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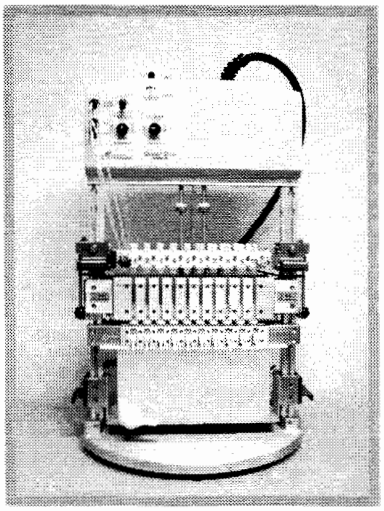
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QUEST 210 MANUAL SYNTHESIZER

Quest 210 - User Manual

ARGONAUT TECHNOLOGIES

Part Number 900114

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Quest 210 Manual Synthesizer — User Manual

Part Number 900114

Revision 1.0, 5 September 2000

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WARRANTY

ARGONAUT WARRANTY AGREEMENT

Limited Warranty

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Exclusions

This limited warranty covers normal use. Exclusions from this limited warranty may include:

- Servicing or modification by anyone other than a qualified Argonaut Technologies Service Representative;
- Non-conformance to recommended instrument operating procedures as noted throughout this manual;
- Failure to perform routine preventative maintenance;
- Any natural disaster, including earthquake, lightning, tornado, flood, or fire; electrical surges or the use of improper power sources.

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1

INTRODUCTION

This manual is intended to assist you with the operation and maintenance of the Quest 210 Organic Synthesizer. It is organized into the following chapters:

Chapter 1: Introduction. Provides general information on the Quest 210 Organic Synthesizer with a brief description of the instrument.

Chapter 2: Safety. Warnings and precautions appropriate to the safe use of the Quest 210 and associated equipment.

Chapter 3: System Overview. Definition of the Quest 210 instrument and an overview of its operation.

Chapter 4: Quest 210 Installation. Step-by-step instructions for installing the Quest 210.

Chapter 5: ASW Option Installation. Step-by-step instructions for installing the Automatic Solvent Wash Option (available separately).

Chapter 6: Preparation For Synthesis. Procedures to be followed prior to each use of the Quest 210.

Chapter 7: Operation. Procedure for carrying out synthesis with the Quest 210.

Chapter 8: Using The ASW Option. Cleaning the Quest 210 with the Automatic Solvent Wash option, for optimum instrument performance.

Chapter 9: Cleaning & Maintenance. Step-by-step instructions for manual cleaning and maintenance of the Quest 210.

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Chapter 10: Tutorial. Step-by-step instructions, using a set of actual examples, for reductive amination to form corresponding secondary amines.

Appendix A: Glossary of Terms.

Appendix B: Controller Screens. A list of the Controller screens and menus.

Appendix C: Specifications.

Appendix D: Parts and Accessories.

Appendix E: Service. Contact information for service and sales.

INTERFERENCE INFORMATION

This equipment generates and uses radio frequency energy and, if not installed and used in strict accordance with the manufacturer's instructions, may cause interference to radio and television reception. It has been type tested and found to comply with the limits for a Class B computing device in accordance with the requirements set forth in the European Standard EN 55011:1997. Class B equipment is suitable for use in domestic establishments and in establishments directly connected to a low voltage power supply network supplying power to buildings used for domestic purposes. However, there is no guarantee that interference will not occur in a particular installation. If this equipment does cause interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try to correct the interference by one or more of the following measures:

- Reorient the receiving antenna
- Relocate the equipment with respect to the receiver
- Move the equipment away from the receiver
- Plug the equipment into a different outlet so that the controller and receiver are on different circuits

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IMMUNITY INFORMATION

This equipment has been type tested and found to comply with the Requirements of the Generic Immunity Standard: EN50082-1/1997.

If the equipment is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired.

QUEST 210 OVERVIEW

WARNING

The Quest 210 Organic Synthesizer is designed for use in chemical synthesis including potentially hazardous chemical reactions. The potential hazards of chemical synthesis include but are not limited to burns, explosions, and exposure to toxic chemicals and carcinogens. Always wear eye protection, suitable gloves, and appropriate clothing. Operate the instrument in a fume hood.

The Quest 210 Organic Synthesizer allows you to perform up to 20 solution or solid phase organic chemistry reactions in parallel. The unique design of the Quest 210 incorporates heating and cooling, agitation, and the inert environment needed for organic synthesis. The system's ease of use and intuitive operation allow new users to quickly integrate the Quest 210 into their syntheses.

The Quest 210 consists of four main parts:

- Reactor
- Interface Harness
- Controller
- Solvent Bottle Assembly

Reactor Compounds are synthesized on the Reactor. With two banks of 10 reaction vessels, the Reactor can synthesize up to 20 compounds in parallel. Each bank of reaction vessels has an integrated heating/cooling block for reaction heating and cooling.

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The Reactor also has rotary controls for solvent and gas delivery. Using these controls, solvents or reagents can be added to the reaction vessels in parallel. Luer ports located on top of each reaction vessel allow for the individual addition of reagent or solvents. Agitation is accomplished by moving a magnetic bar located between the two banks of reaction vessels vertically up and down. Magnets placed in the vessels are attracted to the magnetic agitator bar and follow its movement, effecting mixing of reaction vessel contents.

Interface Harness The Reactor is connected to the Controller by the Interface Harness. Teflon® tubing in the harness delivers gas and solvent to the Reactor. The harness also contains the wiring for the heaters and thermocouple for each bank of reaction vessels. The Interface harness consists of the following lines:

- **Solvent Delivery lines**: Delivers solvent from the solvent bottle(s) to the Reactor. Solvent is delivered in parallel to each bank of reaction vessels.
- **High Pressure Gas line**: Delivers 30 psig (3.1 bar) gas from the Controller to the Reactor. This supplies the pressure to drain liquid from the reaction vessels and to dry solid phase resin.
- **Low Pressure Gas line**: Delivers 10 psig (1.7 bar) gas from the Controller to the Reactor. This line connects to the Low Pressure Gas valve which controls the gas flow for blanketing, draining, and purging the reaction vessels with inert gas.
- **Agitator Gas lines**: Two 30 psig lines (3.1 bar) deliver gas to the pneumatically controlled Agitator Bar.
- **Electrical wiring:** Provides voltage for the heaters, and electrical connections for the temperature sensors for each reaction vessel bank.

Controller The Controller enables fine control of solvent and gas delivery, and the temperature of the RVs in the Reactor. It has a LCD screen and buttons for programming the reaction temperature, time, and agitator conditions using the system's firmware. It also provides connections for system power and 30-40 psig (3.1-3.8 bar) gas.

Solvent, high pressure gas, and low pressure gas deliveries are controlled using the mechanical Control Valves on the Reactor. The User programs the Controller with the agitator and heating values.

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The Controller firmware implements the agitator and temperature program for the synthesis.

Solvent Bottle Assembly The Solvent Bottle Assembly consists of the Solvent Bottle Cap Assembly and a solvent bottle (user-supplied in North America). The Cap has four Teflon lines, for pressure, venting, delivery, and solvent pick up. A two-position valve on top of the Solvent Bottle Cap Assembly allows the user to vent and pressurize the solvent bottle. The Solvent Bottle Assembly connects to the Interface Harness for solvent or reagent delivery to the Reactor.

NOTE

Solvent, high pressure gas, and low pressure gas deliveries are controlled using the mechanical Control Valves on the Reactor. The user programs the Controller with the agitator and heating values.

AUTOMATED SOLVENT WASH OPTION

The optional Automated Solvent Wash (ASW) provides the benefit of programmable wash cycles using up to four solvents. The ASW consists of a Quest Synthesizer Controller with additional software for programming wash cycles, additional gas connections, and a new distribution manifold.

GASEOUS REACTION AND CONCENTRATION MANIFOLD OPTION

The Gaseous Reaction and Concentration Manifold (GRCM) accessory is available separately. It enables the Quest 210 to perform gaseous reactions and concentration by evaporation.

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2

SAFETY

This chapter discusses the safety issues related to the operation and use of the Quest 210 Organic Synthesizer. Please read it carefully before attempting to operate the system.

USER ATTENTION WORDS

"User attention words" appear in the text of this manual. Give careful attention to these entries while consulting the user manual to help ensure your safety and that of the instrument. Each word implies a particular level of observation or action as follows:

➤ NOTE

This word is used to call attention to information.

⚠ CAUTION

This word informs you that damage to the instrument could occur if you do not comply with this information.

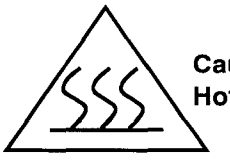
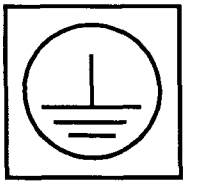
⚠ WARNING

This word informs you that physical injury or death could occur to you or others if these required precautions are not taken.

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WARNING LABELS

Warning labels are applied to the equipment in specific areas where the user must observe safety precautions. Following is a list of these labels along with their locations and meanings:

Label	Picture
When heating, the Reactor Assembly will become hot. The symbol at the right is to warn of a hot surface. Use caution when working near or on the Reactor Assembly. Allow it to cool before attempting to access RVs.	 **Caution! Hot Surface**
Earth ground.	
This symbol indicates a shock hazard. Use care when working around an area with this symbol.	

SAFETY CLASS

This equipment has been tested and found to comply with the requirements set forth in the European Immunity Standard: EN50082-1/1997.

Contact your local Argonaut representative for further information on safety certifications.

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SYSTEM SAFETY

During operation of the Quest 210, hazardous chemicals are stored and manipulated, and high voltage electricity is used. The Quest 210 has been designed to deal with these inherent safety concerns.

Stop Switch A stop switch is located on the front panel of the Controller. Switching this to off will terminate power to the instrument and stop all operations.

⚠ *WARNING*

Failure to provide the Quest 210 with adequate ventilation may result in instrument damage and personal injury.

⚠ *WARNING*

Failure to empty a filled waste reservoir will result in chemical spillage and a possible vapor buildup inside the instrument.

Maximum Temperature of Reaction Vessels The Reaction Vessels will maintain an inert seal at or below the boiling point of their contents. Raising the temperature above the boiling point will jeopardize seal integrity and may cause venting of the reaction and loss of reagent or solvent.

Minimum Temperature of Reaction Vessels Do not reduce the temperature of a Reaction Vessel low enough to freeze its contents. Instrument operation will be adversely affected.

⚠ **CAUTION**

Do not heat Reaction Vessels above the boiling point of their contents, and do not cool the Reaction Vessels to the freezing point of their contents. Instrument operation will be adversely affected. Quest 210 software and hardware does not limit temperature control based on the boiling point and freezing

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point of Reaction Vessel contents, and allows unlimited thermal programming between –40°C and 130°C.

SAFETY EQUIPMENT

The Quest 210 has been designed to handle hazardous chemicals, but should only be operated by personnel trained to safely handle such chemicals. Protective hand and eye equipment should be worn during instrument operation.

We recommend that the following safety equipment be readily available during instrument operation: fire extinguisher, eye wash station, safety shower, eye and hand protection, adequate ventilation, and protection from sources of radiation (i.e. lasers, radioisotopes, contaminated equipment, radioactive waste).

WARNING

If the equipment is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired. Wear adequate eye and hand protection equipment during instrument operation.

OPERATING ENVIRONMENT

The Quest 210 system has been tested and validated in the following operating environments. Argonaut cannot guarantee operation and safety of the Quest 210 in environmental conditions outside these ranges.

Operating Environment

Operational Requirement	Specification
Laboratory Temperature	18° C to 30° C
Laboratory Humidity	≤ 70% relative humidity

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MATERIAL SAFETY DATA SHEETS (MSDS)

The MSDS associated with each chemical used in conjunction with this equipment must be kept at your facility for easy reference by employees. Adherence to the safety precautions listed in the MSDS for each of these chemicals is mandatory for the safe operation of this equipment.

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3

SYSTEM OVERVIEW

The Quest 210 Organic Synthesizer is designed to accelerate traditional organic synthesis. The synthesizer supports both solid and solution phase chemistries, giving scientists flexibility in selecting the appropriate reactions for their syntheses.

The clear Teflon Reaction Vessels are similar to round bottom flasks. This allows continuous monitoring of reactions, so you are always in complete control.

All reagents, solvent washes and reagent additions are performed in a completely inert environment to facilitate the use of air- and moisture-sensitive reagents.

The system's capability to perform on-line TFA cleavages and liquid-liquid extractions streamlines post synthesis work-up and collection of products.

The Gaseous Reactions and Concentration Manifold accessory (GRCM) is available to allow both gaseous reactions and concentration of product by evaporation.

The functional components of the Quest 210 are described below.

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REACTOR

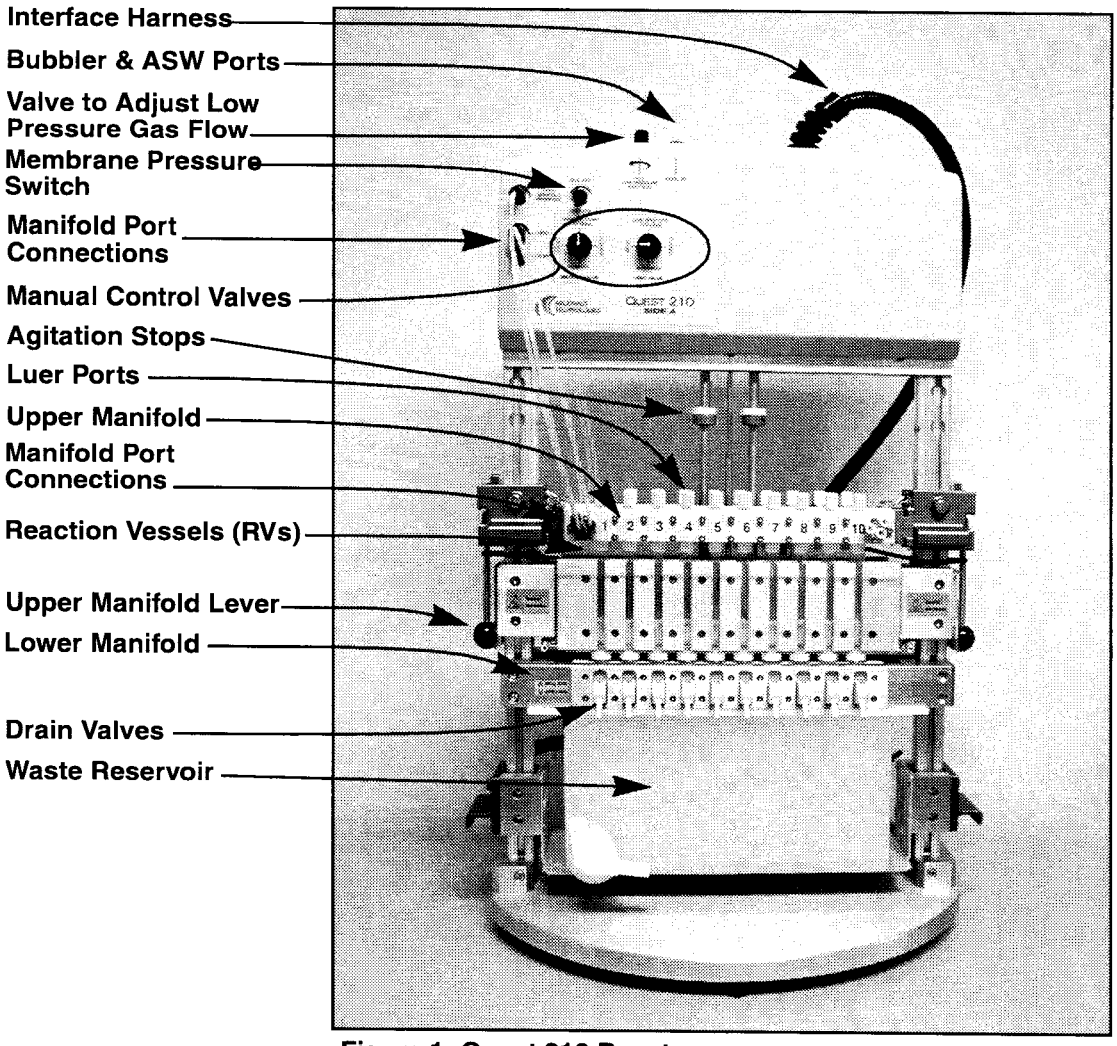


Figure 1: Quest 210 Reactor

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Up to 20 reaction vessels can be mounted onto the Reactor, in two banks of 10, for parallel organic synthesis using solution or solid phase chemistry. Either 5 mL or 10 mL reaction vessels can be used. Reaction vessels are mounted in a spring loaded heating/cooling block for temperature control. The Reactor rotates for easy access to both banks.

A proprietary method of agitation provides efficient mixing during reactions.

The Upper Manifolds enable parallel delivery of solvent and gas to the reaction vessels. Solid or liquid reagent may be added to each reaction vessel through the openings in the Upper Manifolds.

