dewar until instructed to do so by a message on the touch screen. Doing so may cause explosive pressures to develop in the sample tube.

At the completion of analysis, results are displayed on the screen for approximately 2 min. Results are then temporarily removed in order to complete the necessary procedure for sample tube removal. This procedure is as follows:

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1. The instrument automatically evacuates the sample tube in preparation for removing the dewar. When this evacuation is complete, the following message appears:

   REMOVE DEWAR
   Press OK when removed

   Remove the dewar at this time:

   a. Remove the insulating dewar lid.

   b. Lift the dewar up to release it from the lower tab of the dewar rack.

   c. Slide the dewar down and out.

   d. Press OK.

   e. After you remove the dewar and press OK the sample tube is automatically backfilled with gas. The following message appears:

      WAIT
      Backfilling sample

      Backfilling takes approximately 2 min.

CAUTION

Do not remove the sample tube until instructed to do so by a message on the touch screen. Doing so may reduce the lifetime of the vacuum system.

---

   f. When backfilling is complete, the following message appears:

      REMOVE SAMPLE
      Press OK when removed

      Remove the sample tube (simply loosen the knurled nut while holding the tube - then pull out the tube).

   g. Press OK.

   h. The following message appears:

      IS SAMPLE WEIGHT OK?
      [entered weight] g,

      along with YES and NO keys, to allow you to correct any clerical errors made in entering the sample weight, or enter the outgassed weight if you neglected to enter it prior to analysis.

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- If the sample weight displayed is correct: Press YES.
- If the sample weight displayed is incorrect: Press NO, a numeric keypad appears allowing you to enter the correct sample weight.

An analysis report and analysis data file in ASCII format are written to the floppy disk (filenames *.RPT and *.DAT).

To change the results displayed on the screen, press the central area and make a choice from the **Screen Outputs** screen.

7.13 PRINTING RESULTS

General

If a printer is configured (see heading 7.3, SA 3100 Analyzer Configuration) an analysis report can be automatically printed at the completion of an analysis. In the definition of a profile, you determine whether to print a full report, a report summary, or not to print after analysis. Regardless of the profile setting for automatic printing, however, you can print existing results at any time.

- During an analysis, the existing results may consist only of the isotherm, if no calculations have been completed.
- After analysis, you can print an additional copy of results as long as no new analyses have been started.
- If a printer was not configured for an analysis, you can configure a printer and then print out the results of the last analysis if you have not started a new analysis.
- Graphs are only printed on laser printers which support the HPGL2 printer language.

Full Reports and Report Summaries

A full report consists of all data and calculation results from an analysis, in tabular and graphic form where applicable. A full report also begins with a report summary. A report summary consists only of the results of the calculations selected for an analysis. A report summary does not include the freespace data, the isotherm, or any graphs. For more flexibility in printing, for instance to print the results of one calculation and not others, or to print just one graph, use the SA-VIEW program, see Appendix A, COULTER SA-VIEW Software.

Printing

To print results:

1. Press Setup in either the **Analysis** or **Outgas** screen. The **Setup and Print** screen, Figure 7.1, is displayed.

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2. Press Print Summary in the **Setup** screen to print a report summary, or press Print Full Report. Currently available results are printed.

7.14 ACCESSING REPORT AND DATA FILES

General

Data from analyses are saved automatically onto the diskette in the floppy disk drive. Two files are saved for each analysis: the raw data and the report. The report is identical to that printed, except that it contains no graphs. Both files are in ASCII format and can be read by any text editor. The files are assigned a filename by the instrument; data files have a .DAT extension, and report files have a .RPT extension.

COULTER SA-VIEW Software

The most convenient way to access SA 3100 analyzer results is by using SA-VIEW software, see Appendix A, COULTER SA-VIEW Software. This Microsoft Windows-based program allows you to view data files in graphic and tabular format, compare data files, and copy graphs and listings to other Windows applications.

Using the Analysis Log File

If you do not have access to a computer running Windows 3.1 or Windows 95, you can read SA 3100 analyzer results using any text editor. To access a particular file, you need to use the analysis log file (ANALYSIS.LOG), which connects the SA 3100-assigned filename to the SAMPLE ID, CUSTOMER and PROFILE entered for the analysis, in addition to the date and time the analysis was started. The status of a file is also noted in the analysis log: complete, or interrupted by an error or power failure.

To read the analysis log, load ANALYSIS.LOG into a text editor from the diskette. The files are listed in order of the time analyses were started. The order of columns is as follows:

File SAMPLE ID CUST. PROFILE DATE TIME STATUS

Identify the file you wish to view, then load that file into the text editor.

7.15 ADVANCED SETUP

General

The **Advanced Setup** screen allows you to set the date, time, and the filenames to be used by the SA 3100 analyzer to save data. It also contains the Profile Lock feature, which prevents users from creating, modifying, or deleting profiles, and the demonstration feature, which simulates running the instrument without actually driving the hardware. There is one other setting in the **Advanced Setup** screen which should only be used by Beckman Coulter personnel: the hardware revision code.

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Components of Advanced Setup

Demonstration

This setting is intended primarily for Beckman Coulter personnel. The Demonstration key allows you to choose Yes or No. If you select Yes, the user interface will simulate outgassing and analysis without actually running the instrument. It can be used for demonstration and training purposes. The default setting is No.

Profile Lock

The Profile Lock allows you to lock the profile creation, edit, and delete features, so that no further changes can be made to profiles until you change the setting again. To engage the Profile Lock change the setting to Yes. To release the Profile Lock return the setting to No. The default setting is No. Engaging the Profile Lock does not prevent access to profiles through Edit Profiles in the **Setup and Print** screen, but any changes made will be ignored by the system.

Time

The Time setting allows you to change the hour, minute, and seconds of the system clock so that the times of analyses will be correctly recorded and saved with data files. The clock is a 24-h clock, so 2:00:00 pm should be entered as 14:00:00; if you do not use the 24-h format and you change the time in the afternoon, the date will change at midday rather than at midnight. The default setting is local time in the Eastern Time Zone, USA.

Date

The date setting allows you to change the month, day, and year setting so that the dates of analyses will be correctly recorded and saved with data files.

Hardware Revision

The hardware revision is a code that is unique to each instrument. Changes should be made only by, or when directed by, Beckman Coulter personnel.

Next File Name

The SA 3100 analyzer does not save files under the Sample ID you enter, but assigns a name using the filename format set in Next File Name (the analysis log allows you to determine the filename from the Sample ID). Using Next File Name you can specify up to 8 characters. Use digits at the end of the filename to provide a basis for the instrument to increment filenames and prevent overwriting of data. For example, if 'SA0' is the Next File Name, files will be saved as SA0.dat and SA0.rpt (data and report files, respectively), SA1.dat and SA1.rpt , etc., until the disk is full or SA999999.* is reached. Alternatively you can use a name such as BET5QC00, in order to increment as BET5QC01.*, BET5QC02.*, etc., to BET5QC99.

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Changing Advanced Setup Settings

To change settings in advanced setup:

1. Make sure that no outgassing or analysis functions are in progress. If the instrument is performing any functions, wait until samples on outgas ports are outgassed and analysis in progress is complete.

2. Turn off the SA 3100 analyzer. When you turn on the instrument again, the Advanced Setup key will appear in the bottom quarter of the screen at the end of the boot-up sequence for approximately 1 s before the **Analysis** screen appears.

3. Turn on the SA 3100 analyzer. When the Advanced Setup key appears, press it, the Advanced Setup screen displays, Figure 7.24.

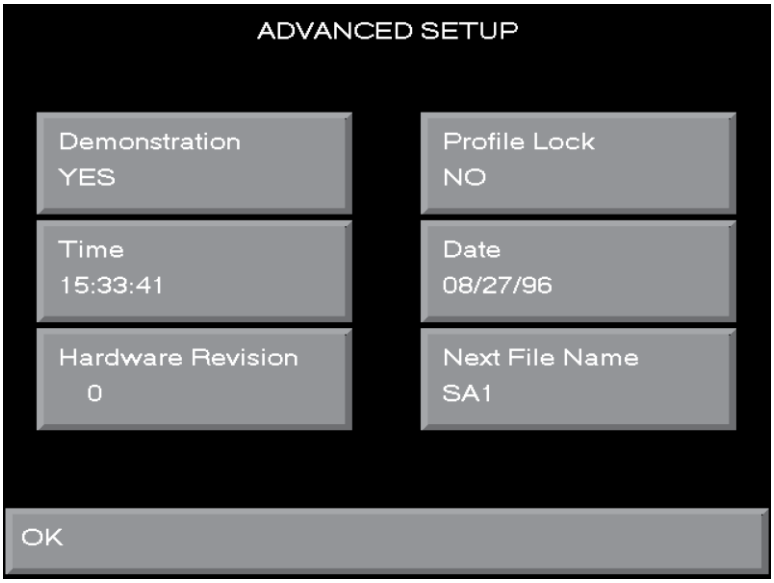


Figure 7.24 Advanced Setup Screen

CAUTION

Do not press the Hardware Revision key. Changes should be made only by, or when directed by, Beckman Coulter personnel.