The system uses two gas pressures. The higher pressure (30 psig or 3.1 bar) is used for reaction vessel draining and purging solvent from the Upper Manifold. The lower pressure (10 psig or 1.7 bar) is used for blanketing the reaction vessels with inert gas and for metered draining.

Each reaction vessel has a corresponding drain valve on the Lower Manifold, and can drain into either the Waste Reservoir or a collection rack with vials or tubes for product collection.

REACTION VESSELS

The Reaction Vessels (RVs) are available in 5 mL and 10 mL sizes, made of clear Teflon, and intended for one-time use. A Teflon frit, with 7 µm porosity, is used in the bottom of each RV to allow solutions to drain. These frits are particularly useful for solution-phase chemistries and are shipped standard with each Quest.

The Lower Manifold below each bank of RVs has valves to control RV draining. Reaction Vessels are held in place between the Upper and Lower Manifold by male fittings which press into the ends to create leak-tight seals.

Reaction Vessels pre-installed with Teflon frits of 30 µm porosity are available separately. They are optimal for solid-phase chemistries.

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LUER PORTS

Solid or liquid diversity reagents can be added to each reaction vessel through the openings, or Luer Ports, of the Upper Manifold. The ports are large enough to accommodate liquid transferring devices (e.g., pipets, syringe and needle, etc.) and the addition of solids. Each is fitted with a Luer Port Adaptor and Luer Plug to allow access and to provide a connection point for accessories.

To add larger quantities of reagents or solids, the Luer Port Adaptors may be removed for wider access to the RVs.

An inert atmosphere can be maintained during reagent additions by a low flow of Low Pressure Gas to the reaction vessel. Each Luer Port may be sealed by a Luer Plug.

➤NOTE

A low flow of low pressure gas can be applied to the Reaction Vessels during manual addition of chemicals through the luer ports or luer port adapter.

⚠ *WARNING*

Always vent the reaction vessels before removing a luer plug or a luer port adapter as contents may be under pressure.

To remove a Luer Plug, twist and pull it out. To re-seal the opening, twist the plug while pressing it into the Luer Adaptor.

To remove a Luer Adaptor, rotate the adaptor in a counter-clockwise direction. To install the Luer Adaptor, rotate the adaptor in a clockwise direction until tight.

HEATING AND COOLING

Each bank of reaction vessels is in direct contact with an aluminum heating/cooling block. Heat is provided by a resistive heater mounted inside each block. Cooling below ambient temperature requires an optional refrigerated recirculating chiller which delivers cold liquid through a channel in the heating/cooling block.

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REACTION MIXING

The mixing system consists of a magnet bar that oscillates vertically between the two banks of reaction vessels. It is driven by a pneumatic cylinder powered by 30 psig (3.1 bar) gas controlled by valves in the Controller. Teflon-encapsulated magnets placed in the reaction vessels are attracted to the magnet bar and follow its up-and-down movements, thereby mixing the contents.

AGITATOR MAGNETS

Argonaut offers two different styles of agitator magnets, for solution-phase and solid-phase applications. Use the correct agitator magnet and Reaction Vessel (RV) for optimum performance.

For solution-phase applications, use the solution phase agitator magnet and the RVs. Insert the magnet so that its hollowed end faces the bottom of the RV. This magnet is approximately 2.2 cm long and ensures homogeneous mixing. The RVs have a nominal frit porosity of 7 µm and are designed for product isolation by precipitation in solution-phase applications.

NOTE

This magnet is the standard agitator magnet included with every Quest 210 shipped after April 3, 2000.

For solid-phase applications, use agitator magnet (Part Number 900085) and the RVs fitted with 30 µm frits. Insert the magnet so that its hollowed end faces the bottom of the RV. This magnet is approximately 1.9 cm long and ensures homogeneous mixing without crushing fragile resins or solid supports. These RVs have a nominal frit porosity of 30 µm and are designed for easy product isolation by filtration without letting solid material pass.

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SOLVENT/GAS DELIVERY SYSTEM

The parallel delivery of solvent and gas to the reaction vessels is controlled by valves located on the top panels on both sides of the Reactor. All wetted parts are made from chemically inert Teflon to withstand the harshest reagents and solvents.

UPPER AND LOWER MANIFOLDS

The aluminum and Teflon manifolds are key components of the solvent and gas delivery system.

The Upper Manifold has two delivery pathways. One is for parallel delivery of solvent and high pressure gas, and is connected to the BUBBLER port on top of the Reactor. The other is for venting and low pressure gas delivery and connected to the AUTOWASH port. Delivery Tubes protrude from the male fittings to allow delivery to each reaction vessel.

The Upper Manifold Membrane switch at the top of the Reactor controls the delivery of 30 psig (3.1 bar) gas to a Teflon membrane behind the Upper Manifold. When this switch is open, solvent and gas can be delivered to the reaction vessels. When it is closed, gas pressure seals the membrane against the manifold to prevent any cross-contamination between vessels or delivery of solvent or gas.

The Lower Manifold allows the reaction vessels to be sealed or drained, individually or simultaneously.

Each side of the Reactor has ten drain valves, one for each reaction vessel in the bank. Levers allow each individual valve to be easily opened and sealed. The Parallel Empty Drain Lever enables all valves on each side of the Reactor to be simultaneously operated. The dead volume of each drain pathway is approximately 100 µl.

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MANIFOLD CONTROL VALVES

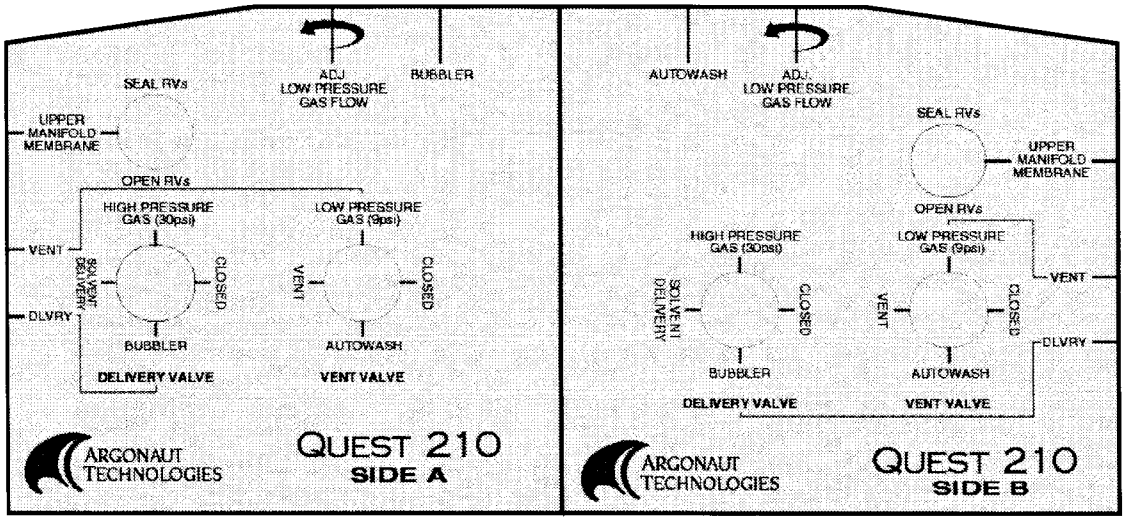


Figure 2: Quest 210 Reactor Labels

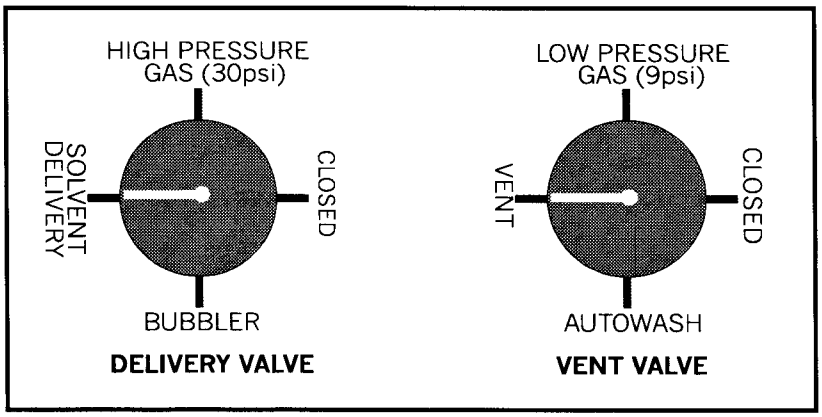


Figure 3: Manifold Control Valves (Solvent Delivery Configuration)

Each side of the Reactor has two control valves as shown in Figure 3. The functions of the Control Valves are as follows:

Delivery Valve (on the left)

- SOLVENT DELIVERY: Parallel delivery of solvent from a solvent bottle to that bank of reaction vessels.
- HIGH PRESSURE GAS: Parallel delivery of 30 psig (3.1 bar) gas to that bank of reaction vessels for reaction vessel draining.

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- BUBBLER: Access to the Bubbler Port on top of the Reactor, for monitoring gas flow.
- CLOSED: Seals the corresponding delivery pathway to the reaction vessels.

Vent Valve (on the right)

- VENT: Vents the reaction vessels
- LOW PRESSURE GAS: Parallel delivery of low pressure gas to that bank of reaction vessels
- AUTOWASH: Connection to the Autowash port and Automated Solvent Wash module
- CLOSED: Seals the corresponding delivery pathway to the reaction vessels

Some common examples of Control Valve settings:

Delivery Valve	Vent Valve	Description
SOLVENT DELIVERY	VENT	Delivers solvent from the solvent bottle to a bank of reaction vessels.
HIGH PRESSURE GAS	VENT	Purges residual solvent in the delivery line into reaction vessels using High Pressure Gas (30 psig or 3.1 bar).
HIGH PRESSURE GAS	CLOSED	Pressurizes the headspace of the reaction vessel with 30 psig (3.1 bar) for draining or product collection
CLOSED	LOW PRESSURE GAS	Delivers low pressure gas to the reaction vessels for blanketing or purging with inert gas, or for solvent draining from reaction vessels.
BUBBLER	LOW PRESSURE GAS	Delivers low pressure gas to the BUBBLER port on the top of the Quest
CLOSED	VENT	Vents a bank of reaction vessels to the waste reservoir
CLOSED	CLOSED	Seals a bank of reaction vessels, preventing solvent and gas delivery.

Low Pressure Gas Valve

This is a valve located on top of the Reactor which controls the delivery of 10 psig (1.7 bar) low pressure gas to both banks of reaction vessels. It allows for precise control of gas delivery for product collection and purging, and blanketing of the reaction vessels with inert gas. The flow rate can be monitored by connecting a bubbler to the Bubbler port, and turning the Delivery valve to Bubbler and the Vent valve to Low Pressure Gas.

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BUBBLER PORT

The top of the Reactor has a port on side A for connecting external accessories such as an oil bubbler, a reagent gas balloon, or other device. The Bubbler is assembled and easily connects to the Bubbler Port via the Teflon tube attached to the Bubbler.

SOLVENT BOTTLE ASSEMBLY

The Solvent Bottle Assembly consists of a Bottle Safety Carrier, and Solvent Bottle Cap. Outside of North America, a 2.5L Solvent Bottle is also supplied. The Solvent Bottle Cap screws onto the solvent bottle and connects to lines in the Interface Harness. One line supplies pressure to the bottle and the other delivers solvent to the Reactor. A third connector is for the vent line, which should be pointed away from the user and anchored within the fume hood.

A two-position valve on the top of the Solvent Bottle Cap Assembly allows venting and pressurizing the solvent bottle.

WARNING

Always vent the bottle prior to unscrewing the cap.

WARNING

Always wear eye protection when operating the Quest.

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PRODUCT COLLECTION

To collect synthesis products, the Waste Reservoir is removed and replaced with a collection rack containing vials or test tubes. The following collection racks are available:

- Collection rack for 20 mL and 40 mL scintillation vials (Part Number 900098)
- Collection rack for 13mm x 100mm screw cap tubes (Part Number 900111)
- Collection rack for 20mm x 125mm screw cap tubes (Part Number 900110)

Each rack can hold up to 20 vials or test tubes. Other collection formats will be available in the future.

AUTOWASH PORT

The top of the Reactor has a port on side B for connecting the Automated Solvent Wash (ASW) module which is available separately.

WASTE TRAY AND WASTE RESERVOIR

The Waste Reservoir is underneath the Lower Manifold on the adjustable Waste Tray and has a drain valve which can be connected by tubing to a larger waste container. This arrangement is required when the Automated Solvent Wash Module is in use, and is always advisable.

The buttons on the Waste Tray handles allow the tray to be raised or lowered for removal or installation of the Waste Reservoir.

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CONTROLLER

The Controller is used to set the reaction temperature, heating duration, and agitator conditions via the keypad and LCD screens. Parameters set on the display screens are executed by the firmware, which is resident on PC boards in the Controller. See Figure 4 on the next page.

The Controller has ports to connect lines from the Reactor. These lines (from the Reactor) make up the Interface Harness and include the electrical, low pressure gas (9 psi), high pressure gas (30 psi), solvent, and two agitator lines.

Other components of the Controller include agitator control valves, Mix Speed valve, gas pressure regulators, and gas distribution manifolds.



Figure 4: Quest Controller

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The Controller controls the following functions:

- Reaction temperature.
- Reaction temperature duration.
- agitator parameters including agitator frequency and stroke duration.
- Connections for the system gas pressure and electrical power.

The Controller LCD screen and keypad are used to set the reaction temperature, temperature duration, and agitator parameters. See Figure 5.

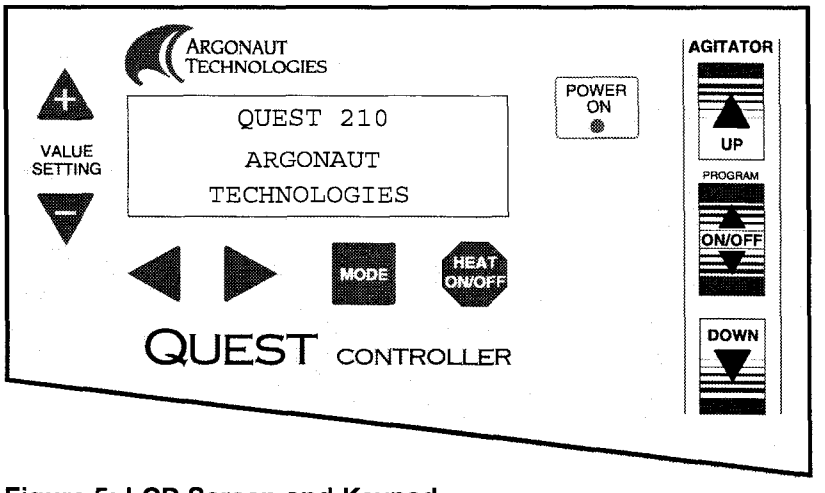


Figure 5: LCD Screen and Keypad

LCD Screen

The LCD screen presents the various Quest 210 programming screens. The screens display the status of the agitator and temperature settings. Reaction temperature, temperature duration, and agitator parameters may be changed using the keypad in association with the appropriate screen. Details of the screens described below are given in Appendix B, "Controller Screens".

Display Screens

- Sign-on Screen: Identifies this instrument as the Argonaut Technologies Quest 210.
- Firmware Version Screen: Shows the Quest 210 firmware version number.

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- Agitator Control Screen: Allows user to set the agitator parameters: MixEvery, UpStroke, and % Upward.

The MixEvery parameter sets the agitator frequency expressed in seconds. The agitator frequency is defined as the time between consecutive up-strokes.

The UpStroke parameter is the amount of time the agitator bar will be in the upper position. This number is necessarily less than the agitator frequency value. The time begins with the UpStroke initiation and ends with the downstroke initiation.

The "% Upward" parameter controls the upstroke in the same manner as the "Upstroke" parameter, but is expressed in terms of a percentage of the agitator frequency rather than a time.

- Set Temperature Screen: Allows user to set the temperature for each of the two Reaction Vessel banks, turn on the heaters, designate how long each Reaction Vessel bank should remain at this temperature, and indicate the volume in the 10 mL Reaction Vessels.
- Monitor Temperature Screen: Displays the set-point temperature of the Reaction Vessel banks, the actual temperature, remaining heating time and heater status (on or off).
- Other Functions Screen: Allows access to functions not available on the main screens. The "other functions" are Proceed to Main, Show Firmware Version, Adjust LCD, Self-Test, Show Agitator History, Show Heater History and Restore Defaults.

KEYPAD

The keypad buttons are used to move between screens, select a parameter on an active screen, and increase or decrease the chosen parameter value. The MODE, FORWARD, BACK, VALUE SETTING, HEAT ON/OFF, and the AGITATOR Control Buttons are described in the next pages.

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MODE Button

Pressing the MODE button allows the user to move from the current display screen to the subsequent screen. See Figure 6.

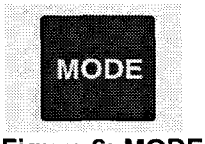


Figure 6: MODE Button

For information on the function of the MODE button in a particular screen, see Appendix B, "Controller Screens".