4. Each key in the Advanced Setup screen will take you to a screen which allows you to change the setting. Change settings as required.

5. Press OK, the **Analysis** screen displays and the instrument will initialize. Your settings will be in effect until you change them; they will not be affected by turning the instrument off and then on again.

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8.1 GENERAL

These cleaning and maintenance procedures for your SA 3100 analyzer are organized by frequency. The frequency varies from per use in the case of cleaning sample tubes to per year or less frequently in the case of replacing gas cylinders.

8.2 CLEANING SAMPLE TUBES

Frequency

Before each use.

General

The entire sample tube assembly must be clean, and most importantly, dry, before each use. It must be completely free of moisture in order to obtain a correct dry weight of the tube assembly before adding sample. This dry weight is subtracted from the outgassed weight of the tube assembly and sample after outgassing to determine the true sample weight. Therefore, if the dry weight is incorrect, the sample weight will be incorrect, and your analysis results will suffer accordingly.

The sample tube caps should be kept free of dust, but do not need regular cleaning.

Note. Unless the sample tube assembly is weighed immediately after drying (see heading 7.7 Weighing the Sample Tube Assembly), the assembly should be stored in a drying or desiccating cabinet.

Procedure

Cleaning

1. Clean the sample tubes and tube inserts with warm water and laboratory detergent in an ultrasonic bath. Leave the glassware in the bath until clean (at least 5 minutes).

2. Rinse the tubes and inserts with filtered water.

3. Place the tubes in a rack upside down to drain bulk water.

Drying

There are a few methods that can be used to dry sample tubes. Inserts will dry sufficiently in open air.

Method 1.

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Bake in an oven at 105° C for 30 min.

Method 2.

1. Swirl a small quantity of acetone or other water soluble solvent in the tubes, and drain into an appropriate waste container.

2. Place the tubes in a dry air stream under a fume hood until all solvent has evaporated.

Method 3.

Dry in a vacuum oven.

8.3 CLEANING AND GREASING O-RINGS

Frequency

Check daily for cleanliness.
Grease weekly.

General

The O-rings in the sample port and outgassing ports must be checked for cleanliness daily, and greased weekly to ensure leak-free performance.

Procedure

CAUTION

Do not use any tools to remove or install port fittings. Fittings are designed to seal when finger-tight.

1. See Figure 8.1. Remove the knurled nut from the port. Ensure that there is a sleeve and O-ring inside the nut. If the O-ring is not inside the nut, it may still be inside the fitting, and may be removed with a small blunt object.

2. Check the O-ring for dust, particles, or wear. If the O-ring is worn or damaged, replace the O-ring. If dirty, wipe the O-ring clean with lint-free laboratory tissue.

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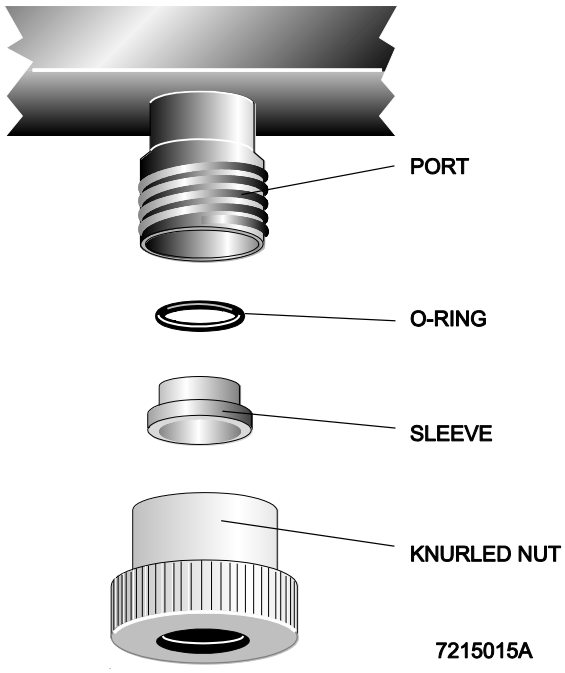


Figure 8.1 Port Fittings

IMPORTANT

Excess grease on O-rings will be deposited on sample tubes, causing weighing and analysis errors.

3. If the O-ring does not appear slightly wet, it needs to be greased. Grease the O-ring very lightly with a thin film of high vacuum silicone grease. The surface of the O-ring should appear to be just slightly wet. There should be no excess grease on the O-ring.

4. See Figure 8.2. Slide the knurled nut, sleeve (ensure the sleeve is narrow side up), and O-ring over the end of a clean sample tube.

5. Insert the tube into the port until it stops against the manifold, then slide the knurled nut up the tube and tighten until finger-tight. The O-ring is now seated.

6. Loosen the nut 1/4 turn and remove the tube.

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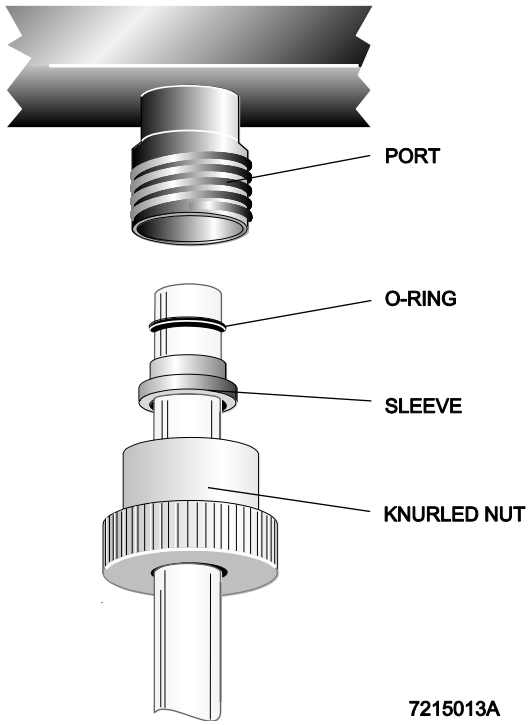


Figure 8.2 Seating the O-ring

8.4 CHECKING VACUUM PUMP OIL

Frequency

Check daily for level and cleanliness.

Change every six months or when oil appears cloudy or discolored.

General

The vacuum pump oil should fill 3/4 of the oil sight glass, and should be clear. The oil must be replenished when the level is low, and changed every six months or when the oil appears cloudy or discolored.

Adding Oil

1. Turn off the power to the instrument.

2. Lift the vacuum pump out of the pump shelf and place it on the table or bench immediately behind the unit.

3. See Figure 8.3. Remove the oil fill plug.

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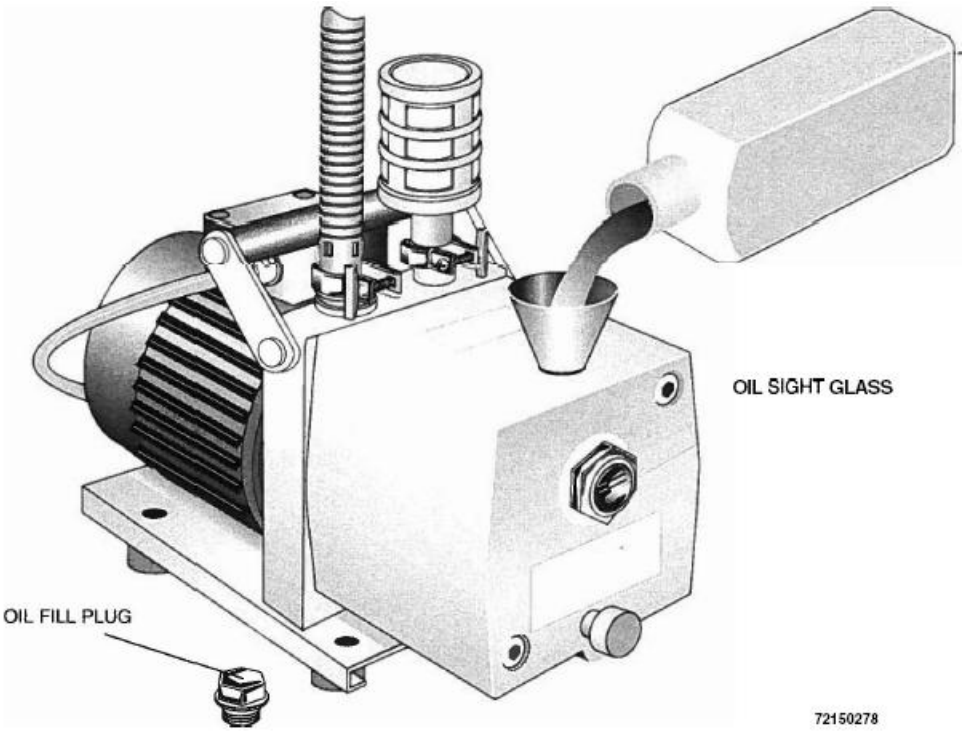