FORWARD and BACK Buttons

The FORWARD and BACK buttons (right and left arrow buttons) allows the user to select a parameter on the display screen. See Figure 7.

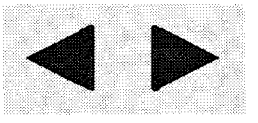


Figure 7: FORWARD and BACK Buttons

Press the FORWARD button (right arrow) to move to the next parameter or digit on the display screen.

Press the BACK button (left arrow) to move to the previous parameter on the display screen.

For information on the function of the FORWARD and BACK buttons in a particular display screen, see Appendix B, "Controller Screens".

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VALUE SETTING Buttons

The VALUE SETTING buttons (up and down arrow buttons) allows the user to increase or decrease the display parameter selected with the FORWARD and BACK buttons. See Figure 8.

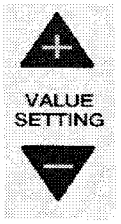


Figure 8: VALUE SETTING Buttons

Press the + VALUE SETTING button (↑) to increase the value of the selected parameter.

Press the – VALUE SETTING button (↓) to decrease the value of the selected parameter.

For information on the function of the VALUE SETTING buttons in a particular display screen, see Appendix B, "Controller Screens".

HEAT ON/OFF Button

The HEAT ON/OFF buttons start and stop various functions depending on the screen that is displayed at the time the button is pressed. See Figure 9.

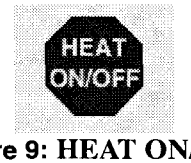


Figure 9: HEAT ON/OFF Button

Pressing the HEAT ON/OFF button when the Set Temperature screen is displayed starts or stops the heaters, as specified.

Press the HEAT ON/OFF button to turn on the heater for the first bank of Reaction Vessels after the reaction bank temperature and temperature duration on the Set Temperature screen have been set.

Set the reaction bank temperature and temperature duration for the second bank of Reaction Vessels and press HEAT ON/OFF to turn on that heater.

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The heaters remain at the specified temperature for the specified time, at which point the heaters turn off.

The HEAT ON/OFF Heaters display screen appears when the HEAT ON/OFF button is pressed while in any other display screen. For more information, see Chapter 7, "Operation".

AGITATOR Control Buttons

The three AGITATOR Control buttons are located on the keypad to the right of the LCD screen. See Figure 10.



Figure 10: AGITATOR Control Buttons

The uppermost button causes the agitator bar to move to, and stay in, the up position.

The lower button causes the agitator bar to move to, and stay in, the down position.

The middle button causes the agitator bar to oscillate continually. Press the middle agitator button to start or stop mixing the reaction solution once the agitator parameters have been set on the Agitator Control screen.

For detailed information on setting the agitator, see Chapter 7, "Operation", page 7-79.

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MIX SPEED VALVE

The Mix Speed valve, located in the right side of the Controller controls the speed at which the agitator bar moves.

NOTE

The valve is set to closed during shipment. Turn the valve counter clockwise to start agitator.

Turn the valve counter clockwise to increase the speed.

Turn the valve clockwise to decrease the speed.

For detailed information on setting the agitator frequency, see Chapter 7, "Operation" on page 7-82.

CONTROLLER GAS AND POWER CONNECTIONS

Gas

The Quest 210 can use either one or two sources of gas. With two gas sources, the inert gas is used for delivering solvents, draining Reaction Vessels, and creating an inert reaction environment. The compressed air drives the agitator system. If a single source of gas is used, the agitator system is driven by the inert gas. Using compressed air for agitator significantly reduces the quantity of inert gas consumed by the Quest 210.

If a single source of gas is connected to the Quest 210, the gas should be a regulated 55-60 psig high-purity (>99%) inert gas. If two gas sources are connected to the Quest 210, one should be a regulated 40- 60 psig high-purity (>99%) inert gas and the second should be a regulated 55-60 psig source of clean, dry compressed air.

The gas ports are located on the rear of the Controller adjacent to the power switch and the electrical socket. Quick-connect fittings are used to interface the Controller with the gas sources.

Electrical Outlet

The power cord connects to the rear of the Controller. The instrument can operate on 110-120 or 200-240 VAC, 50-60 Hz.

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Power Switch

The power switch on the back of the Controller turns the power on and off after the power cord is plugged into the power outlet.

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QUEST 210 INSTALLATION

Installation services are not included in the purchase price of the Quest 210. Unless other contractual agreements have been arranged, it is the responsibility of the customer to unpack, move, and install the Quest 210 Organic Synthesizer. If required, contact your local Argonaut representative for pricing of installation services by a qualified Argonaut Service Engineer.

We recommend that you retain the packing material used to ship the Quest to the installation site in case it is necessary to return the Reactor or Controller for repair. If the packaging has been discarded, new shipping material can be purchased from Argonaut Technologies.

If you intend to install the Automated Solvent Wash Option, you must first complete the installation of the Quest 210.

SITE PREPARATION

It is the responsibility of the customer to prepare the site environmentally and provide for the required space and services such as power, venting, bottled and compressed gases, permits, licenses, approvals, etc. Argonaut will supply the customer with pre-installation information detailing important site requirements prior to shipment.

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INSTALLATION REQUIREMENTS

Before you begin the installation, check carefully that the place you have chosen for the Quest 210 meets the requirements below:

SPACE

Reactor The Reactor requires space 3 ft. x 2 ft. x 3.5 ft. (W x D x H) within and on the left side of a fume hood.

Controller The Controller requires space 1 ft. x 1.75 ft. x 1.5 ft. (W x D x H) on a laboratory bench or cart adjacent to the fume hood.

GAS

Single Source

40-60 psig (3.8-5.2 bar) nitrogen or other high-purity (>99%) inert gas. This gas is used for delivering solvents, reaction vessel draining, creating an inert reaction environment, and to power the agitator system.

Dual Source

40-60 psig (3.8-5.2 bar) nitrogen or other high-purity (>99%) inert gas. This gas is used for delivering solvents, reaction vessel draining, and creating an inert reaction environment.

55-60 psig (4.8-5.2 bar) clean, dry compressed air to power the agitator system. While the inert gas can also be used to power the agitator system, the use of compressed air significantly reduces the amount of inert gas consumed by the system.

ELECTRICAL

90-240 VAC for the Quest Controller.

REFRIGERATION

Optional recirculating chiller for operation below ambient temperature.

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MATERIALS

Solvents and reagents for syntheses.

COMPONENTS

Ensure that you have the following components of the Quest 210:

- Reactor
- Controller
- Interface Harness (Shipped with one end connected to the Reactor)
- Starter Kit: Waste Reservoir, Solvent Bottle Cap Assembly, Solvent Bottle Safety Carrier, and 5 mL and 10 mL reaction vessels.

Installation Glossary

Teflon Tubing Semi-transparent, semi-rigid tubing (1/8 in. OD) used for gas and liquid connections.

Tygon Tubing Flexible tubing with a slight yellow color used to interface the Controller to the gas sources.

Nuts Tan colored threaded fittings (PEEK) used for Teflon tubing connections.

Ferrule Yellow conical-shaped Tefzel sleeve used with PEEK nuts for Teflon tubing connections.

Bulkhead Fitting Female threaded ports for the connection of tubing found on the front of the Controller and on various places on the Reactor.

Interface Harness Tubing and electrical harness which connects the Controller to the Reactor.

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INSTALLATION PROCEDURE

Installation of the Quest is simple and involves connecting the Interface Harness to the Controller, connecting the Controller to the house power and gas sources, and connecting the Solvent Bottle Cap Assembly. It must be done with care to ensure that all connections are correct, but total installation time should take less than one hour.

1. **Clear a space 3 ft. x 2 ft. x 3.5 ft. (W x D x H) on the left side of the fume hood for the Reactor. A 1 ft. x 1.75 ft. x 1.5 ft. (W x D x H) space outside the fume hood and within 6-7 ft. of the Reactor must be provided for the Controller.**
2. **Carefully unpack the instrument and place the Reactor (Figure 11) in the cleared space in the fume hood. Place the Controller (Figure 12) on the lab bench or on a cart within 6-7 ft. of the Reactor. Remove any shipping material.**

⚠ *WARNING*

The Controller should not be used inside the fume hood. The Controller should not be operated in the presence of flammable gases or fumes. Operation of any electrical instrument in such an environment constitutes a safety hazard.

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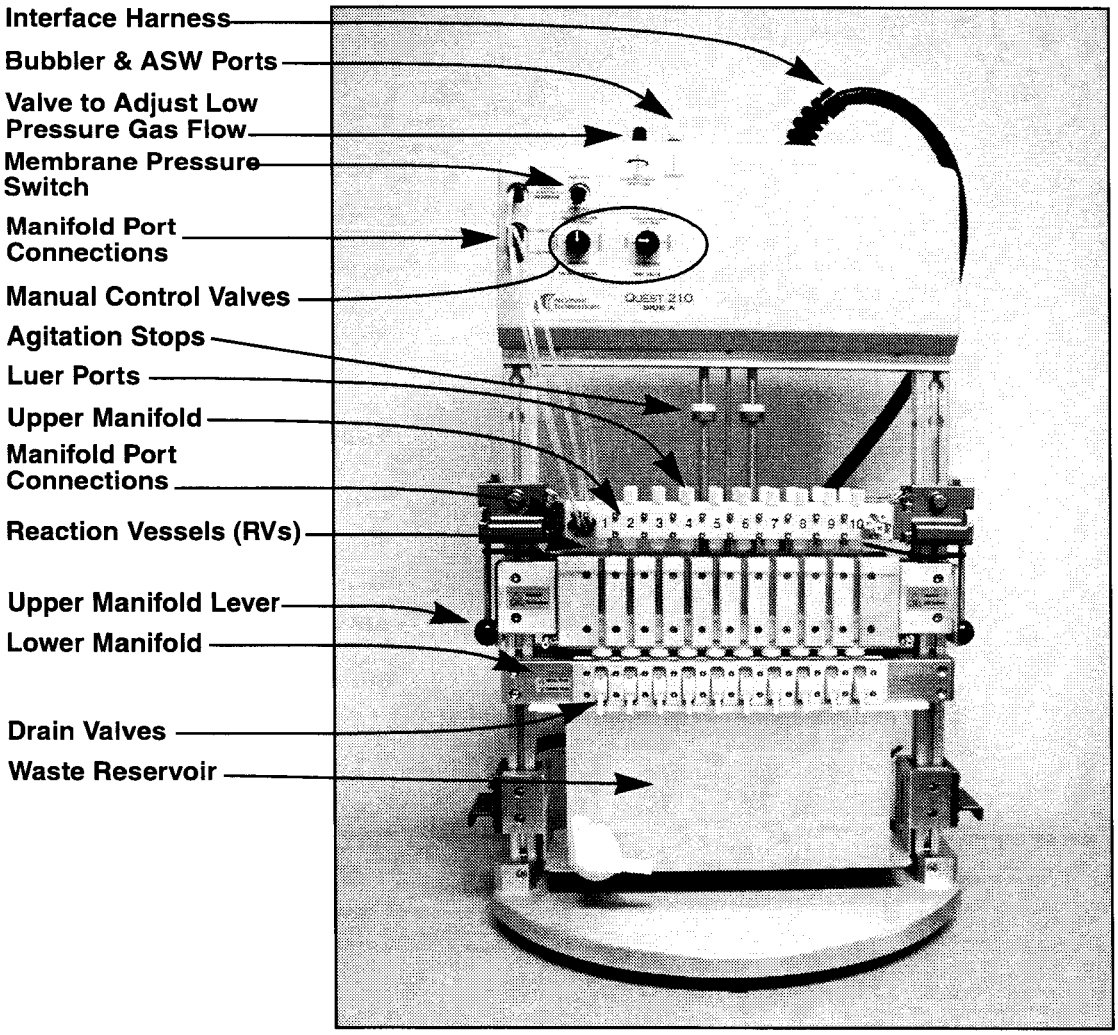


Figure 11: Quest 210 Reactor

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Figure 12: Controller

INTERFACE HARNESS CONNECTIONS

1. **The Interface Harness is the thick cable assembly coming out of the top of the Reactor. Free the Interface Harness from any packing material and position it so the free end is in front of the Controller (Figure 12).**
2. **Insert the round male electrical connector labeled P2 (connection with large pins) on the Interface Harness into the round female connector labeled HEATERS on the Controller. Twist the knurled locking ring of the connector housing and lock the cable into place.**
3. **Connect the male electrical connector labeled P3 (connector with smaller pins) into the round female connector labeled SIGNAL on the Controller. Twist the knurled locking ring of the connector housing and lock the cable in place.**

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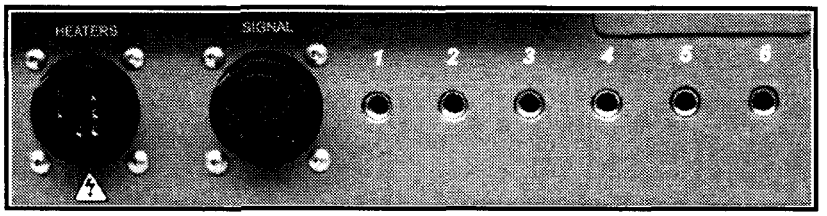


Figure 13: Controller - Interface Harness Connections

4. **Remove the tape from the female fittings labeled 1, 2, 3, 4, and 5 on the front of the Controller.**
5. **There are 6 Teflon lines labeled 1, 2, 3, 4, 5, and MEM on the free end of the Interface Harness. Carefully match the tube label with the label on the front panel of the Controller. Insert the tube with the ferrule and nut into the appropriate female bulkhead fitting and carefully screw in the tan PEEK nut until the fitting is finger-tight. The tube and fitting labeled MEM is not connected. This tube is used in conjunction with the Automated Solvent Wash module which is also available separately. See Chapter 5 for instructions on installing the Automated Solvent Wash module.**

NOTE

Ensure that the nut is loose on the Teflon tube prior to screwing in the fittings. The nut should not be "bound" to the yellow ferrule, as this can cause twisting of the Teflon tube during installation. To release a bound nut from a ferrule, grasp the ferrule between your thumb and index finger and gently rock the nut back to free it. Do not cross-thread the fittings. Ensure that the flat surface of the ferrule (perpendicular to the end of the Teflon tube) is free of dust and fibers. This is the surface that creates the seal for the tubing connection.

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SOLVENT BOTTLE CAP ASSEMBLY

1. **The Solvent Bottle Cap has 3 female ports that are used to vent, pressurize, and deliver solvent from the solvent bottle (Figure 14). A vent tube is included: Insert the ferrule and nut end of this line into the vent port and carefully screw in the tan nut until the fitting is finger-tight.**

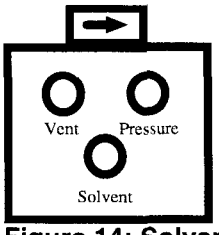


Figure 14: Solvent Bottle Cap - Female Ports

2. **In the middle of the Interface Harness are two free teflon lines labeled PRESSURE and SOLVENT. Insert the line labeled PRESSURE into the Pressure port and finger-tighten.**
3. **Insert the line labeled SOLVENT into the solvent port and finger-tighten.**
4. **The SOLVENT pick-up line extends from the Solvent Bottle Cap to the bottom of the solvent bottle and has a polyethylene Solvent/Reagent Filter on the end to prevent particulates from entering the solvent delivery system. The filter should be changed periodically, depending on usage.**

➤ NOTE

When the dial is turned to Vent, gas and solvent fumes will escape through the Vent port of the Solvent Bottle Cap.

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REACTOR MANIFOLD TUBES

Teflon tubing is used to transport gas and solvents from the control valves to the Upper Manifold. The tubes in each group are labeled as follows:

DELIVERY

VENT

MANIFOLD

1. **Identify the tube labeled MANIFOLD. Insert the ferrule and nut end of the line into the upper female fitting labeled MANIFOLD on the same side of the Reactor. Carefully screw in the tan nut until it is finger-tight.**
2. **Repeat this process for the tubes labelled VENT and DELIVERY. Insert them into the ports labelled VENT and DELIVERY, respectively.**
3. **Repeat the installation of the tubes on the other side of the Reactor.**

WASTE RESERVOIR

1. **Unpack the Waste Reservoir from the box.**
2. **Grasp the handles of the lower Waste Tray of the Reactor. Press the buttons in on the handles and slide the tray to its lowest position.**
3. **Carefully place the Waste Reservoir on the tray and raise the tray back to its upper position. The Waste Reservoir should rest up against the Lower Manifold.**

MANIFOLD CONTROL VALVES

The two control valves on both sides of the Reactor should be set to Closed (Figure 15).