Figure 8.3 Adding Vacuum Pump Oil

CAUTION

Use only Balzers P3 pump oil in the vacuum pump. Use of other oil may damage the pump and will void the pump warranty.

4. Add Balzers P3 pump oil until the sight glass is 3/4 full.
5. Replace the oil fill plug and tighten until finger-tight - do not use a wrench.
6. Replace the vacuum pump in the pump shelf, fitting the rubber feet into the provided depressions.
7. Restore power to the unit.

Changing Oil

1. Turn off the power to the instrument.
2. Turn the SA 3100 analyzer so that when the pump is removed from the pump shelf, the oil drain plug will be next to the edge of the table or bench to facilitate draining.

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3. Lift the vacuum pump out of the pump shelf and place it on the table or bench immediately behind the unit with the oil drain plug facing the edge of the table. Do not stretch the vacuum line.

4. See Figure 8.4. Remove the oil fill plug.

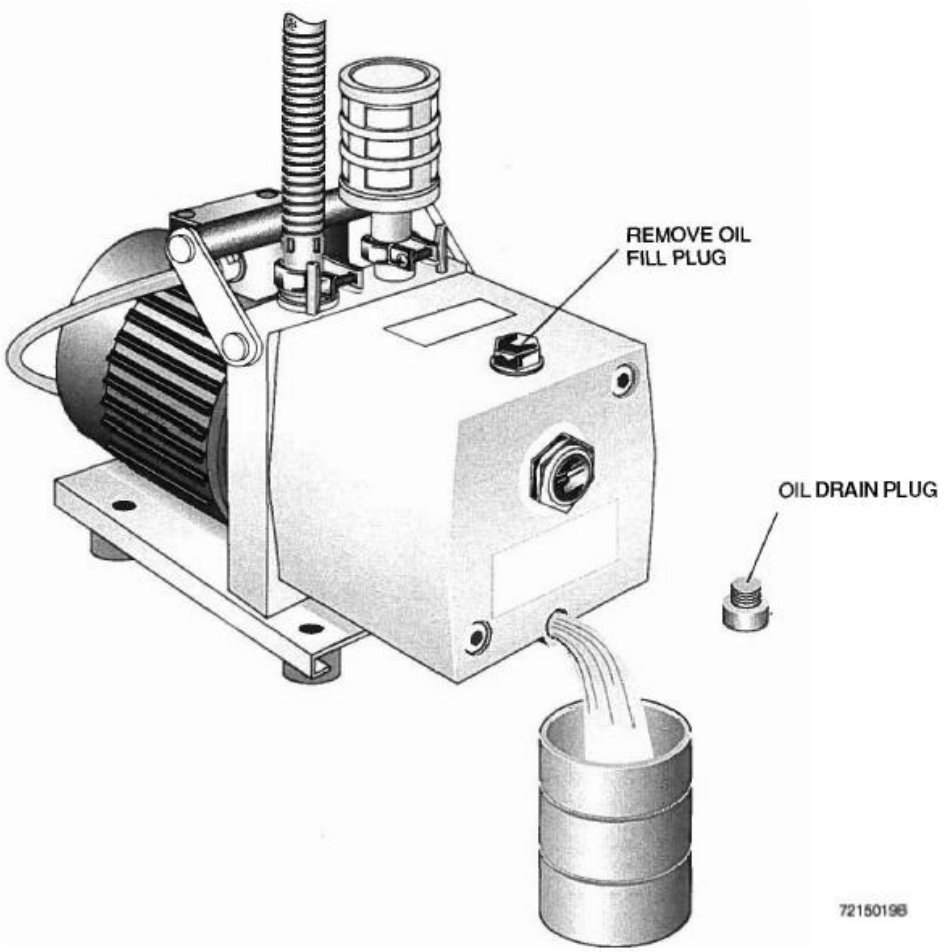


Figure 8.4 Draining Vacuum Pump Oil

5. Place a 500 mL waste container below the oil drain plug and remove the plug. Allow oil to drain completely.

   Dispose of oil properly. Used vacuum pump oil may contain traces of sample material, and therefore must be treated as if contaminated with any hazardous or toxic materials that have been measured by the instrument.

6. Replace the oil drain plug and tighten until finger-tight. Do not use a wrench.

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CAUTION

Use only Balzers P3 pump oil in the vacuum pump. Use of other oil may damage the pump and will void the pump warranty.

7. See Figure 8.3. Fill the pump with oil until the sight glass is 3/4 full.
8. Replace the oil fill plug and finger-tighten. Do not use a wrench.
9. Replace the pump in the pump shelf, fitting the rubber feet into the provided depressions.
10. Restore power to the unit.

8.5 CLEANING THE FAN FILTER

Frequency

Every six months or when visibly dirty. If the SA 3100 analyzer is in an environment with airborne dust, the filter may require cleaning more often.

Procedure

1. Turn off the power to the SA 3100 analyzer.
2. The fan guard is located on the rear of the instrument, in the upper left corner. The fan guard is held in place by four tabs. Pull up on the upper tab to release the fan guard and then pull the guard off.
3. Remove the filter and rinse in water. See Figure 8.5.

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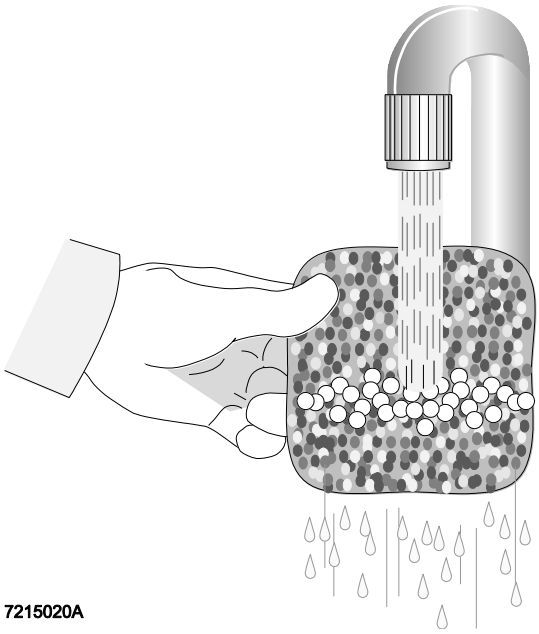


Figure 8.5 Rinsing the Fan Filter

4. Dry the filter with a paper towel.

5. Replace the filter in the fan guard and press-fit the fan guard onto the fan housing.

6. Restore power to the SA 3100 analyzer.

8.6 CHANGING THE OIL MIST FILTER

Frequency

Once a year.

Procedure

1. Turn off the power to the SA 3100 analyzer.

2. Lift the vacuum pump out of the pump shelf and place it on the table or bench immediately behind the unit. Do not stretch the vacuum line.

3. See Figure 8.6. Remove the clamp holding the filter and discard the used filter.

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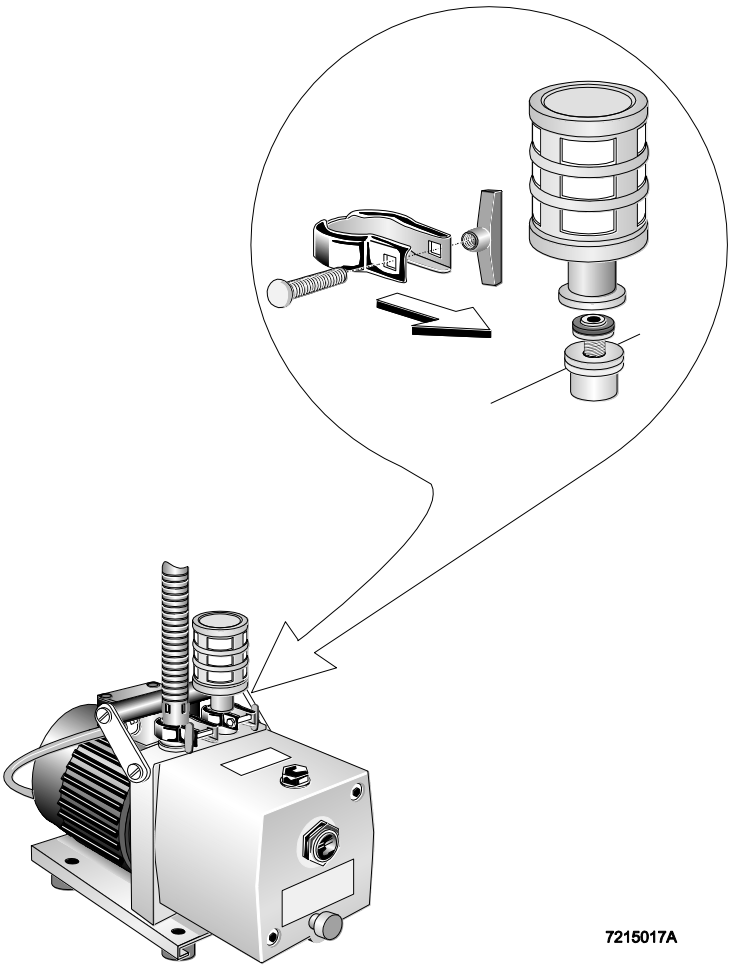


Figure 8.6 Oil Mist Filter Installation

4. Clamp on a new filter and replace the pump in the pump shelf, fitting the rubber feet into the provided depressions.

5. Restore power to the unit.

8.7 CHANGING GAS CYLINDERS

General

This procedure is applicable if you are using gas cylinders with the optional pressure regulator kit supplied by Beckman Coulter. Refer to Chapter 2, Precautions and Hazards, heading 2.3 Compressed Nitrogen and other Inert Gases, before performing this procedure.

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WARNING

This procedure is NOT to be used for replacement or installation of gas cylinders containing corrosive or hazardous gases.

Frequency

When cylinder pressure drops below 100 psig. Unless more than one SA 3100 analyzer is being serviced by the gas cylinder, frequency is unlikely to be more often than yearly.

Procedure

1. Refer to Figure 8.7. Close the cylinder valve by tightening the knob or other mechanism clockwise.

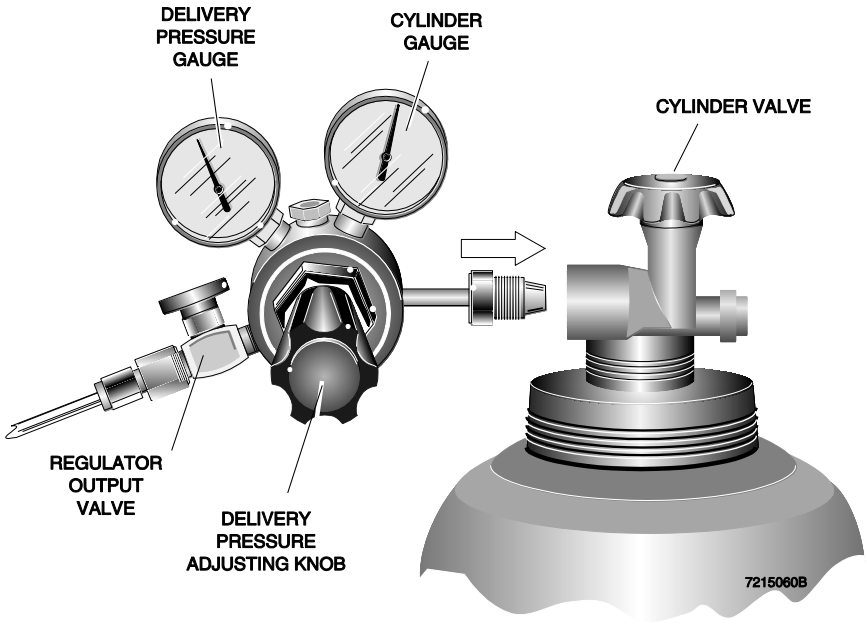


Figure 8.7 Gas Cylinder and Pressure Regulator

2. Close the regulator output valve by turning the small wheel clockwise.

3. Close the delivery valve by turning the delivery pressure adjusting knob counterclockwise until it turns freely.

4. Use a 11/8-inch AF (Across Flat) wrench to loosen the regulator connection, then unscrew the regulator by hand.

5. Carefully inspect the regulator for signs of contamination or damage. Examine all exposed and connecting threads for signs of wear or abuse.

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6. Make sure the cylinder valve on the new cylinder is closed by turning the knob or other mechanism clockwise.

7. Remove the used cylinder and secure the new cylinder.

WARNING

To prevent valve damage and personal injury, do not force the regulator-cylinder connection. If the regulator does not screw into the cylinder valve outlet easily, check for cross-threading or contamination.

CAUTION

Never use oil, grease, or Teflon tape on any cylinder or regulator valve or connection threads.

---

8. Screw the regulator onto the new gas cylinder valve outlet by hand. Never force a regulator connection. When the connection is hand tight, use the 11/8-inch AF wrench to secure the connection by tightening approximately 1/8 turn clockwise.

9. Open the cylinder valve SLOWLY. When opening a valve, always turn the knob counterclockwise until it stops, then clockwise 1/4 turn. This prevents jamming while ensuring that the valve is completely open. The cylinder gauge (right-hand, high pressure gauge) registers the cylinder pressure.

10. Check for leaks in the regulator-cylinder connection by using soapy water around the threads. Bubbles from the joint will indicate a leak.

    If there are any leaks:

    a. Close the cylinder output valve.

    b. Turn the delivery pressure adjusting knob clockwise to open the regulator.

    c. SLOWLY open the regulator output valve to release pressure within the regulator. Both regulator gauges should descend to zero.

    d. Remove the regulator and inspect the joint components for cleanliness and repeat steps 3 through 7. If there is still a leak, either the regulator or gas cylinder may be damaged. Try another cylinder and repeat steps 3 through 7. If a leak persists, contact your Beckman Coulter Representative.

IMPORTANT

The cylinder pressure must be at least 100 psig.

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11. Make a note of the cylinder pressure.

12. Close the cylinder output valve. The measured pressure should not change. If the pressure changes, see step 10, above, about leaks.

13. Open the cylinder output valve.

14. Turn the delivery pressure adjusting knob clockwise to set the regulator output pressure to between 12 psig and 15 psig.

15. Slowly open the regulator output valve.

16. Purge the external gas lines:

    a. At the instrument, hold the gas line while loosening the Swagelok nut. Stop loosening the nut when you feel the tube starting to back out of the connection (you will also hear the gas escaping). Do not remove the nut.

    b. Hold the tube for 20 to 30 s to purge the tube, then push it back into the connector and finger-tighten the nut. Finger tighten the nut, then use the wrench to tighten the nut approximately 1/2 turn more.

17. Flush the internal gas lines and manifolds:

    a. On the touch screen, press Setup, the **Setup and Print** screen displays.

    b. Press Flush Gas Lines, the key will depress momentarily and you will hear a click. Press OK to return to the **Analysis** screen. This initiates an automatic procedure which flushes both the Helium and Adsorbate gas lines and manifolds inside the instrument. The procedure takes about 5 min per gas line. You can monitor the process by pressing Analysis Status to display the system schematic.