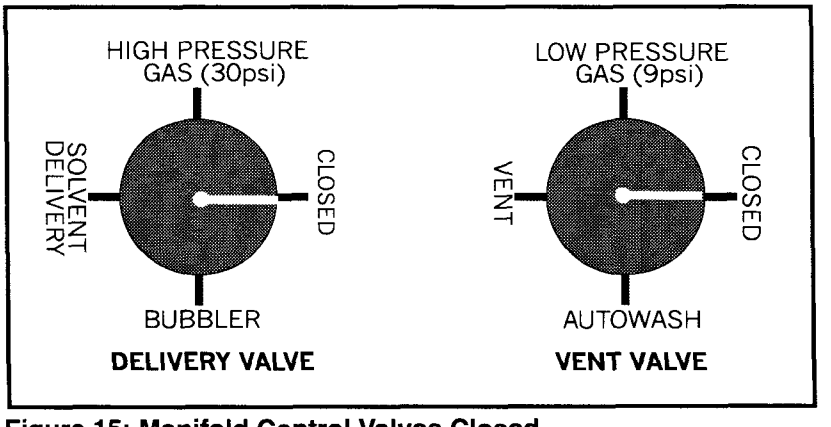


Figure 15: Manifold Control Valves Closed

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GAS CONNECTION

The Controller is equipped with a Dual Gas feature which allows the Reactor to be supplied by either a single inert gas or two sources of gas (for example, an inert gas and another gas such as compressed air).

The inert gas inlet requires a regulated 40-60 psig (3.8-5.2 bar) source of high-purity (>99%) inert gas. This gas is used for delivering solvents, reaction vessel draining and creating an inert reaction environment.

The compressed gas inlet requires a regulated 55-60 psig (4.8-5.2 bar) source of clean, dry gas to power the agitator system. High-purity (>99%) inert gas may be used, but the use of compressed air to operate the agitation system significantly reduces the amount of inert gas consumed by the Quest Synthesizer.

The Gas Supply Assembly connects the Controller to the gas source(s). The gas source(s) must have outlets capable of interfacing with the ¼ inch O.D. polyethylene tubing of the Gas Supply Assembly. Refer to the appropriate procedure below to connect the Controller to either one or two sources of gas.

Figure 16 shows the location of the two gas inlets on the rear panel of the Controller.

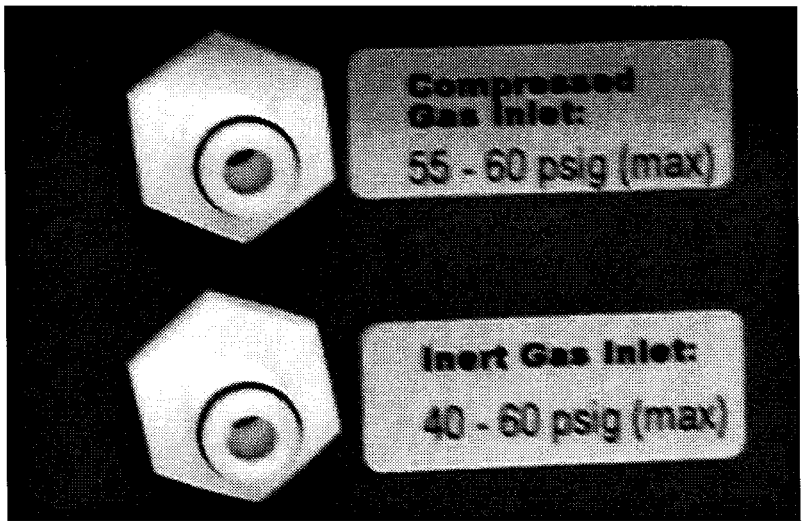


Figure 16: Controller - Gas Inlets

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Single Gas Source

1. **Attach the end of the Gas Supply Assembly *without* the quick connect fittings to an inert gas source capable of providing a regulated output of 55-60 psig (4.8-5.2 bar).**
2. **The other end of the Gas Supply Assembly has a "T" with two quick connect fittings attached. Connect one quick connect fitting to the Inert Gas inlet on the rear panel of the Controller. Push the fitting over the port until a distinct click is heard.**
3. **Connect the other fitting to the Compressed Gas inlet on the rear panel of the Controller. Push the fitting over the port until a distinct click is heard.**
4. **Turn on the gas supply and check for any leaks.**

Dual Gas Source

➤ NOTE

If either gas source is greater than five feet from the Controller additional ¼ inch OD polyethylene tubing will be required for dual gas operation.

1. **Remove the "T" and quick connect fittings from the end of the Gas Supply Assembly.**
2. **Cut the polyethylene line in half and install a quick connect fitting onto one end of each piece of tubing to create two inlet lines.**
3. **Attach the end of the first line to an inert gas source capable of providing a regulated output of 40-60 psig (3.8-5.2 bar). Push the quick connect fitting on the end of this line onto the Inert Gas inlet on the rear panel of the Controller until a distinct click is heard.**
4. **Attach the end of the second line to a compressed air source capable of providing a regulated output of 55-60 psig (4.8-5.2 bar). Push the quick connect fitting on the other end of the line to the compressed gas input on the rear panel of the Controller until a distinct click is heard.**
5. **Turn on the gas supplies and check for any leaks.**

➤ NOTE

A dryer should be installed on the compressed gas line to prevent water from entering the system plumbing.

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Bubbler

The top of the Reactor has two Ports for connecting external accessories such as an oil bubbler, a reagent gas balloon, or other device. Connection is by 1/8 in. Teflon tubing, a 1/8 in. PEEK nut, and a Tefzel ferrule.

Power Connection

1. **Ensure that the ON/OFF switch is set to OFF.**
2. **Plug the power cord into the female receptacle on the rear of the Controller.**
3. **Plug the power cord into the wall outlet. If desired, a UPS or power strip may be used (10A minimum rating).**
4. **Turn on the power to the Reactor using the switch located above the power cord on the rear panel of the Controller.**

Solvent Bottle

This procedure describes the initial installation only. If you are replacing an existing bottle, follow the instructions on page 6-68 "Solvent Bottle".

1. **Place a solvent bottle on the laboratory bench in the safety carrier.**
2. **Install a Polyethylene Solvent/Reagent Filter onto the end of the solvent pick-up line protruding from the bottom of the Solvent Bottle Cap Assembly.**
3. **Place the Teflon bottle seal provided with the Solvent Bottle Cap Assembly into the neck of the solvent bottle. This seal is required to pressurize the Solvent Bottle.**
4. **Insert the pick-up line into the solvent bottle and tighten the Solvent Bottle Cap Assembly onto the solvent bottle.**
5. **Turn the dial on the top of the Solvent Bottle Cap Assembly towards the Teflon tube marked Pressure. Wait a minute or so for the bottle to pressurize.**

➤ NOTE

When pressurizing a Solvent Bottle, ensure that all four Control Valves are set to CLOSED.

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⚠ WARNING

Always vent the bottle prior to unscrewing the cap.

This completes the installation of the Quest 210 Organic Synthesizer. To start your Quest 210 warranty you must fill out and mail the warranty return card provided with the instrument. Retain the packing material used to ship the Quest to the installation site. This packaging can be used in the event the Reactor or Controller needs to be returned to the factory for repair. If the packaging has been discarded, new shipping material can be purchased from Argonaut Technologies. If you experience any problems or difficulties related to the installation of the Quest 210 Organic Synthesizer, contact the Quest 210 Technical Support Hotline. (See Appendix E: "Service" for details.)

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5

ASW OPTION INSTALLATION

⚠ CAUTION

The Automated Solvent Wash (ASW) Option should be installed only after the Quest 210 has been properly installed and proven.

OVERVIEW

The Automated Solvent Wash (ASW) Option provides the benefit of programmable wash cycles using up to four solvents.

- The ASW holds up to four solvent bottles with their associated Bottle Cap Assemblies. These are identical to the one used on the standard Synthesizer.
- The ASW also contains the tubing and valves that control the automated delivery of the four solvents to the Reactor. Four toggle switches on the front panel allow for manual delivery of solvents, and a fifth toggle switch controls purge gas (inert gas used to clear lines after solvent delivery).
- A harness connects the ASW to the Controller and provides the operating gas pressures for the ASW valves.

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INSTALLATION REQUIREMENTS

Space, electrical, gas, waste, and ventilation requirements for the Automated Solvent Wash Option are described below. Adherence to the following will simplify installation.

SPACE

The Controller is 10 inches (25 cm) wide by 16.5 inches (42 cm) deep by 12 inches (30 cm) high and weighs 20 pounds (9 kg). Place the Controller in a cleared space on the laboratory bench adjacent to the fume hood.

The ASW requires bench space within a fume hood 17 inches (43 cm) wide by 22 inches (56 cm) deep by 18 inches (46 cm) high. The bench space must be capable of supporting 58 pounds (26 kg) and be located between the Reactor and Controllers.

Figure 17 shows the recommended setup for the Automated Solvent Wash Option with a Quest 210.

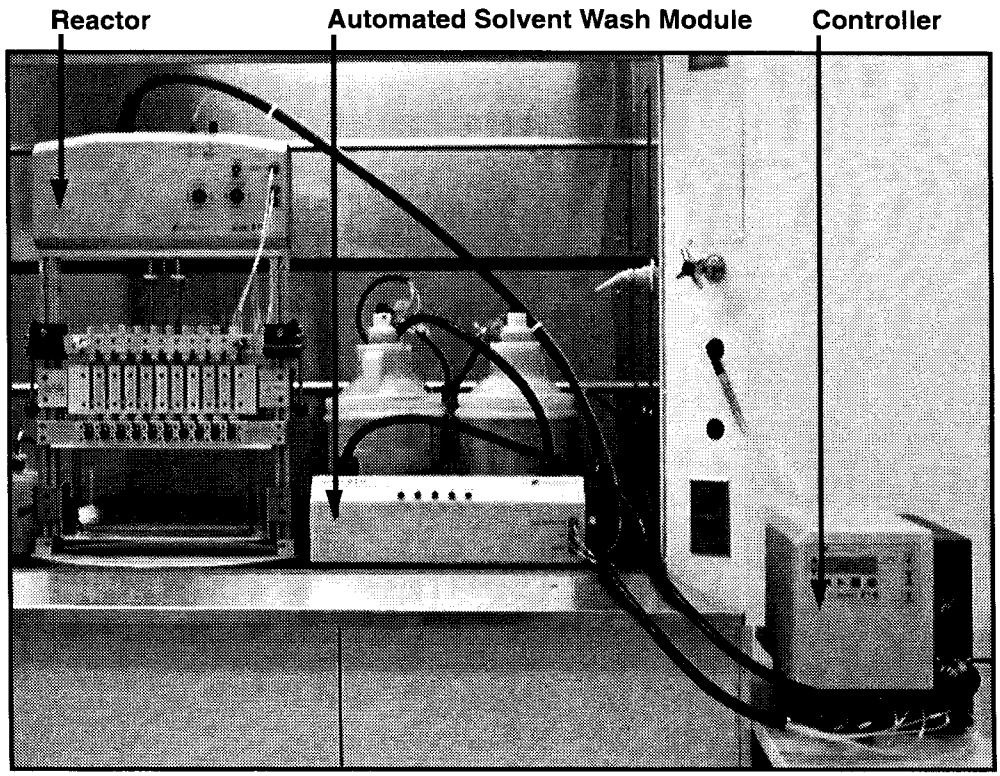


Figure 17: Recommended Set-Up of a Quest with the ASW Option

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ELECTRICAL SUPPLY

The electrical requirements for the ASW Controller are the same as for the standard Quest synthesizer. Because the ASW is controlled pneumatically there are no additional electrical requirements.

The electrical requirements for the ASW Controller are 100-120 or 200-240- VAC, 50/60 Hz, wall outlet (10A minimum rating).

GAS SUPPLY

Single Source

40-60 psig (3.8-5.2 bar) nitrogen or other high-purity (>99%) inert gas. This gas is used for delivering solvents, reaction vessel draining, creating an inert reaction environment, and to power the agitator system.

Dual Source

40-60 psig (3.8-5.2 bar) nitrogen or other high-purity (>99%) inert gas. This gas is used for delivering solvents, reaction vessel draining, and creating an inert reaction environment.

55-60 psig (4.8-5.2 bar) clean, dry compressed air to power the agitator system. While the inert gas can also be used to power the agitator system, the use of compressed air significantly reduces the amount of inert gas consumed by the system.

➤ NOTE

A dryer should be installed on the compressed gas line to prevent water from entering the system plumbing.

WASTE FACILITIES

A Synthesizer with the Automated Solvent Wash Option uses the same Waste Reservoir for waste collection as does the standard Synthesizer. Because of the quantity of solvent used for washing, a line should be attached between the Waste Reservoir valve and a larger waste container to handle the overflow.

Refer to State and Local regulations on the handling and disposal of hazardous waste.

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VENTILATION

The Synthesizer with Automated Solvent Wash Option can generate volatile waste when certain syntheses are performed. The ASW must be placed directly in a fume hood before use. This allows any hazardous vapors to be properly vented and provides optimal laboratory safety.

⚠ WARNING

If a Quest 210 with the Automated Solvent Wash Option is installed outside a fume hood, a method to evacuate any hazardous vapors created during operation is required (e.g., snorkels, adjustable hood).

INSTALLATION PROCEDURE

⚠ CAUTION

The Quest synthesizer should be installed and tested before installing the Automated Solvent Wash Option.

⚠ WARNING

The Quest Synthesizer with Automated Solvent Wash is used for chemical synthesis, including potentially hazardous chemical reactions. The potential hazards of chemical synthesis include but are not limited to burns, explosions, and exposure to toxic chemicals and carcinogens. Always wear eye protection, protective clothing and suitable gloves. Operate the instrument in a fume hood.

LOCATION

Figure 17 shows the recommended arrangement of components within and outside the fume hood.

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GAS CONNECTIONS

The dual gas feature on the Automated Solvent Wash Option allows the use of either one or two gas sources with the instrument (for example, an inert gas and another gas such as compressed air).

The gas connections to the Reactor are on the right rear panel of the Controller. The gas supplies are connected to the Controller using Tygon tubing and Quick Connect fittings.

The procedure for installing gas connections is described fully in Chapter 4, "Quest 210 Installation," starting on page 4-40.

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POWER CONNECTION

1. **Ensure that the ON/OFF switch on the rear of the Controller is set to OFF.**
2. **Plug the power cord into the female receptacle on the rear of the Controller.**
3. **Plug the power cord into the wall outlet. If desired, a UPS or power strip may be used (10A minimum rating).**
4. **Turn on the power to the Reactor using the switch located above the power cord on the rear panel of the Controller.**

CONTROLLER AND REACTOR

Attach the ASW Interface Harness to the front of the ASW Controller (Figure 18) by lining up the surfaces and rotating the locking collar clockwise until tight.

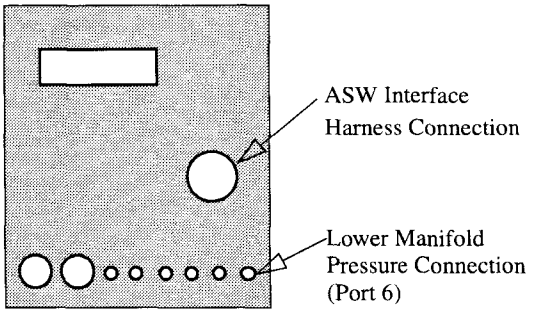


Figure 18: ASW Controller Front View

1. **The remaining connection that differentiates the ASW Controller from the standard Controller is an additional port on the front panel labeled "Lower Manifold Pressure connection" (Port 6) (see Figure 18).**
2. **Locate the tubing labeled MEM in the Interface Harness and connect it into port 6 of the Controller front panel. The connection should be finger-tight.**
3. **Complete the remaining tubing and cable connections from the Reactor Interface Harness to the ASW Controller. These connections are identical to the standard Synthesizer connections and are described in Chapter 4, "Quest 210 Installation.".**

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ASW Module

The locations of the ports on the front of the ASW are shown in Figure 19.

1. **Connect the PRESSURE line from the Interface Harness to the Bottle Pressure port on the front of the ASW.**
2. **Connect the SOLVENT line from the Interface Harness to the SOLVENT port on the front of the ASW.**

Connect one end of the 1/8 inch (0.64 cm) OD Teflon tubing with fittings to the VENT port on the front of the ASW and the other end to the AUTOWASH port on top of the Reactor.

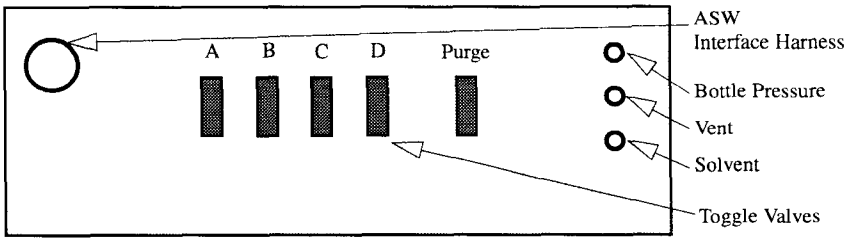


Figure 19: Automated Solvent Wash module (ASW) Front Panel

Solvent Bottles

Three Bottle Cap Assemblies are included with the Automated Solvent Wash Option. The Bottle Cap Assembly originally shipped with the Quest serves as the fourth assembly.

For each Bottle Cap Assembly there are two pieces of tubing identified as either PRESSURE or SOLVENT for each of the four solvents.

Connect each pair of tubes to the corresponding ports on the corresponding Bottle Cap Assembly (see Figure 20).

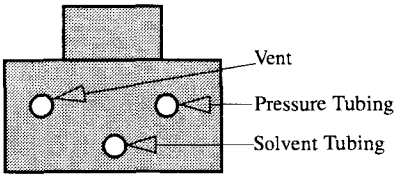


Figure 20: Bottle Cap Assembly

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NOTE

Additional tubing assemblies are provided for connection to the Vent ports on the Bottle Cap Assemblies to allow for flexibility in locating the solvent bottle vents.

1. **Place four solvent bottles in the safety carriers and remove the clear cap from the top of the each carrier.**
2. **Place each solvent bottle with carrier into the ASW.**
3. **Remove the caps from the solvent bottles.**
4. **Insert a Teflon bottle seal into each solvent bottle. This item is required to pressurize each bottle.**
5. **Attach a Bottle Cap Assembly to each solvent bottle.**
6. **Place the four solvent bottles, and their appropriate caps, into the ASW as indicated in Figure 21 below.**

Bottle Cap Assembly	Location in the ASW (as viewed from the front)
A	Left Front
B	Right Front
C	Left Rear
D	Right Rear

Figure 21: ASW Solvent Bottle Locations

NOTE

Solvent bottles are provided for all Buyers outside of North America.

If installing solvents bottles for the first time, refer to "Manual Functions" on page 5-55 for the recommended solvent to use for that procedure.

7. **Using Figure 20 as a guide, turn the valves on the top of the Bottle Cap Assemblies towards the vent ports.**

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WARNING

Always vent the bottle prior to unscrewing the Bottle Cap Assembly.

WARNING

Always place the solvent bottles in the plastic safety carriers when working with the Quest Synthesizer.

This completes the installation of the Automated Solvent Wash Option. Before using it, follow the "Installation Verification" procedure below.

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INSTALLATION VERIFICATION

The installation verification process is designed to ensure that the Automated Solvent Wash Option has been properly installed on the Synthesizer and that it functions correctly.

The first Operational Checkout procedure verifies the configuration of the ASW Controller. The second procedure verifies the manual operation of the Automated Solvent Wash Option by demonstrating that a specific bottle will deliver solvent when selected.

⚠ WARNING

Always wear eye protection and appropriate clothing when operating the Synthesizer.

CONFIGURATION

1. **Turn on the Controller and the gas supply.**
2. **On power-up, the firmware determines if the hardware and software necessary for Auto Solvent Wash are present.**
3. **Press the MODE button on the front of the Controller one time to display the Firmware Version screen. If the Firmware Version screen includes the line "with Auto Solv. Wash" as shown in Figure 22, then the Controller is properly configured.**

```
---- QUEST 210 -----
with Auto Solv. Wash
Rev:   2.0
Date:  Jun 12, 1998
```

Figure 22: Quest Firmware Version Screen

➤ NOTE

The Firmware Version screen can also be accessed from the Other Functions menu.

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MANUAL FUNCTIONS

NOTE

Methanol is the recommended solvent when performing the Manual Operation Verification procedure.

1. **Verify that the five toggle valves on the ASW front panel are all in the OFF (down) position. (See Figure 19 for the location of the toggle valves.)**
2. **Install ten (10) Reaction Vessels on Side A of the Reactor.**
3. **Turn the Control Valves to SOLVENT DELIVERY and VENT.**
4. **Set the Upper Manifold Membrane toggle valve to the OPEN RVs (down) position.**
5. **On Side B of the Reactor, set the two Control Valves and the Upper Manifold Membrane toggle valve to the CLOSED RVs (up) position.**
6. **Ensure all Lower Manifold drain levers on both sides of the Reactor are in the SEAL RVs position.**
7. **Rotate the Reactor to access Side A.**
8. **On the ASW, pressurize bottle A by turning the valve on top of the Bottle Cap Assembly toward the tubing labeled PRESSURE.**
9. **Wait ten minutes for bottle A to pressurize before delivering solvent.**
10. **On the ASW front panel, flip the toggle valve labeled A to the ON (up) position. Within 30 seconds, solvent should be flowing into the RVs on Side A of the Reactor.**
11. **Deliver approximately 4mL of solvent to the RVs and return toggle valve A to the OFF (down) position. Verify that the solvent flow stops.**
12. **On the ASW, flip the purge toggle valve to the ON position. Observe that the remaining solvent in the delivery tubing is forced into the RVs followed by purge gas.**
13. **Return the purge gas toggle valve to OFF.**
14. **Depressurize solvent bottle A by turning the valve on the top of the Bottle Cap Assembly towards the VENT port.**
15. **Ensure the waste reservoir is installed on the Reactor.**
16. **Flip the Lower Manifold drain levers to OPEN RVs.**
17. **Drain the reaction vessels on Side A of the Reactor by turning the Delivery valve to HIGH PRESSURE GAS and the Vent valve to CLOSED.**

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18. Repeat steps 3 through 17 for ASW bottle positions B, C and D. If any of the above steps do not produce the expected results, the installation should be re-examined.

➤NOTE

If, after re-examining the installation process, any of the above steps still does not produce the expected results, contact Argonaut Technologies. To reach an Argonaut Technologies Technical Support Representative, refer to Appendix E.

Because the ASW is designed to run entirely unsupervised, its first use after installation requires that times be recorded for solvents of differing viscosities.

If this is the first time the ASW is being used, follow the procedure below. This will provide the final system verification.

➤NOTE

In the following procedures, it is assumed that the system has been fully tested in manual mode.

DELIVERY TIMES

Automated solvent delivery is measured by time, and delivery times vary for each solvent due to differences in viscosity. Consequently, it is essential to know exactly how much time is required to deliver a specific quantity of each solvent This is done using a stopwatch while manually delivering to the RVs using the toggle valves on the front of the ASW.

⚠WARNING

Establish proper solvent delivery times before operating the Automated Solvent Wash Option. Inappropriate solvent delivery times can cause overfilling of the RVs and create cross contamination between RVs, upper manifold clogs, and solvent overflow into the ASW.

The four solvents chosen for this procedure (listed in Figure 23) are representative of the most commonly used solvents. Any solvents

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may be used, but delivery times may need to be adjusted based on the viscosity of the chosen solvent.

Solvent	Bottle Position	Viscosity (cP)	4 mL Delivery Time	20 mL Delivery Time
DMF	A (Left Front)	0.92	31 sec	65 sec
THF	B (Right Front)	0.55	22 sec	55 sec
MeOH	C (Left Rear)	0.55	23 sec	51 sec
DCM	D (Right Rear)	0.44	27 sec	63 sec

Figure 23: Representative Solvent Delivery Times

1. **Ensure that the ASW, the bottles and their caps have been properly installed as previously described.**
2. **Turn the valves on top of each solvent Bottle Cap Assembly toward the tubing labeled PRESSURE.**
3. **Install ten 5mL Reaction Vessels on side A.**
4. **Turn the Control Valves to SOLVENT DELIVERY and VENT.**
5. **Set the Upper Manifold Membrane valve to the OPEN RVs position.**
6. **Close the Lower Manifold Drain Valves.**
7. **Start the stopwatch as you flip the ASW Solvent A toggle valve to OPEN.**
8. **Stop the stopwatch when 4 mL is delivered to the RVs.**
9. **Return the toggle valve for solvent A to OFF to stop the flow of solvent.**
10. **Record the time on the stopwatch as the delivery time for the specific solvent and volume dispensed from bottle A.**
11. **Flip the Purge toggle valve on the front of the ASW to ON. This purges any remaining solvent in the delivery line. Notice that purging the delivery line adds additional solvent to the RVs.**
12. **Return the Purge toggle valve to OFF.**

➤NOTE

During an Automated Solvent Wash Program, purging is automatically performed after each delivery of solvent.

13. **Ensure that the waste reservoir is installed on the Reactor and that it is connected by a line to another larger waste container.**
14. **Flip all the Lower Manifold drain levers to the OPEN RVs position.**

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15. **Drain the RVs on Side A of the Reactor by turning the Delivery valve to HIGH PRESSURE GAS and the Vent valve to CLOSED.**
16. **Repeat steps 2 through 15 for the solvents in position B, C, and D to determine and record the delivery times for the remaining three solvents. As a guide, the delivery times determined in the Argonaut Technologies laboratories are listed in Figure 23, but actual delivery times may be different.**

AUTOMATED FUNCTIONS

Follow the procedure in Chapter 8, "Using the ASW Option," by programming a wash cycle and watching it run. This will confirm that the ASW is operating correctly and that it can be used without supervision.

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6

PREPARATION FOR SYNTHESIS

GENERAL

Ensure that the system has been properly installed.

⚠ WARNING

The Controller should not be operated in the presence of flammable gases or fumes. Operation of any electrical instrument in such an environment constitutes a definite safety hazard.

LINE POWER

To turn on the line power, press the switch on the rear panel of the Controller located above the power cord towards 1. The Controller initializes and displays the start-up screen (Figure 24).

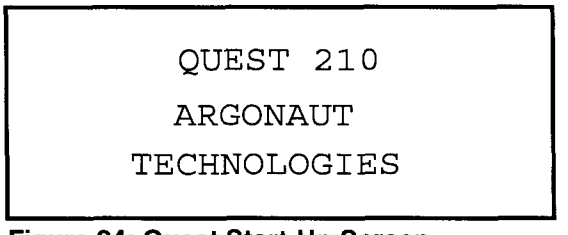


Figure 24: Quest Start-Up Screen

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REMOVING REACTION VESSELS

This procedure describes how to remove reaction vessels (RVs).

⚠ CAUTION

Always wear gloves and eye protection when working with the Quest.

1. **Rotate the Reactor so that the reaction vessels for Bank A are facing forward.**
2. **Raise the Upper Manifold by inserting the levers into the slots of the blue handles of the Upper Manifold (early production Quest 210 Systems have two steel pins instead of the slot in the Upper Manifold handles).**
3. **While pushing in the silver buttons on the Upper Manifold handles, pull the levers up. Slide the Upper Manifold up as far as possible until the silver buttons snap out, indicating that the manifold is locked into position.**
4. **Swing the Upper Manifold out of place by removing a pin either at the left- or right-hand side of the Upper Manifold.**
5. **Using the RV extraction tool (red pliers) supplied in the Quest starter kit, grasp the top of the reaction vessels (Figure 25).**



Figure 25: Grasping the Top of the RV

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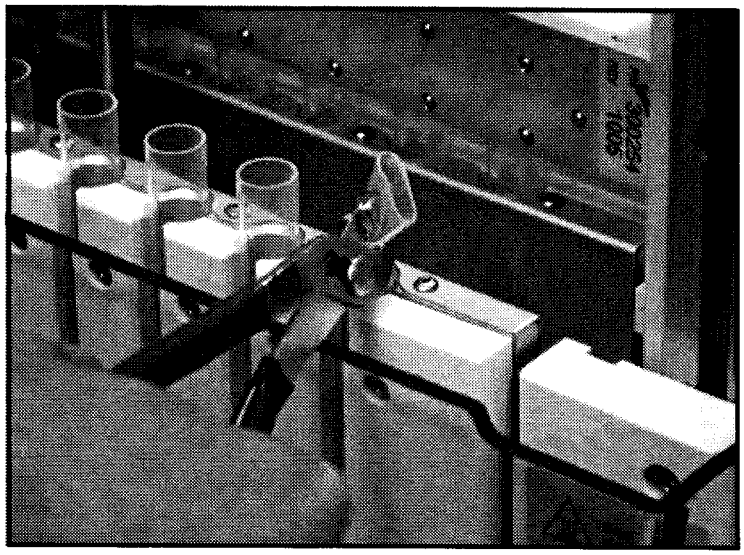


Figure 26: Breaking the RV free of the Lower Drain Manifold

6. **"Twist-up" using the reaction vessel holder as a fulcrum (Figure 26) to break the reaction vessel free (Figure 27).**
7. **The RV can now be removed by hand.**

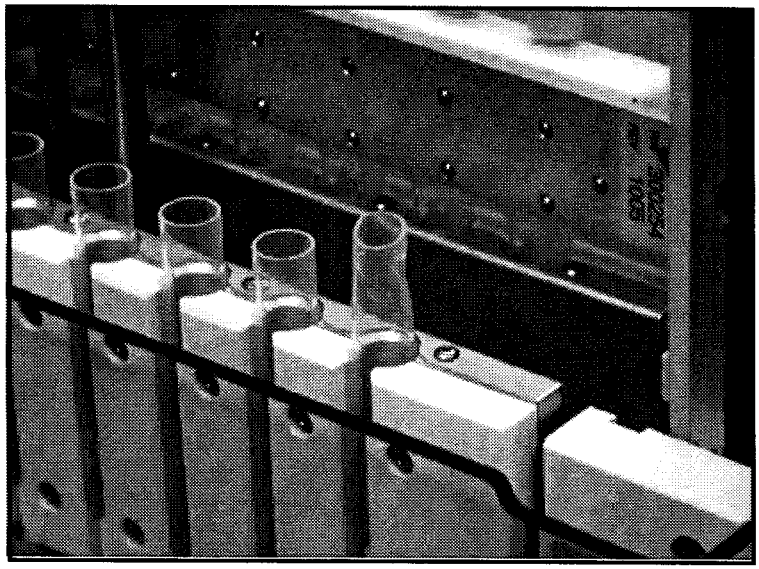


Figure 27: The RV free of the Lower Manifold

8. **Use the RV Retrieval Magnet to remove the agitator magnet from the vessel.**
9. **Wash the agitator magnets with an appropriate solvent such as acetone.**

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10. **Remove the Luer Plugs (twist and pull) from the Upper Manifold, rinse them with an appropriate solvent (e.g., acetone), and wipe clean.**
11. **Use a wash bottle or syringe to rinse the Luer Ports of the Upper Manifold with an appropriate solvent. Hold a soft cloth or paper towel beneath the Upper Manifold to absorb the rinse solvent.**
12. **Use a cotton swab wetted with an appropriate solvent to wipe the inside of the Luer Ports of the Upper Manifold and to wipe around the Upper Manifold male Teflon reaction vessel fittings.**
13. **Use a cotton swab wetted with an appropriate solvent to wipe clean the male Teflon reaction vessel fittings of the Lower Manifold. Lift the vessel collars and wipe around the bottom of the male Teflon reaction vessel fittings.**
14. **Finally, re-examine the Upper and Lower Manifold male reaction vessel fittings. Clean the fittings and remove any residue or particulates with a soft cloth and an appropriate solvent. Do not allow any particulates to enter the drain holes of the Lower Manifold male reaction vessel fittings. Take care not to scratch the male reaction vessel fittings as this may result in leaks.**

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INSTALLING REACTION VESSELS

This procedure describes how to install reaction vessels (RVs).

Two sizes of reaction vessels (RVs) are available: 5 mL and 10 mL. This procedure can be used to install both sizes of reaction vessels. To perform a synthesis, either actual or blank RVs must be installed on all 10 vessel positions of each bank.

NOTE

The Reactor has a Side A and Side B. Each side contains a bank of 10 reaction vessels. If less than 10 reactions are to be performed, blank vessels must be installed onto the unused reaction vessel positions.

1. **Determine if your synthesis requires 5 mL or 10 mL reaction vessels. For proper heating the Controller must be programmed with the size of the reaction vessel. To program the reaction vessel size do the following:**

- Press the MODE button to display the Set Temperature menu.
- Press the FORWARD button and move the cursor to the RV's line
- To change the vessel size for Bank A, align the cursor next to A:. Press either the + or – VALUE SETTING button to select the appropriate vessel size. Verify that the selected vessel size corresponds to the vessels actually on the instrument.
- To change the vessel size for Bank B, align the cursor next to B:. Press either the + or – VALUE SETTING button to select the appropriate reaction vessel size.

2. **If the synthesis uses a solid phase support (resin), weigh and add the resin to each reaction vessel. As a general rule up to 200 mg of ArgoGel or similar type resin can be used with 5 mL reaction vessel. The maximum amount of resin used depends upon the resins density, swelling characteristics, and the solvents used in the synthesis.**
3. **Slide ten reaction vessels between the aluminum heating plate and spring loaded insulators on one side of the Reactor (e.g., either Reaction Bank A or B). Press them down over the male Teflon reaction vessel fittings on the Lower Manifold *but do not attempt to fully seat the reaction vessels at this time.***

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⚠ WARNING

Always use frits in the reaction vessels, even if the reaction is solution phase. Frits prevent particulates from harming or clogging the Lower Manifold.