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9.1 ENVIRONMENTAL REQUIREMENTS

Ambient Temperature

Storage

0 to 50°C

Operation

10 to 35°C

Operating Altitude

300m (1000 ft) below MSL to 2100m (7,000 ft) above MSL

Operating/Storage Relative Humidity

10 - 85% RH non-condensing

9.2 POWER REQUIREMENTS

Supply Voltage/Frequency

100-120/220-240 V at 50/60 Hz

Operating Power Consumption

Nominally 500 W

9.3 GAS REQUIREMENTS

Gas Purity

Helium

99.995%

Nitrogen

99.99%

Liquid N_2

99.9%

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9.4 MEASUREMENT SPECIFICATIONS

Specific Surface Area Measurement Range

0.1 m^2/g and higher using nitrogen

Pore Size Range

3 to > 200 nm diameter

Reproducibility

BET Surface Area

< 2% CV

Pressure Measurement Specifications

Overall Pressure Range

Vacuum to 1,000 mm Hg

Minimum Relative Pressure

6×10^{-5}

Sample Pressure Resolution

0.046 mm Hg typical

Manifold Temperature and Stability

45°C ±0.1°C typical

9.5 VACUUM PUMP SPECIFICATIONS

Volume Rate Displacement

1.5 m^3/hr

Ultimate Vacuum

10^{-3} mm Hg

9.6 OUTGASSING SPECIFICATIONS

Number of Outgassing Ports

3

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Furnace Specifications

Temperature Range

40 to 350° C

Temperature Setting Resolution

1° C

Temperature Stability

5° C typical

Temperature Accuracy

±5° C

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APPENDIX A
COULTER SA-VIEW SOFTWARE

A.1 GENERAL

The COULTER SA-VIEW program is a 32-bit Microsoft Windows-based application for reviewing, comparing, and printing data files from the COULTER SA 3100 analyzer. SA-VIEW software enables you to overlay graphs from different analyses, preview printing output, and copy graphs or tabular data to the Windows clipboard for insertion into other Windows applications such as spreadsheets and word processors. SA-VIEW software can be installed on any IBM-compatible PC running Microsoft Windows 3.1 (or higher), or Windows 95. SA 3100 analyzer data files are stored by default on the floppy disk in the SA 3100 analyzer, but they may also be directed to a network server or other computer if the instrument has been configured accordingly. SA-VIEW software can read files from any drive accessible to the computer on which the program is installed. The SA-VIEW program has been implemented in standard Windows format, with pull-down menus, a toolbar, and mouse-linked explanations of menu choices.

For experienced users of Windows, the SA-VIEW program is largely self-explanatory. Nevertheless, all operations *within* the program are explained in this appendix. Certain properties of SA-VIEW software which are common to all Windows applications, however, are *not* covered (see Program Structure, below). Please refer to Windows documentation if you need help with Windows.

A.2 INSTALLATION

General

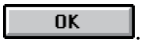
The SA-VIEW program comes on two 3.5" floppy disks. Disk 2 is not required for systems running Windows 95. On systems running Windows 3.1 (or higher) that are already running 32-bit applications, Disk 2 may not be required.

Installation Procedure for Windows version 3.1 or higher

To install the program in Windows version 3.1 (or higher):

1. Insert the disk labeled Disk 1 in the floppy disk drive.

2. From the Program Manager or File Manager, select File/Run.

3. In the Command Line Field, type a:\setup and select 

.

Dialog boxes in the Setup Program will inform you of the status of installation and request disk 2 when required. When installation is complete, a new Program Group appears in the Program Manager with the icon for the SA-VIEW program. To start the program, double-click on the icon or press <Enter> when the icon is highlighted.

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Installation Procedure for Windows 95

To install the program in Windows 95:

Insert the disk labeled Disk 1 in the floppy disk drive.

From the Start button on the task bar, select Run.

In the Open Field, type a:\setup and select OK.

When installation is complete, a window containing the icon for SA-VIEW software appears on the desktop. To start the SA-VIEW program, double click on the icon, or press <Enter> when the icon is highlighted. The program is also added to the Programs list, accessible from the Start button on the task bar.

A.3 PROGRAM STRUCTURE

General

See Figure A.1. The principal components of the SA-VIEW program are the menu bar, toolbar, status bar, and workspace of the main window. In the workspace of the main window are open file windows or file icons, if any. In addition, there are standard Windows items common to all Windows applications: the control icon, title bar, and minimize and maximize/restore buttons. The appearance of some of these items is different depending whether Windows 3.1 or Windows 95 is the operating environment. The function of these standard items, which are along the top of main and file windows, will not be explained here; if you are unfamiliar with Windows, refer to Microsoft Windows documentation.

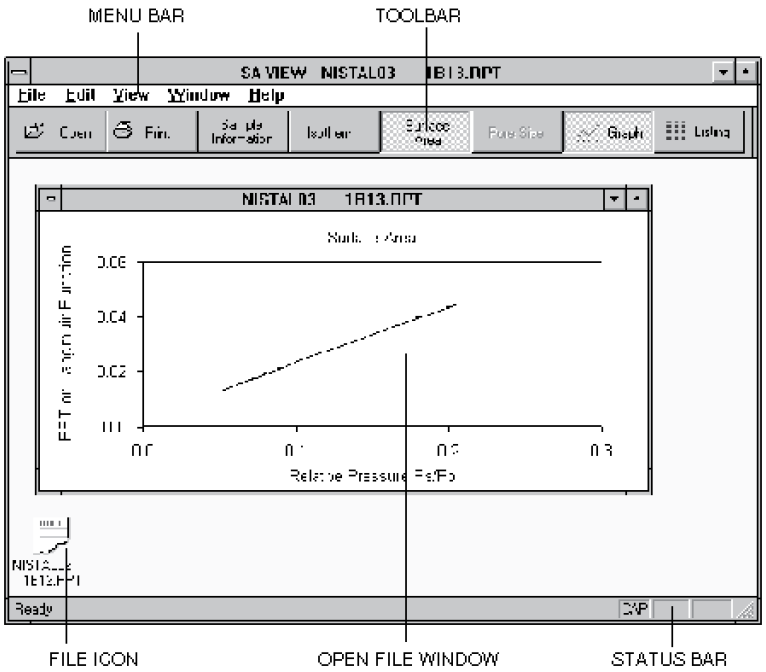


Figure A.l SA-VIEW Program Window

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General information about the menu bar, toolbar, status bar, and file windows and icons in the main window workspace, is in the sections below. Table A.1 shows where in this Appendix to find more specific information about menu items and toolbar buttons.

Menu Bar

On the menu bar up to five menus in the SA-VIEW program are available, depending whether or not any files are open. The File, View, and Help menus are always available; the Edit and Window menus are only available when one or more files have been opened. All operations in the program can be accessed via the menus. The commands or options in the menus can be viewed and activated in a few different ways, using the mouse or keyboard. These techniques for accessing and activating menu items are standard in Windows programs, but they also are explained below.

Viewing Menus

You can view a menu either by clicking on the menu title using the mouse, or by pressing <Alt> and then the underlined letter in the menu title using the keyboard.

Activating Menu Commands or Options

Once a menu is in view, you can activate an item in that menu by:

- Clicking on the item with the mouse.
- Scrolling up or down the menu using the keyboard arrow keys and then pressing <Enter> when the desired item is highlighted.
- Pressing the underlined letter or number in the item (the menu item access character) with the keyboard.

Using Keyboard Shortcuts

Alternatively, the thirteen more commonly used items in the File, Edit, and View menus can be directly activated using keyboard shortcuts *without* first viewing the menu. These items have a code next to the item in the menu consisting of Ctrl+[capital letter]. This code means that to activate the item, hold the <Ctrl> key down while pressing the indicated letter. The letter to use with <Ctrl> is the same as the underlined letter in the menu item with one exception: the Total Pore Volume item in the View menu uses 'T' to avoid conflict with another item using the letter P, the Print... item in the File menu. The keyboard shortcut codes are listed in Table A.1. A subset of eight of the thirteen items with keyboard shortcuts can also be activated using the toolbar buttons, see below.

Activated Options

Activated options in menus are denoted by a check next to the option.

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Ellipses

An ellipsis ('...') after a menu item indicates that selection of the menu item leads to a dialog box that either requires further choices or provides information. Selection of menu items without ellipses results in the immediate activation of the command or option.

Toolbar

On the toolbar are eight buttons which provide direct access with a mouse click to the most commonly required options or commands. Table A.1 shows which menu items correspond to the toolbar buttons and where to find explanations of these items. You can choose whether to display or hide the toolbar.