4. Place the Reaction Vessel Insertion Tool on top of the reaction vessel as shown in Figure 28.

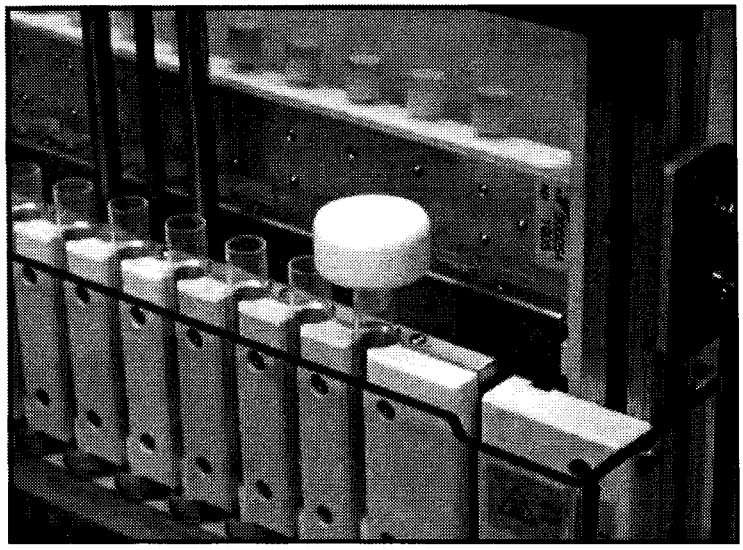


Figure 28: Inserting the 5 mL RV Insertion Tool

5. Press down on the reaction vessel with the RV Insertion Tool to seat the vessel over the male Teflon reaction vessel fitting of the Lower Manifold (Figure 29).

➤ NOTE

The correct RV Insertion Tools must be used for the 5 mL and 10 mL RVs. These Insertion Tools are not interchangeable between the two RV sizes.

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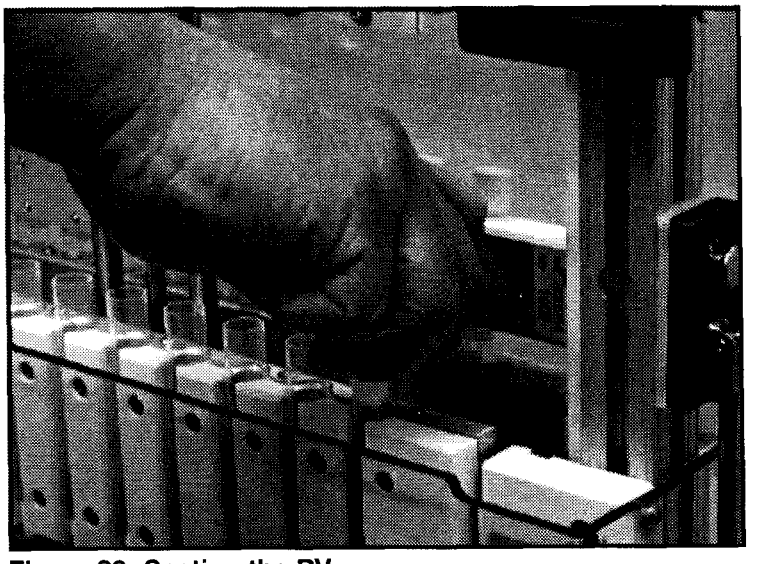


Figure 29: Seating the RV

6. **Check that the vessels are fully seated over the male Teflon reaction vessel fittings of the Lower Manifold. If the vessels are seated properly, the tops of the vessels will be horizontally uniform and the frits will be seated just above or on the male Teflon RV fittings of the Lower Manifold (Figure 30).**

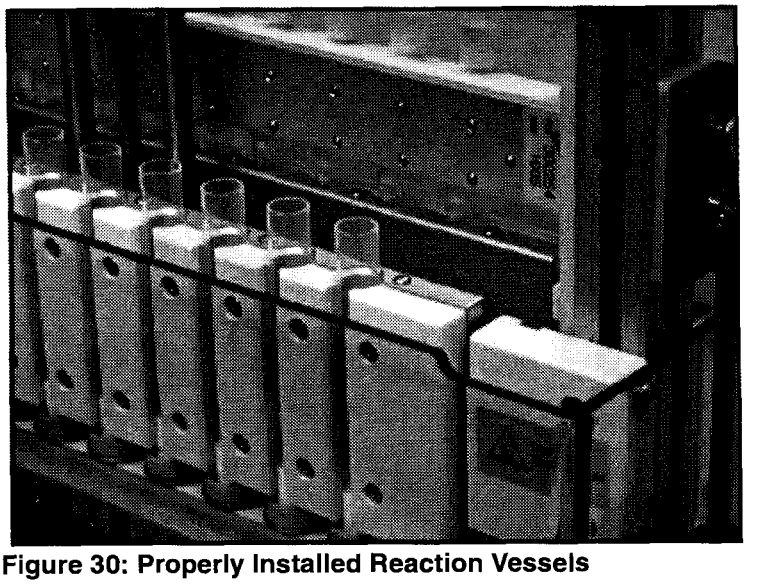


Figure 30: Properly Installed Reaction Vessels

7. **Move the agitator to the up position by pushing the AGITATOR UP button located in the upper right corner of the Controller panel (Figure 31).**

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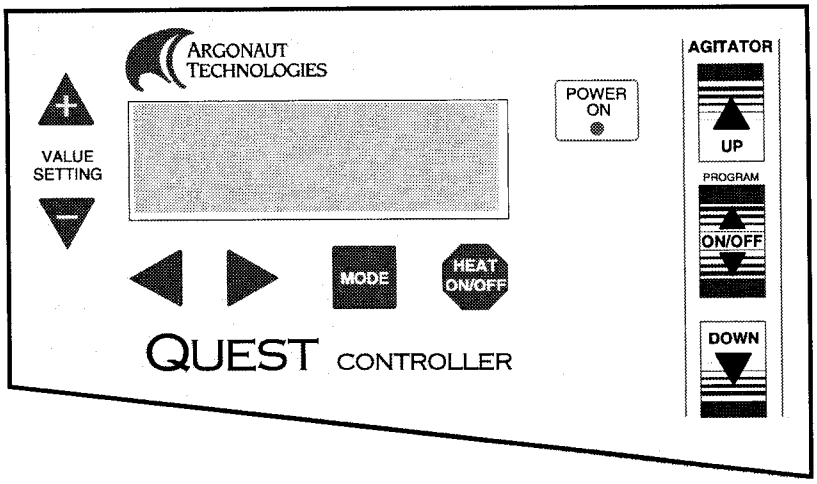


Figure 31: Controller Panel

8. Place the correct agitator magnet into each reaction vessel. Use the RV retrieval magnet to align the agitator magnets with the agitator bar.

Lower the Upper Manifold onto the reaction vessels as follows:

1. Push in the silver buttons on the blue handles of the Upper Manifold.

2. Slowly slide the Upper Manifold onto the tops of the reaction vessels. Do not push the male Teflon fittings of the Upper Manifold into the reaction vessels at this time.

3. Use the Rotating Levers on either side of the Reactor to simultaneously lock both sides of the Upper Manifold into place:

- First, move the levers so they stick out from the sides of the Reactor parallel to the bench top.
- Next, rotate the levers towards the front of the Reactor and insert them into the slots in the blue handles of the Upper Manifold.
- Finally, push down on the Rotating Levers and slide the Upper Manifold into the reaction vessels. Keep pushing until the silver buttons on the blue handles snap out indicating that the Upper Manifold is locked into position.

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WASTE RESERVOIR

The Reactor is equipped with a Waste Reservoir for waste collection. The Waste Reservoir has a valve for draining - a hose can be attached to the valve with the other end placed into a larger waste container.

To install the Waste Reservoir, place the empty reservoir onto the Waste Tray located below the Lower Manifold of the Reactor.

Using the Waste Tray Handles, lift the tray until the top of the Waste Reservoir is in contact with the Lower Manifold. The silver buttons on the Waste Tray Handles will snap out to verify that the Waste Tray is locked into position.

To remove the Waste Reservoir, push the silver buttons on the handles of the Waste Tray and slowly slide the tray down to its lowest position.

Remove the Waste Reservoir and dispose of waste in accordance with local laws and regulations.

⚠ WARNING

The Waste Tray will be heavy when full of solvent. Lower the tray carefully.

⚠ WARNING

Dispose of all hazardous waste in accordance with all local laws and regulations.

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SOLVENT BOTTLE

Parallel delivery of solvents and reagents to the Upper Manifolds of the Reactor are from the solvent bottle. To work with different solvents or reagents the solvent bottle must be changed, and it is essential to have sufficient solvent in the bottle to complete the synthesis. This procedure describes how to change the solvent bottle.

WARNING

Always wear eye protection and appropriate clothing when operating the Reactor.

1. **Ensure that the Upper Manifold Membrane Valve is in the OPEN RVs position.**
2. **Vent the Upper Manifold by turning the Control Valves to CLOSED and VENT. Wait approximately 5 seconds.**

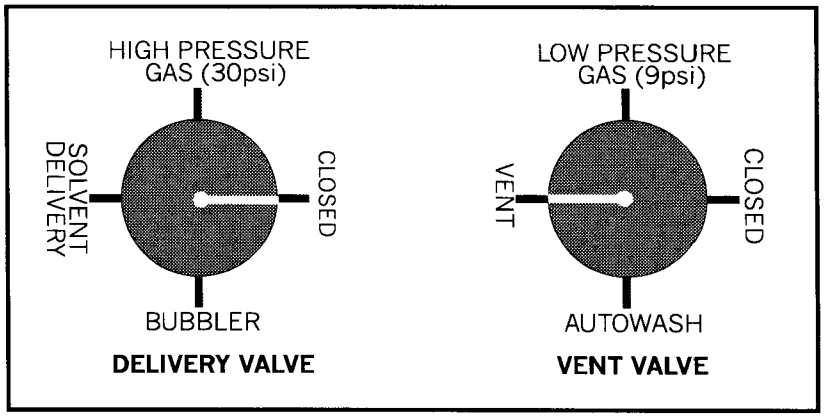


Figure 32: Manifold Control Valves - Closed and Vent

3. **Vent the solvent bottle by turning the valve on top of the Solvent Bottle Cap Assembly towards the tube labeled VENT. Allow the solvent bottle to vent for approximately 60 to 90 seconds.**

WARNING

Always vent the bottle prior to unscrewing the cap.

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4. **Back-flush the solvent in the Upper Manifold and solvent delivery line into the solvent bottle by turning the Control Valves to SOLVENT DELIVERY and LOW PRESSURE GAS.**

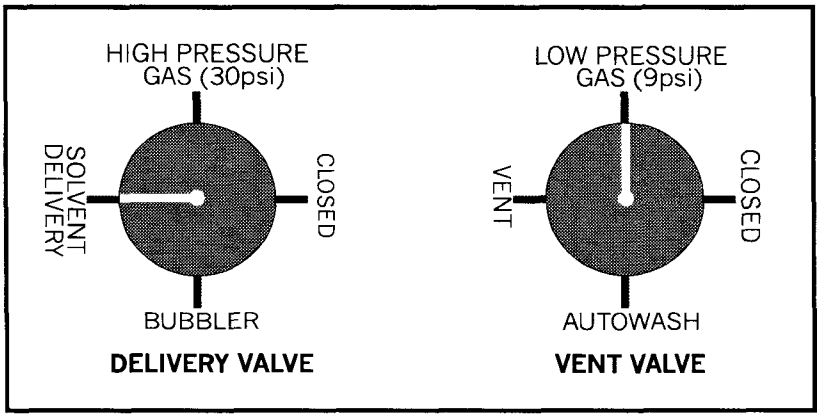


Figure 33: Manifold Control Valves - Solvent Delivery and Low Pressure Gas

5. **Slowly increase the Low Pressure Gas flow rate. When the solvent pick-up line in the solvent bottle begins to bubble, the line has been completely purged of solvent.**
6. **Turn both Control Valves to CLOSED, and unscrew and remove the Solvent Bottle Cap Assembly from the solvent bottle.**

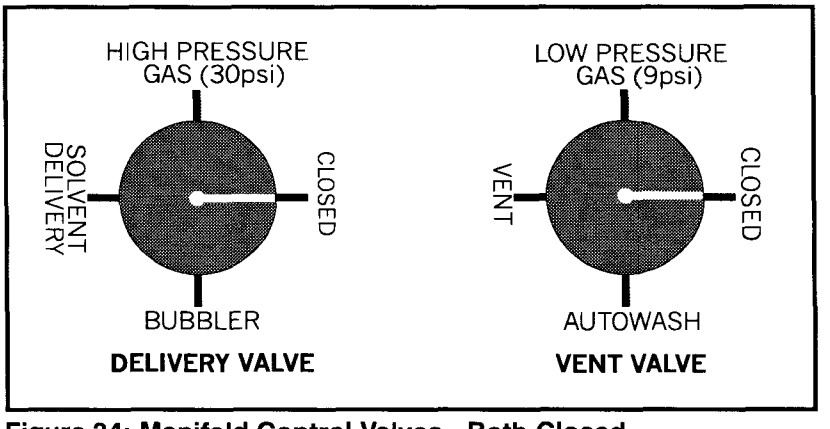


Figure 34: Manifold Control Valves - Both Closed

7. **Remove the white Teflon Solvent Bottle Cap Seal from the neck of the old solvent bottle.**
8. **Wipe off any residual solvent from the Solvent Bottle pick up line and replace the Polyethylene Solvent/Reagent Filter on the end of the line.**

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➤ NOTE

Change the Polyethylene Solvent/Reagent Filter once per week.

9. **Put the new solvent bottle into the Bottle Safety Carrier and remove the bottle cap.**

➤ NOTE

Always place the solvent bottle in the plastic Safety Carrier when working with the Quest.

10. **Insert the Teflon Bottle Cap Seal into the neck of the new solvent bottle.**
11. **Insert the Solvent Bottle Cap Assembly pick-up line with the new Polyethylene Solvent/Reagent Filter through the neck of the solvent bottle. Screw the Solvent Bottle Cap Assembly onto the new solvent bottle.**
12. **Turn the valve on top of the Solvent Bottle Cap Assembly toward the tube labeled PRESSURE. Wait 60 to 90 seconds for the solvent bottle to pressurize.**

GAS

Verify that there is adequate inert gas as well as any necessary reactive gas available to complete the reaction.

The flow rate of the inert gas can be monitored by connecting a bubbler to the Bubbler Port and turning the Delivery valve to BUBBLER and the Vent valve to LOW PRESSURE GAS.

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7

OPERATION

This chapter provides step-by-step instructions for operating the Quest 210 Organic Synthesizer to complete a liquid or solid phase chemistry reaction.

Prior to starting a synthesis

- Read this Quest 210 User Manual.
- Verify that the Quest was installed according to the procedures in Chapter 4, "Quest 210 Installation."
- Verify that the requirements described in Chapter 6, "Preparation For Synthesis," have been fulfilled.
- Familiarize yourself with the nomenclature and various components of the Reactor.

This chapter explains the following

- Programming the reaction temperature, temperature duration, and agitator values.
- Adjusting the Agitator Bar height
- Closing the Upper Manifold Membrane valve.
- Performing parallel solvent additions.
- Adding reagents or solvents to the reaction vessels through the Luer Ports.

Determine the steps that you wish to use to complete a liquid or solid phase chemistry reaction, then perform the corresponding procedures provided in this section as needed. When you have finished your reaction, clean the instrument as described in Chapter 8, "Using the ASW Option." or Chapter 9, "Cleaning & Maintenance."

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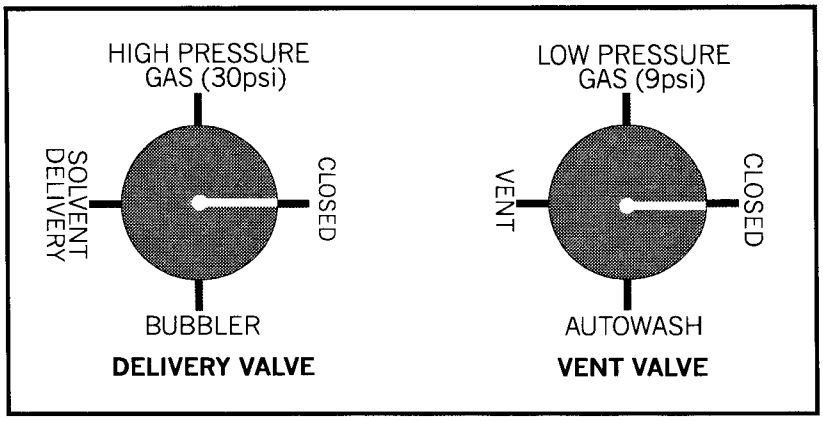


Figure 35: Manifold Control Valves - Both Closed

FILLING THE DEAD VOLUME

This procedure fills the dead volume between the bottom of the RV frit and the lower manifold drain valve with solvent. This is necessary to eliminate air from the system which might otherwise affect the synthesis.

1. **Toggle the Upper Manifold Membrane switch to OPEN. Install a bottle of methanol (MeOH) onto the system by following the procedures in Chapter 6.**
2. **Deliver approximately 2 mL of MeOH to each RV by rotating the Control Valves to SOLVENT DELIVERY and VENT. Stop the flow by turning the Delivery Valve to HIGH PRESSURE GAS. The high pressure gas purges the residual solvent in the delivery pathway into the reaction vessels. After a few seconds rotate both Control Valves to CLOSED.**
3. **Rotate the Vent valve to VENT. Turn the Low Pressure Gas Valve completely clockwise to close it. Rotate the Vent Valve to LOW PRESSURE GAS to deliver a steady stream of inert gas to the RVs.**
4. **Open the drain valve of one RV and increase the inert gas flow to the RV by slowly turning the Low Pressure Gas Valve counter-clockwise. When the solvent reaches the top of the RV frit, close the drain valve.**
5. **Turn the Low Pressure Gas Valve completely clockwise to close the valve.**
6. **Rotate the Vent valve to VENT and then to LOW PRESSURE GAS.**
7. **Repeat steps 4 and 5 for each remaining RV.**

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REAGENT AND SOLVENT DELIVERY

UPPER MANIFOLD MEMBRANE VALVE

The Upper Manifold Membrane isolates the reaction vessels from each other and prevents any cross contamination between vessels. It is controlled by the Upper Manifold Membrane valve located in the upper corner of the Reactor. In the SEAL RVs position this switch delivers 30 psig (3.1 bar) to the membrane within the Upper Manifold, which seals the reaction vessels to create a closed reaction environment. In general, the valve is set to SEAL RVs during reactions.

WARNING

The Upper Manifold Membrane seals the reaction vessels. Solvent or gas cannot be delivered to the reaction vessels by the Manifold Control Valves when the Upper Manifold Membrane valve is set to SEAL RVs. To deliver solvent or gas to the reaction vessels using the Manifold Control Valves the Upper Manifold Membrane valve must be set to OPEN RVs.

PARALLEL DELIVERY

Solvent from the solvent bottle can be added to a bank of reaction vessels in parallel using the Manifold Control Valves. Parallel solvent addition can be used to add a reagent or solvent to a reaction mixture or for flushing the system after a synthesis. To deliver solvent to a bank of reaction vessels in parallel use this procedure.

WARNING

Always wear gloves and safety glasses when operating the Reactor.

1. **Before delivering solvent to the reaction vessels check the following:**
   - The Solvent Bottle Cap Assembly is screwed onto the solvent bottle.

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- The valve on top of the Solvent Bottle Cap Assembly is turned toward the tubing labeled PRESSURE.
- The Upper Manifold Membrane valve is turned to Open RVs.

2. To deliver solvent to a bank of reaction vessels in parallel, pressurize the solvent bottle and vent the reaction vessels by turning the Manifold Control Valves to SOLVENT DELIVERY and VENT (Figure 36).

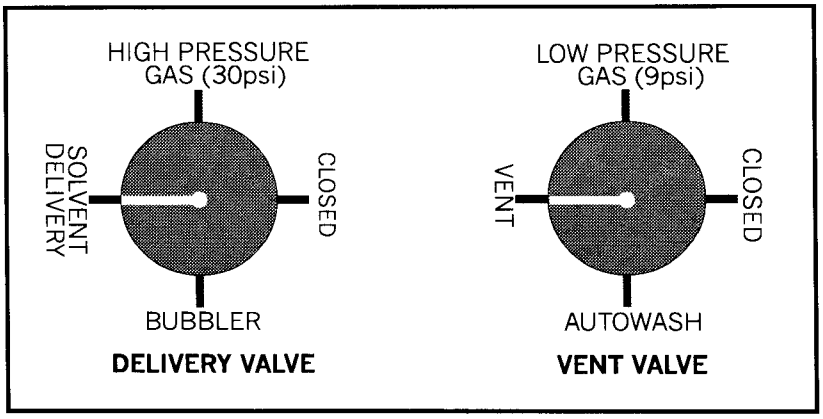


Figure 36: Pressurizing the Solvent and Venting the RVs

3. When the solvent has reached the desired level, purge the manifold by *slowly* rotating the Delivery valve from SOLVENT DELIVERY to HIGH PRESSURE GAS. Allow the Upper Manifold to purge for approximately 5 seconds. Purging the Upper Manifold will flush residual solvent into the reaction vessels using inert gas (Figure 37).

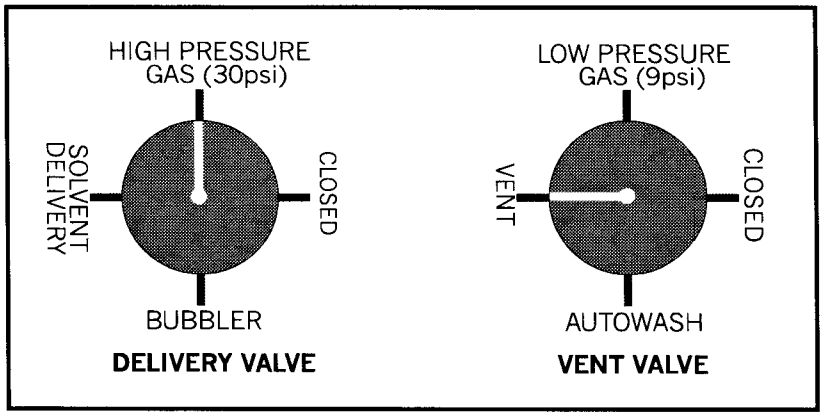


Figure 37: Purging the Manifold

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4. **Seal the Upper Manifold by first turning Delivery valve to CLOSED, then turn the Vent valve to CLOSED (Figure 38).**

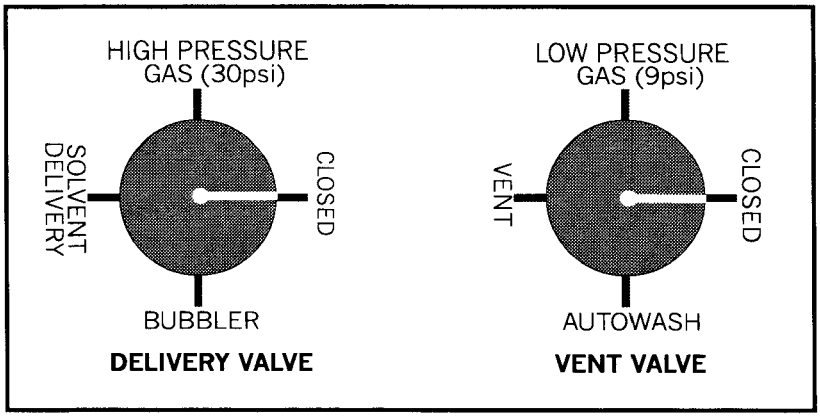


Figure 38: Sealing the Manifold

INDIVIDUAL DELIVERY

Liquid or solid reagents can be added to reaction vessels manually through the Luer Ports located on the Upper Manifold. Typically, reagents are added using syringes or pipettes. To add a liquid or solid reagent to a reaction vessel use this procedure:

1. **The reaction vessels may be under pressure, so before adding reagents through the Luer Ports the system must be vented. To vent the vessels, rotate the Delivery valve to CLOSED, and slowly rotate the Vent valve to VENT (Figure 39). This vents the pressure in the reaction vessels slowly and prevents bumping the solution.**

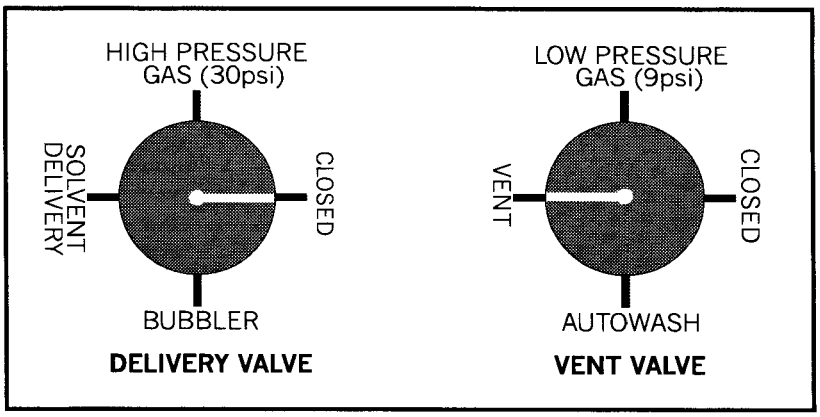


Figure 39: Venting the RVs

2. **Remove the Luer Plug by slowly twisting it while pulling the plug out of the Upper Manifold.**

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WARNING

The reaction vessel contents may be under pressure. Wear safety glasses, gloves and appropriate clothing.

WARNING

Do not open the upper Luer Ports when the RVs are being heated. Turn off the heaters and allow the reaction vessels to return to ambient temperature before removing the Luer Plugs.

3. Add solid or liquid reagent through the Upper Manifold Luer Port.

- To add liquid reagents, insert a pipette or syringe through the Luer Port and dispense the solution into the reaction vessel.
- To add a solid, use a funnel to transfer reagent into the reaction vessel through the Luer Port. If residual solid is left on the bore of the Luer Port, rinse the solid into the reaction vessel using a small amount of solvent.

WARNING

Be careful not to score the interior surfaces of the Luer Port. Scratching the surface can affect the sealing of the Luer Plug.

4. Seal the reaction vessel by inserting the Luer Plug into the Luer Port.

5. Rotate both Manifold Control Valves to CLOSED.

Using the Low Pressure Gas it is possible to purge the reaction vessel head space with the system gas (e.g., nitrogen, argon, etc.). An oil bubbler connected to the Bubbler Port located on top of the Reactor can be used to monitor the flow rate of the Low Pressure Gas. The Bubbler outlet should be pointed towards the back of the fume hood away from the Reactor. If an oil bubbler is not available, connect a length of 1/8 in. Teflon tubing to the Bubbler Port with its outlet pointed toward the back of the fume hood. To add reagents while maintaining an inert reaction environment use this procedure.

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NOTE

Purging reaction vessels with inert gas may result in solvent loss, especially when heating.

1. **Switch the Upper Manifold Membrane Valve to OPEN RVs.**
2. **Turn the Manifold Control Valves to BUBBLER and LOW PRESSURE GAS (Figure 40). This provides a low flow of inert gas to purge the reaction vessels. Low Pressure Gas will flow out of the Bubbler Port to the oil bubbler.**

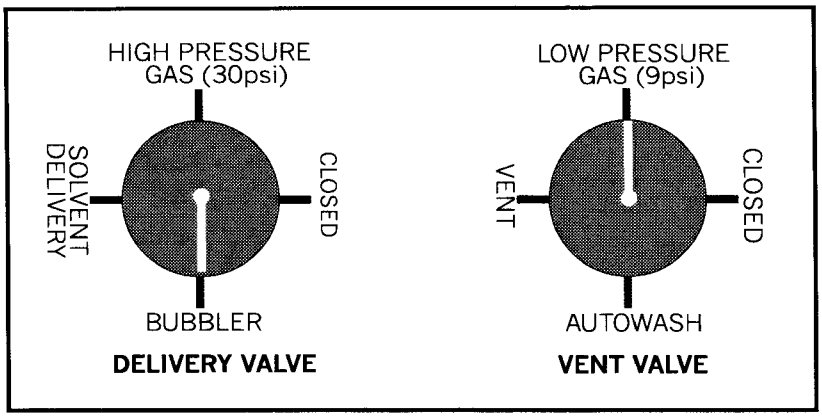


Figure 40: Purging the RVs

3. **Adjust the Low Pressure Gas Flow valve located on the top of the Reactor until you see a steady stream of gas bubbling through the oil bubbler with one of the Luer Plugs removed from the Upper Manifold. The gas flow rate can be modified by turning the Low Pressure Gas valve counter clockwise (left) to increase the gas flow and clockwise (right) to decrease the gas flow. This will ensure the establishment of an inert atmosphere when the Luer Plugs are removed for reagent addition.**
4. **Remove the Luer Plug from the first Luer Port (twist and pull) and add the reagent below the Teflon lines of the male Teflon reaction vessel fittings of the Upper Manifold. Take care not to score the interior surface of the Luer Port. Marring the surface can affect the sealing of the Luer Plug. After delivering the reagent, replace the Luer Plug (twist and push) and add reagent to the remaining RVs in the same manner as the first.**
5. **Blanket the reaction vessels with nitrogen by turning the Delivery valve to CLOSED (Figure 41).**

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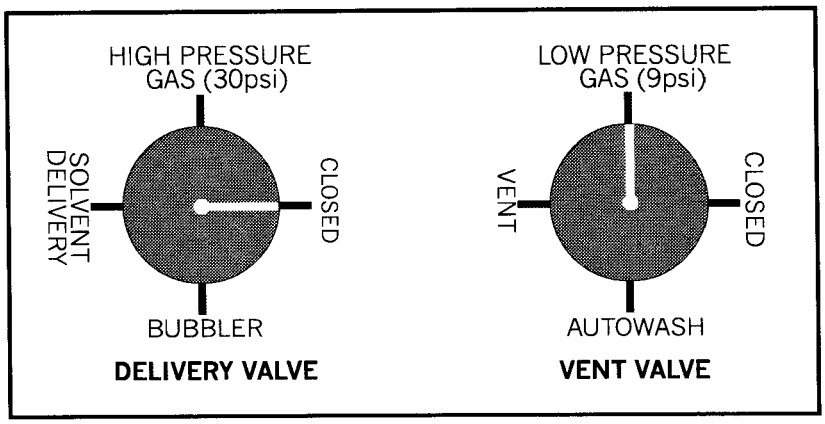


Figure 41: Blanketing RVs with Nitrogen

6. Then, turn the Vent valve to CLOSED (Figure 42).

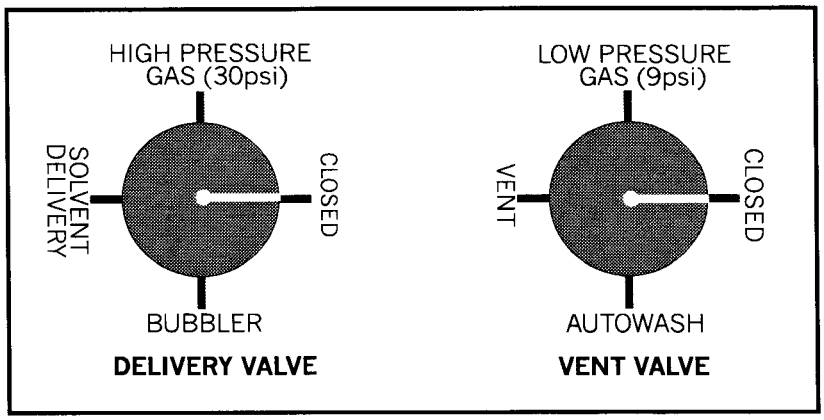


Figure 42: Both Valves Closed

7. Turn the Upper Manifold Membrane valve to SEAL RVs.

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PROGRAMMING

Reaction temperature, heating duration, and agitator values are programmed using the LCD screen and buttons on the Controller. Programming is facilitated by the system's menu-driven software. This section describes how to program the Controller.

AGITATOR BAR

Adjusting

The Agitator Bar is located between the two Banks of reaction vessels. Two round Agitator Stops on the Agitator Bar set the maximum agitator height. Use this procedure to set the maximum agitator height.

1. **Ensure that the Agitator Bar is in the UP position (move the agitator up by pushing the AGITATOR UP button located to the right of the Controller screen).**
2. **Use the 7/64" hex wrench supplied with the Reactor to loosen the Agitator Stops.**

- For 5 mL reaction vessels, adjust the Agitator Stops on the Reactor so the maximum magnet height is about 2 cm below the solvent level in the RVs.
- For 10 mL reaction vessels, adjust the Agitator Stops on the Reactor for the maximum magnet height. If the agitator magnets break the surface of the solvent, adjust the maximum magnet height so that the magnets are about 2 cm below the solvent level in the reaction vessel.

3. **Tighten the Agitator Stops so both stops are at the same height.**

Values

Use the buttons, LCD screen, and the AGITATING menu to program the following agitator values:

- MIX EVERY: Time between consecutive down strokes of the Agitator Bar (agitator frequency)
- UPSTROKE: Amount of time in seconds that the agitator bar remains at its highest position (the entered time must be less than the MIX EVERY entry)
- % UPWARD: Same as the UPSTROKE value but expressed as a percentage of the MIX EVERY value

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NOTE

Changing the UPSTROKE value will automatically change the % UPWARD value to the corresponding value. Likewise, changing the % UPWARD value will automatically change the UPSTROKE value.

Figure 43 shows a graphical representation of a typical agitator cycle to illustrate the relationships between the three values.