Status Bar

The status bar along the bottom of the main window has three functions:

- To indicate the status of the SA-VIEW program, such as 'Ready'.
- To provide you with a brief explanation of the function of a highlighted menu item, or toolbar button if you are currently pointing at the button or clicking on the button with the mouse.
- To display the status of the keyboard functions Caps Lock, Num Lock, and Scroll Lock - if any of these are activated, the fields on the right of the status bar display CAP, NUM, and/or SCRL as appropriate.

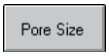
You can choose whether to display or hide the status bar.

Files and Icons in the Main Window Workspace

When you open a file in the SA-VIEW program it appears in the workspace in a file window. You can change the type of data, or the form of data displayed, using the menu items or toolbar buttons. You can copy graphs or tabular data to the Windows clipboard, or print it out in various formats. You can also compare two or more different data sets graphically in the same file window. All of these types of operations are covered in this appendix. You can also manipulate the size and shape of these windows, or minimize them into icons, using standard Windows operations.

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Table A.1 Menu Items in The SA-VIEW program

Menu	Command/Option	Keyboard Shortcut	Toolbar Button	Explanation on page...
File				
	Open...	Ctrl+O		A-6
	Close*	None	None	A-12
	Overlay...*	None	None	A-8
	Print...*	Ctrl+P		A-10
	Print Preview*	None	None	A-10
	Print Window*	None	None	A-12
	Print Setup...	None	None	A-10
	1, etc. Most Recent Files*	None	None	A-8
	Exit	None	None	A-14
Edit*				
	Copy	Ctrl+C	None	A-12
View				
	Sample Information*	Ctrl+M		A-8
	Report Summary*	Ctrl+R	None	A-8
	Isotherm*	Ctrl+I		A-8
	Surface Area*	Ctrl+S		A-8
	Film Thickness*	Ctrl+F	None	A-8
	Total Pore Volume*	Ctrl+T**	None	A-8
	Pore Volume Distribution*	Ctrl+V		A-8
	Pore Area Distribution*	Ctrl+A	None	A-8
	Graph*	Ctrl+G		A-8
	Listing*	Ctrl+L		A-8
	Toolbar	None	None	A-4, A-13
	Status Bar	None	None	A-4, A-13

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Menu	Command/Option	Keyboard Shortcut	Toolbar Button	Explanation on page…
Window*				
	New Window	None	None	A-13
	Cascade	None	None	A-13
	Tile	None	None	A-13
	Arrange Icons	None	None	A-13
	1, etc. Currently Open Files	None	None	A-13
Help				
	About SA-VIEW...	None	None	A-13

*Menus or menu command/options not visible unless a file is open.
**Keyboard shortcut uses a different character from the menu item access character.

A.4 USING THE SA-VIEW PROGRAM

General

This section of the appendix gives instructions for using the SA-VIEW program. When a toolbar button can be used instead of a menu selection, it is pointed out in the text. Keyboard shortcuts, however, are not mentioned; refer to table A.1 for keyboard shortcuts. When a menu selection is referred to, the syntax 'Menu/Selection' is used. For example, to open a file, you can select Open from the File menu - this instruction is written 'select File/Open'.

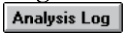
Opening Files

You can always open a file using the standard procedure, below. Once a file has been opened and then closed, however, it enters the list of recently opened files, and can be opened directly from the File menu, see Opening Most Recent Files, below.

Opening Any File

To open any file:

1. Select File/Open or click 

, the Open Report File Dialog box displays, Figure A.2. The Open Report File Dialog box is a standard Windows File Open dialog box with the exception of the 

button. As the SA 3100 analyzer report files listed in the dialog box are named by the instrument and are not the same as the Sample ID's assigned by you, the Analysis Log allows you to pick the file based on the Sample ID or other fields from the **Sample Information** screen. If you know the file you wish to open by the filename assigned by the SA 3100 analyzer, select the file and select 

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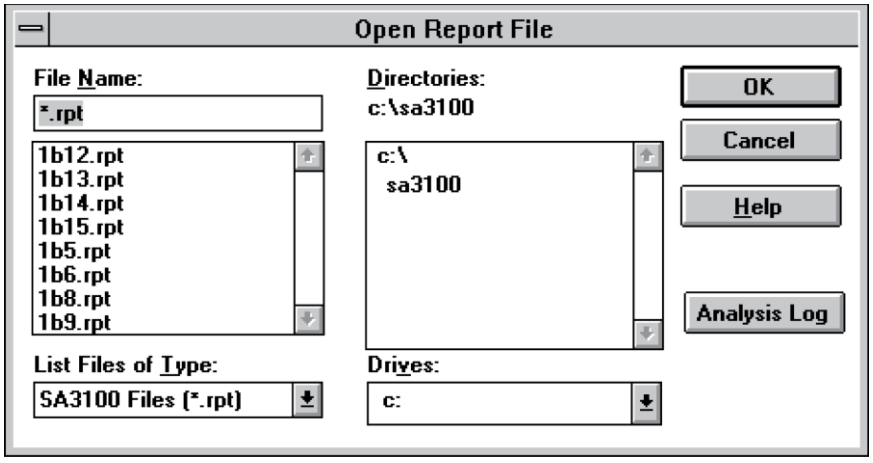


Figure A.2 Open Report File Dialog Box

2. If you do not know the name of the desired file as assigned by the instrument, select Analysis Log. The Analysis Log displays, Figure A.3. The Analysis Log lists the files by Filename, the Sample ID field (Sample), the Customer field, the Operator field, the Profile used, the Date and Time the analysis was started, and the status of the report (completed, aborted, etc.).

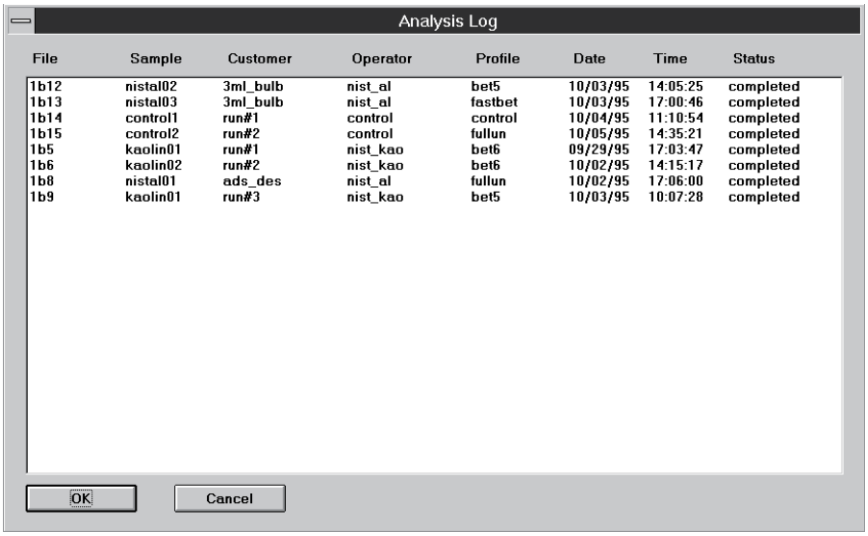


Figure A.3 Analysis Log Dialog Box

3. To select the file from the Analysis Log, highlight the file and select OK. Select OK in the Open Report File Dialog box to open the file. Alternatively, double-click with the mouse on the desired file in the Analysis Log, and the file will open directly.

Multiple data files can be open in the main window workspace at a time.

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Opening Most Recent Files

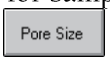
The four most recently opened files are listed in the File menu, numbered starting with the most recently opened file.

To open a recently open file, select File, and then select the desired file from the list in the menu. The number of the file in the list is underlined, so to re-open the file that was last closed, for example, you can press Alt+F, 1.

Viewing Files

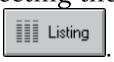
When you open a file, the Sample Information is the default display. At a minimum, an SA 3100 analyzer report file consists of Sample Information and Isotherm data. Most of your files will have one or more calculations stored with them and you can view any of these using the SA-VIEW program. The selections in the View menu and on toolbar buttons for data types *not* present in the active (highlighted) data file window appear as gray, and cannot be selected.

To change the viewed data type in an open data file:

1. Select the View menu and select the desired data type item, or use the toolbar buttons. Toolbar buttons are available for Sample Information, Isotherm, Surface Area, and Pore Volume Distribution (

).

2. Choose either Graph or Listing display by selecting View and selecting the appropriate option, or by using the toolbar buttons 

 and 

. These options apply to each window in the workspace separately; one window can be in Graph view while another is in Listing View. The toolbar buttons and View menu selections reflect the options for the active (highlighted) window.

Overlaying Files

Once a file is open, you can overlay other files in the same window to compare graphic data. If multiple files are open in the main window workspace, the highlighted file is the one into which overlaid files will be placed. The Sample Information, and all data when viewed in Listing form, will be from the original file in the window. Only the graphs will show the original *and* overlaid data.

To overlay files on an open file:

1. Select File/Overlay, the Overlayed Results dialog box appears, Figure A.4. This dialog box is the same as the Open Report File dialog box, with the addition of the Files in Group section at the bottom of the box . If the window into which you are overlaying already has files overlaid on the original window, these will be listed under Files in Group.

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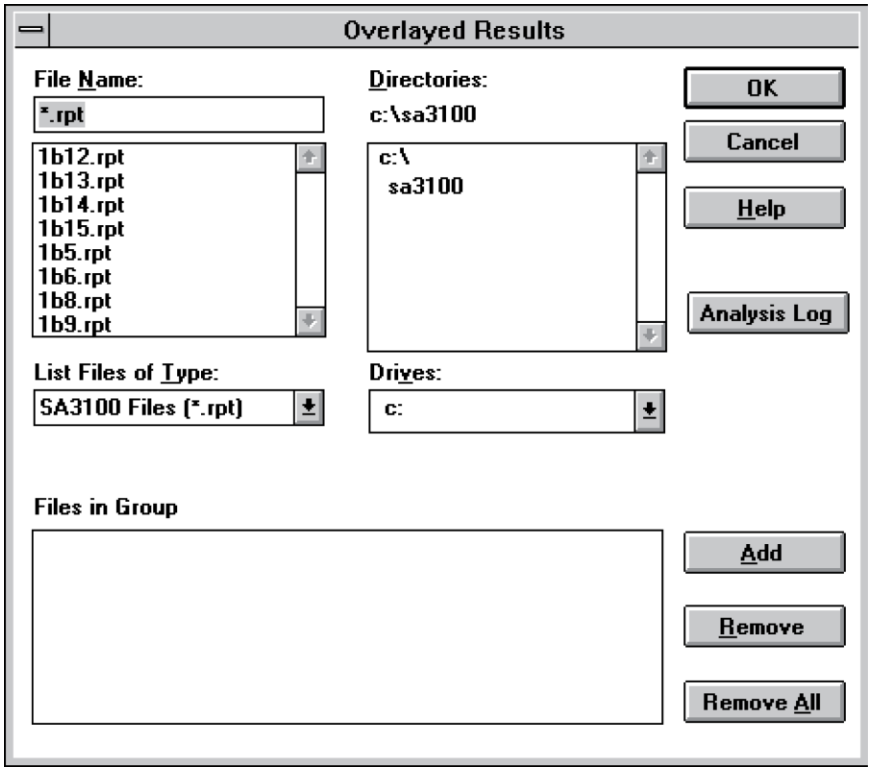


Figure A.4 Overlayed Results Dialog Box

2. If you know which file(s) you want to overlay based on the filenames assigned by the SA 3100 analyzer, select each filename from the filename list and select Add.

3. If you need to refer to the Analysis Log to select the files for overlay, select Analysis Log. Highlight the file to overlay and select OK, then select Add in the Overlayed Results dialog box. Alternatively, double-click on the file in the Analysis Log to add it directly to the list.

4. When you have added all files you wish to compare to the Files in Group list, select OK.

To remove an overlaid file from a window:

1. Select File/Overlay, the Overlayed Results dialog box appears, Figure A.4.

2. Highlight the file to remove in the Files in Group list, then select Remove. Repeat to remove other files. All overlaid files can be removed at once without highlighting by selecting Remove All.

3. Select OK.

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Printing Files

To print from the SA-VIEW program, the file you want to print from must be open in the main window workspace. If more than one file is open, data from the active (highlighted) file window will print. There are two printing options in the SA-VIEW program: you can print a complete file report or a section of it, or you can print the data currently displayed in the file window.

Setting up to Print

If a default printer has been selected in Windows, the SA-VIEW program will automatically send printing output to this printer. To send printing output to another printer, select File/Print Setup, the Print Setup dialog box displays. This is a standard Windows dialog box which allows you to select a printer other than the default choice from the list of installed printers, if any, and select portrait or landscape output. Other choices in this dialog box depend on the configuration of your system. Refer to Windows documentation or documentation for your printer if you need help installing or configuring printers.

Printing a File Report

Before you print a report, you can view the contents of the complete report by using Print Preview. If you don't want the entire report, Print Preview will help you decide which pages to print.

To use Print Preview:

- Select File/Print Preview, the Print Preview screen appears, see Figure A.5. The pages of the report are displayed exactly as they will print, with the pages numbers of the currently displayed pages listed on the status bar. Note that the buttons on the Print Preview toolbar have underlined letters; these buttons can be activated without the mouse by holding down the <Alt> key and pressing the underlined letter.

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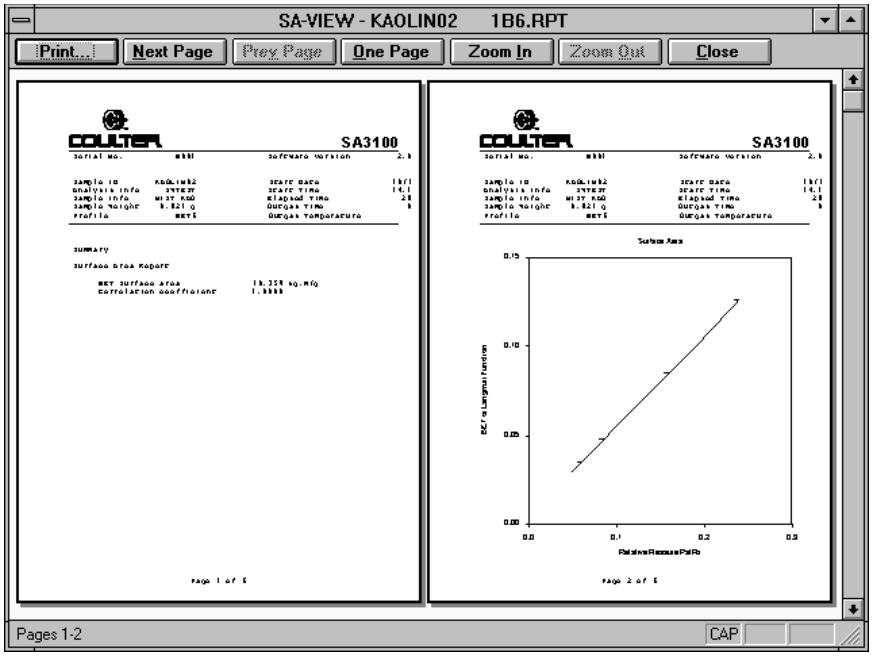


Figure A.5 Print Preview Screen

- To scroll through the pages of the report, use the PgUp and PgDn keys on the keyboard, the vertical scroll bar, or the Next Page and Prev Page buttons.

- To change the display from two-page to one-page view, use the One Page button. After activation, this button changes to Two Page so you can switch back.

- To magnify the display so you can examine a region more closely, click with the mouse on the region you want to look at (the pointer looks like a magnifying glass when it is over the page), or use the Zoom In button. There are two levels of magnification - zoom in a second time to increase the magnification. The Zoom Out button will reduce magnification from the first level, but using the mouse you must zoom in to the second level before a click takes you back to the original display.

- To close the Print Preview screen *without* printing, use the Close button.

- To print, use the Print... button, the Print Preview screen closes and the Print dialog box appears. This is a standard Windows dialog box, allowing you to select how much of the report you want to print in the Print Range box. Select All to print the entire report, or select Pages and enter a page range to print a section of the report. Select OK to start printing.

To print without using Print Preview:

- Select File/Print, or use Print, the Print dialog box appears. This is a standard Windows dialog box, allowing you to select how much of the report you want to print in the Print Range box. Select All to print the entire report, or select Pages and enter a page range to print a section of the report. Select OK to start printing.

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Printing Window Contents

When a file is open in the main workspace area, the contents of the window and the type of display are determined by the selections in the View menu (you may have made these selections using the toolbar). Using the Print Window command, you get the window contents in the form currently displayed (graph or listing).

To print window contents:

- Select File/Print Window, the Print dialog box appears. This is a standard Windows dialog box. Select OK to start printing. Note that this is the same Print dialog box that appears under File/Print, allowing you to decide to print fewer pages than the entire window if the window contains tabular data. Print Preview is not available for Print Window, however, so if you *do* want to print less you have to estimate the contents of a page based on the number of lines per page for your printer and paper size.

Copying Data to the Windows Clipboard

With the SA-VIEW program you can copy data from the currently active (highlighted) file window to the Windows clipboard, allowing you to paste graphs or listings into other Windows applications such as spreadsheets or word processors.

To copy data:

1. Select the data you want to copy in the file window using the View menu or toolbar buttons, including whether you want a graph or listing.

2. Select Edit/Copy. The data is now on the Windows clipboard, and can be pasted into another Windows application.

Closing Files in the Workspace

You do not have to close the active file in the workspace to open another; many files can be open at one time in the SA-VIEW program. The more files you have open, however, the more memory is used by the system and the more cluttered the workspace is.

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To close a file you can use any of the standard Windows techniques:

- Select File/Close, or press Ctrl+F4 to close the active (highlighted) file window.
- Double-click the Control Icon in the upper left hand corner of the file window.
- Access the control menu of the file window and select Close.

Arranging the Workspace

The SA-VIEW program, like most Windows applications, is very flexible in allowing you to arrange the workspace. You can remove the toolbar or status bar from view to increase the available space for viewing files. You can maximize the active file window to fill the available space, change the size of the window, or reduce it to an icon. You can arrange the open file windows in cascade (overlapping) or tile fashion. You can also duplicate an open file window to view two parts of a file, or two data types, simultaneously.

Viewing or Removing the Toolbar or Status Bar

To remove or restore the toolbar or status bar, select View and select the Toolbar or Status Bar options; a check next to one of these options indicates that it is currently in view.

Maximizing, Minimizing, Changing Window Size/Shape

Use the file window title bar, minimize button, maximize/restore button, control menu, or mouse to manipulate the size, shape or position of the window, or reduce it to an icon, using the standard Windows conventions.

Arranging Windows or Icons

The Window menu has the standard Windows options for arranging windows in the workspace, Cascade (overlapping) and Tile. Arrange Icons can be used to clean up the position of icons in the workspace.

Changing the Active Window

As in all standard Windows applications, you can switch the active window in various ways: by clicking on another window with the mouse, by selecting another window from the open file list in the Window menu, or by pressing Ctrl+F6 to cycle through the open windows or icons sequentially.

Duplicating the Active Window

If you wish to duplicate the active file window so that you can view two parts of a file, or two data types, simultaneously, select Window/New Window. The options in the View menu apply to each window in the workspace separately. The toolbar buttons and View menu selections reflect the options for the active (highlighted) window.

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Accessing the COULTER SA-VIEW Program Copyright Dialog Box

To access the copyright dialog box, select Help/About SA-VIEW..., the copyright dialog box appears, see Figure A.5.

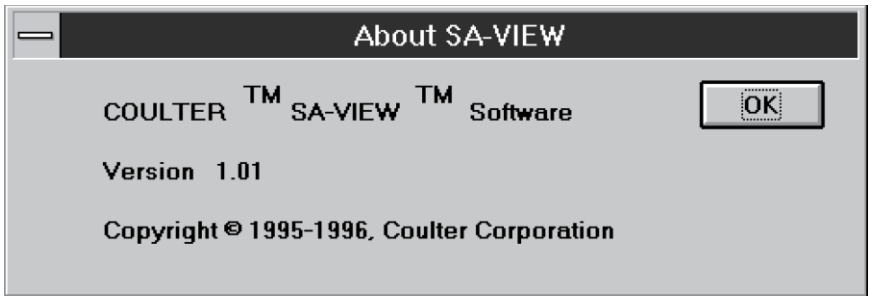


Figure A.6 SA-VIEW Program Copyright Dialog Box

Exiting the Program

To exit the SA-VIEW Program, select File/Exit.

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APPENDIX B
OUTGASSING NOTES

B.1 General

The sample preparation or sample outgassing procedure is necessary to achieve reproducible and accurate surface area and pore size data. The outgassing process removes water vapor and other adsorbed gases /vapors from the sample surface. Mostly water vapor is removed. The SA 3100 series instruments have three integral vacuum outgassing stations. Also available from Beckman Coulter is the SA-PREP Surface Area Outgasser which may be used to increase surface area sample throughput. This six-station unit uses a flowing gas method instead of vacuum.

B.2 Outgassing Temperature

In general choose a higher rather than lower temperature to remove adsorbed water and other vapors. Pick a temperature at which no sample damage will occur and avoid melting temperature-sensitive materials during the outgassing process. Particular care must be given to samples containing water of crystallization or which have water as part of their chemical structure. With this type of sample, overheating can considerably change the results.

CAUTION

If a sample is melted, it will probably sublime within the instrument and vacuum pump. This will cause severe operational problems; if this happens contact your Beckman Coulter Service Representative.

B.3 Outgassing Time

General

The higher the temperature, the shorter the outgassing time required. Longer times are needed for samples with higher water content. The vacuum system removes water vapor at a fixed rate dependent upon temperature: the higher the water content the longer the outgassing time needed. If the water content is more than 2 to 3% a significant throughput advantage can be gained by pre-drying samples in a laboratory oven at a temperature of 100° to 110° C.

Finding the Optimum Outgassing Time

Experimental Determination

If the outgassing time is not known, outgassing the sample for different and increasing amounts of time will indicate the correct time to use. Presence of water vapor on the sample surface decreases the available surface for

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adsorption of gas molecules. A sample which has not been outgassed will give a lower surface area than one which has. As the outgassing time is increased, monitoring the surface area results will indicate a continued increase until no further water is removed. Plot the measured surface area vs. The outgassing time. The optimum outgassing time is then indicated where the surface area vs. outgassing time plot reaches a plateau.

Rough Estimation

Some indication of how the outgassing procedure is progressing can be observed from the status display of vacuum pressure on the **Outgas** screen. During the pump down procedure, the pressure will be reduced gradually from atmospheric pressure until a pressure of 5 torr is reached, at which time the furnace begins to ramp up to the desired outgas temperature. When the outgas temperature is reached and the pressure is 5 torr or less, the vacuum is applied directly to the sample. At this point the indicated pressure should drop to zero as water is removed from the sample. When a stable value close to zero has been achieved, the assumption that the sample has been outgassed may be made. This is only a guideline and should not be taken as an absolute method for determining how long to outgas samples.

B.4 Vacuum System vs. Flowing Gas System

It has been reported that with some samples, particularly pharmaceutical powders, that different surface area values are obtained when the samples are outgassed using a vacuum system (SA 3100 analyzer), when compared with the flowing gas method (SA-PREP outgasser). These differences can be due to chemical changes which occur to samples when under a vacuum. The operator must give attention to this possibility and should outgas the samples in a consistent manner and should also make reference to the outgassing conditions in all data reports.

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A.1 LIMITED WARRANTY

This instrument, when purchased from Beckman Coulter or from an authorized distributor or subsidiary company, is warranted against defects in materials and workmanship for a period of one (1) year from date of the original invoice to the customer for this instrument or for longer periods if purchased.

This warranty is limited to the repair and replacement of parts which prove to be defective during the warranty period. This warranty is not valid for parts damaged, lost or which fail because of accident, fire, theft, acts of nature (storms, floods, etc.) negligence of the use of chemicals which have a deleterious effect.

This warranty is conditioned upon Beckman Coulter, retaining the unqualified option of replacing parts up to and including an entire instrument.

This warranty will not extend to any repairs or modifications made to the instrument by some party other than Beckman Coulter, or a party authorized to do so by Beckman Coulter. Also, this warranty shall be effective only upon written notice of the defect to Beckman Coulter or its authorized distributor within five (5) days after occurrence of said defect.

This warranty shall apply only to use of the instrument at a location within a state of the United States and in Canada and shall not apply to use of the instrument at a location outside the continental limits of the United States, including any territory, possession, military or government facility therein and in any other Country foreign to the United States. Upon request of the purchaser, Beckman Coulter can undertake to arrange for special warranty service upon agreed written terms only at a location where this warranty does not apply. No other warranty of any kind is made, expressed, or implied.



Beckman Coulter, Inc.
250 S. Kraemer Blvd.
Brea, California 92821

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