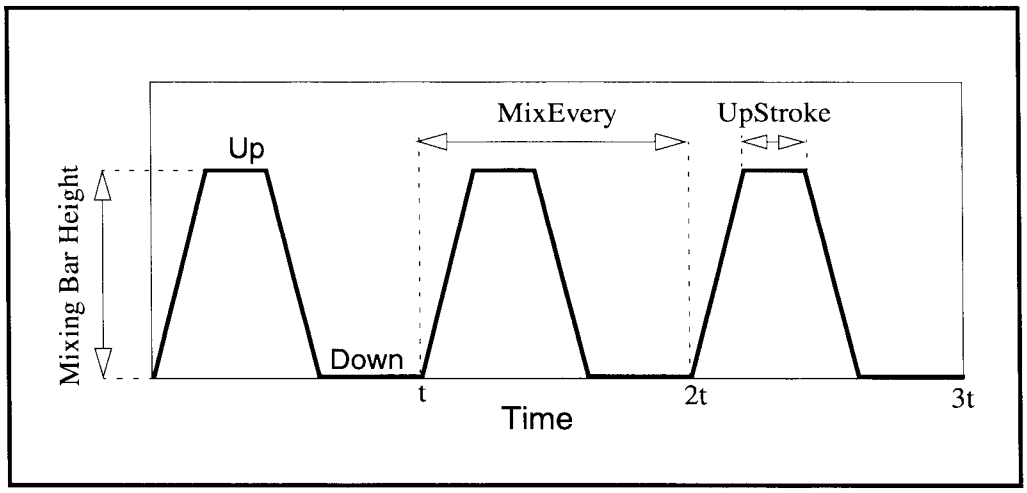


Figure 43: Example Agitator Cycle Plot

```
MixEvery   :    4.0 sec
UpStroke   :    3.8 sec
% Upward   :      95 %

- - Not Agitating - - -
```

Figure 44: Agitating Menu

An example of the AGITATING menu is shown in Figure 44. In this example:

- The agitator frequency (MIX EVERY) has been programmed for 4 sec/cycle, so the Reactor will complete a single agitator (one complete down and up cycle) every 4 seconds.
- The UPSTROKE and % UPWARD are programmed for 3.8 sec and 95%, respectively. This means that the Agitator Bar will spend 3.8 sec or 95% of its time at its highest position.

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As a general guideline, if the synthesis support is very delicate, increase the MIX EVERY time (e.g., 5 to 6 sec.) to reduce the supports exposure to mixing. If a liquid-liquid extraction is to be performed, decrease the MIX EVERY time (e.g., 1 to 2 sec) and increase the MIX SPEED (turn the MIX SPEED valve counter clockwise) to increase the mixing efficiency. Use this procedure to program the agitator values.

➤ NOTE

Program the agitator values so as to minimize splashing of solution onto the Upper Manifold Male Fittings. This will prevent build up of resin and/or particulates in the Upper Manifold Delivery Tubes.

1. **On the Controller, press the MODE button until the AGITATING menu is displayed (Figure 44).**
2. **Press the FORWARD and BACK buttons (← →) to select the "Mix Every" value (agitator frequency) on the AGITATING menu.**
3. **Use the VALUE SETTING buttons (↑↓) to increase or decrease the MIX EVERY time.**
4. **Press the FORWARD and BACK buttons (← →) to select the % Upward value (the amount of time the agitator bar remains in the up most position expressed as a percentage of the MIX EVERY time).**
5. **Use the VALUE SETTING buttons (↑↓) to increase or decrease the % Upward value.**
6. **Turn the agitator on by pushing the Agitator ON/OF button. If solvent is splashing up onto the Upper Manifold, readjust the height of the agitator bar via the agitator stops so that the magnets are lower in the reaction vessels.**

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Starting and Stopping

Three buttons located to the right of the Controller screen control the agitator as shown in **Figure 45.**

The middle button (ON/OFF) starts and stops the agitator. It also starts a program. The upper button (UP) raises the Agitator Bar to its highest position and moves the bar out of the way allowing the user to monitor the contents of the reaction vessels. The lower button (DOWN) lowers the Agitator Bar to its lowest position to facilitate the addition of solid or liquid reagents to the reaction vessels



Figure 45: Agitator Control Buttons

Mix Speed

The agitator speed is controlled using the MIX SPEED valve in the lower right hand corner of the Controller. This valve controls the gas pressure used to drive the Agitator Bar. Turning the MIX SPEED valve to the right (clockwise) slows the agitator. Eventually the valve will close completely and the agitator will stop. Turning the valve counter clockwise opens the valve. When the valve is open completely the agitator is at its maximum speed. For viscous solutions or solutions with a large amount of resin, decrease the MIX SPEED (turn the Mix Speed Valve clockwise) to facilitate efficient mixing.

➤NOTE

The Mix Speed Valve is fully closed during shipment. Turn it counter clockwise to start agitation.

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TEMPERATURE CONTROL

Use the buttons, LCD screen and SET TEMPERATURE menu to program the reaction temperature and heating duration. The temperature range of the Quest 210 is -40°C to +130°C.

For cooling below ambient temperature an external recirculating chiller is required.

WARNING

Do not program a temperature which exceeds the boiling point of the reaction solvent.

NOTE

When using the chiller, reactions can be performed on one side only of the Reactor.

HEATING

Reaction temperature and heating duration are programmed using the Set Temperature menu. The reaction temperature and heating duration for reaction vessel Bank A and B are programmed independently. Use this procedure to program the reaction temperature and heating duration.

WARNING

When heating, the Reactor will become hot. The symbol at the right is to warn of a hot surface. Use caution when working near or on the Reactor. Allow the Reactor to cool before attempting to access the reaction vessels



**Caution!
Hot Surface**

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1. **Press the MODE button until the Set Temperature menu is displayed (Figure 46).**

```
- - - Set Temperature - -

A:      60C     03:00    ON
B:      20C     00.00    OFF
RV's     A:5mL    B:5mL
```

Figure 46: Set Temperature Menu

2. **To program the reaction temperature for Bank A, use the BACK or FORWARD (←→) buttons to move the cursor to line labeled A:.**
3. **Using the same buttons, move the cursor across to the temperature value. Use the VALUE SETTING (↑↓) buttons to increase or decrease the value until the desired reaction temperature is displayed.**

⚠ *WARNING*

As a general rule, do not program a temperature above the boiling point of the reaction solvent being used. This prevents over pressurization of the system.

4. **Use the FORWARD or BACK buttons (← →) to move the cursor to the time setting. Use a combination of the FORWARD or BACK (← →) and the VALUE SETTING (↑↓) buttons to program the temperature duration. Heater duration is programmed in minutes and seconds. In Figure 46 the heater duration is programmed for 3 minutes.**
5. **Program the reaction temperature and heater duration for Bank B in a similar manner as Bank A.**
6. **Use the FORWARD or BACK buttons (← →) to move the cursor to the bottom of the screen to program the reaction vessel size. Use the VALUE SETTING (↑↓) buttons to select either the 5 mL or 10 mL vessel for Banks A and B.**
7. **Use the FORWARD or BACK (← →) buttons to move the cursor to line A. Press the HEAT ON/OFF button to start the heating program. The cursor must be on line A: of the Set Temperature menu to activate the heaters for Bank A. To activate the heaters for Bank B use the FORWARD or BACK (← →) buttons to move the cursor to line B: and press the HEAT ON/OFF button.**

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8. **To monitor the reaction temperature select the Monitor Temperature screen on the Controller by pressing the MODE button (Figure 47).**
9. **When the heaters reach the programmed temperature the software starts the clock for the heating duration.**
10. **The heaters shut off when the heating duration time runs out and the reaction vessels will begin cool to ambient temperature.**

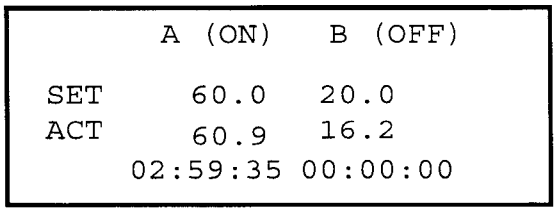


Figure 47: Temperature Monitor Screen - Heating

To turn the heaters off manually press the HEAT ON/OFF button to display the SET TEMPERATURE menu. Use the → button to move the cursor next to either Stop A or Stop B, depending on which reaction vessel bank heater you wish to turn off. Press the HEAT ON/OFF button to abort the temperature program.

COOLING

For cooling below ambient temperatures, an optional external recirculating chiller is required. Consult the recirculating chiller user manual for instructions on its installation and operation. Fittings are provided on the left and right side of the heater banks to connect the tubing from the recirculating chiller.

For systems with serial number 2491055 and below, 3/16 in. brass barbed fittings are used. Starting with serial number 2941056 these connections were changed to 1/16 in. male brass pipe fittings. To hook a recirculating chiller to systems with serial number 2941056 and higher, use the Quest Chiller Interface Kit (Part Number 900123). Otherwise order Part Number 900137.

The Chiller Interface Kit requires the recirculating chiller inlet and outlet tubes be terminated with ¼ inch male pipe fittings. Use the following procedure to connect a recirculating chiller.

1. **Install the Chiller Interface Kit onto the Reactor using the instructions supplied with the kit.**
2. **Connect the recirculating chiller outlet tubing to the ¼ in. brass female pipe fitting at the left of the reaction vessel bank.**

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3. **Connect the recirculating chiller return tubing to the ¼ in. brass female pipe fitting on the right of the reaction vessel bank.**
4. **Ensure that the recirculating chiller hoses and brass fittings are covered with foam insulation.**
5. **Turn on the recirculating chiller to pump the coolant liquid through the cooling channel of the Reactor.**
6. **Press the MODE button until the MONITOR screen is displayed.**
7. **Adjust the bath temperature of the recirculating chiller until the desired reaction temperature is achieved. The reaction temperature for each bank is read from the ACT line of the screen (Figure 48).**

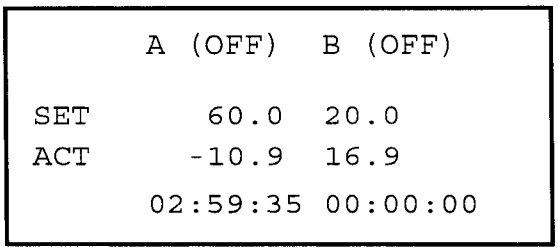


Figure 48: Temperature Monitor Screen - Cooling

⚠ *WARNING*

Disconnect the chiller when not in use. Purge the cooling channel of refrigerant liquid after a the chiller is disconnected. Do not heat a reaction vessel bank with the chiller connected, or with residual liquid in the cooling channel.

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DRAINING REACTION VESSELS

⚠ CAUTION

Before draining reaction vessels make sure the instrument has returned to room temperature and vent the Upper Manifold by turning the Control Valves to CLOSED and VENT. Purge the Upper Manifold with Low Pressure Gas and turn the Upper Manifold Membrane Valve to OPEN RVs.

NON-METERED DRAINING

Reaction vessels are drained using 30 psig (3.1 bar) High Pressure Gas and the drain valves located below each vessel on the Lower Manifold. To drain an individual reaction vessel, rotate the Delivery valve to HIGH PRESSURE GAS and push down on the reaction vessel drain valve lever. Return the lever to the SEAL RVs position when finished. To drain a bank of reaction vessels, rotate the Delivery valve to HIGH PRESSURE GAS and use the Parallel Drain Lever to open the ten drain valves simultaneously.

METERED DRAINING

For metered draining of reaction vessels use the Low Pressure Gas delivery with the following procedure:

1. **After the instrument has returned to room temperature, open the Upper Manifold Membrane valve by following these steps:**
   - Vent the Upper Manifold by turning the Manifold Control Valves to CLOSED and VENT.
   - Supply Low Pressure Gas to the Upper Manifold by turning the Manifold Control Valves to CLOSED and LOW PRESSURE GAS.
   - Turn the Upper Manifold Membrane valve to OPEN RVs.
2. **Verify that the Waste Reservoir is installed.**
3. **Move the agitator up by pushing the Agitator UP button on the Controller.**
4. **To drain an individual reaction vessel to the Waste Reservoir turn the Control Valves to HIGH PRESSURE GAS and CLOSED. Open the Lower Manifold Drain Valve by pushing the Drain Lever for the**

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reaction vessel down. To drain all ten reaction vessels in parallel to the Waste Reservoir use the Parallel Drain Lever.

5. **Close the Lower Manifold Drain Valves by flipping the Drain Levers upward.**
6. **After draining the vessel(s), set both Control Valves to CLOSED.**

PRODUCT COLLECTION

To collect products on the Reactor, remove the Waste Reservoir and install an appropriate collection rack. The following collection racks are available:

- Twenty 20 mL or 40 mL scintillation vials
- Twenty 20 mm x 125 mm test tubes
- Twenty 13 mm x 100 mm test tubes

Follow the procedure below.

NOTE

For product collection, use the 9 psig (1.6 bar) Low Pressure Gas, instead of the 30 psig (3.1 bar) High Pressure Gas to provide the head pressure for reaction vessel draining. Using higher pressure Drain Gas will cause splashing of the products in the collection test tubes/vials.

WARNING

Always wear gloves, eye protection, and proper clothing such as a lab coat when working with the Reactor.

1. **Remove the Waste Reservoir and dispose of the waste in accordance with all local laws and regulations.**
2. **Rinse the bottom of the Lower Manifold Reactor where the Teflon lines for collection are located with a solvent compatible with your synthesis and wipe clean.**
3. **Place the collection rack under the Reactor and align the feet of the collection rack with the four holes in the Waste Tray.**
4. **Push the silver buttons on the Waste Tray Handles and raise the Waste Tray slowly to its upper notched position so that the Lower Manifold outlet tubes are below the lip of the test tubes/vials. The**

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silver buttons will snap out to verify that the collection tray is locked into position.

5. **Raise the Waste Tray slowly to its upper notched position being careful not to bend any of the Teflon tubes protruding below the Lower Manifold.**
6. **Ensure that each Teflon outlet tubes is inside its own test tube/vial and that the two vent lines are clear of any test tubes/vials.**
7. **Move the agitator up by pushing the AGITATOR UP button on the Controller.**
8. **Vent the Upper Manifold of the Reactor to prepare for the draining of the reaction vessels:**
   - Turn the Manifold Control Valves slowly to CLOSED and VENT.
   - Toggle the Upper Manifold Membrane Valve to OPEN RVs.
   - Close the Low Pressure Gas Valve by turning it clockwise (to the right).
   - Turn both Manifold Control Valves to CLOSED and LOW PRESSURE GAS.
9. **Collect the contents of the reaction vessels into the test tubes/vials by doing the following:**
   - Open the Lower Manifold Drain Valve for the first reaction vessel.
   - Slowly turn the Low Pressure Gas Valve counterclockwise (to the left) until the reaction vessel begins to empty. Adjust the Low Pressure Gas valve pressure to control the speed at which the reaction vessel empties. Drain the contents of the reaction vessel slowly to prevent the product from splashing.
10. **After the first reaction vessel has emptied proceed to empty the other reaction vessels.**
11. **Close the Lower Manifold Drain Valves after each product collection**

➤ NOTE

Each time you finish a reaction or synthesis, follow the procedures described in Chapter 8, "Using the ASW Option,", or Chapter 9, "Cleaning & Maintenance."

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8

USING THE ASW OPTION

This chapter provides step-by-step instructions to perform an automated solvent wash using the Automated Solvent Wash module (ASW). The reaction vessels will be filled with solvent, agitated, and drained several times.

Make sure that the installation process has been completed, including the proving run, and that washing times have been recorded for the solvents that you will use.

PROGRAMMING

The Automated Solvent Wash firmware can support up to 60 wash programs and a single program can have up to 25 lines. However, the cumulative total number of lines for all programs cannot exceed 120 lines.

Programs are entered through the Auto Solvent Wash menu and require the following information:

1. **Program number**
2. **Solvent volume**
3. **Reactor side**
4. **Draining method**
5. **Number of repetitive washes with a specific solvent**
6. **Specific solvent**
7. **Solvent addition time in seconds**
8. **Agitator time**
9. **Drain time**

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PROGRAM NUMBER

1. **Press the MODE button until the AUTO SOLVENT WASH menu is displayed.**
2. **A flashing cursor is aligned to the right of Pgm: (program). Use the VALUE SETTING buttons (+ or –) to increase or decrease the program number until Pgm 1 is displayed. The screen should now look like Figure 49.**

```
--AUTO SOLVENT WASH-
 Pgm:1    5ml Side:A&B
 #  R S Add  Mix  Pul
----Empty Program---
```

Figure 49: Auto Solvent Wash Menu with Program 1 Selected

SOLVENT VOLUME

1. **Press the FORWARD button (→) once to move the flashing cursor to the Reaction Volume value (in Figure 49 this shows as "5mL").**
2. **Use the VALUE SETTING buttons (+ or –) to increase or decrease the reaction volume to display 4 mL.**

➤NOTE

If an asterisk appears next to the reaction volume, then that volume is different from the volume entered in the Set Temperature menu of the Reactor.

REACTOR SIDE

1. **Press the FORWARD button (→) once to move the flashing cursor to the Side value.**
2. **Use the VALUE SETTING buttons (+ or –) to change the Side value to A.**

DRAINING METHOD

There are two draining methods. **Pul** (pulsed) draining causes the gas pressure used to drain the reaction vessels to be pulsed to overcome minor flow restrictions. The gas is not pulsed when **Drn** (drain) is selected. Pulsed draining is the default setting and the recommended draining method.

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1. **Press the FORWARD button (→) once to move the flashing cursor to the Draining Method value.**
2. **Use the VALUE SETTING buttons (+ or –) to set the Drain Method value to Pul.**

VARIABLES

Press the **FORWARD button (→)** to display the first line of the Automated Solvent Wash program. The values are displayed as shown in Figure 50:

```
Pgm: 1   5ml Side:A&B
#   R S Add   Mix   Pul
1) 3xA   60   5:00   25
```

Figure 50: Auto Solvent Wash Menu with First Line Displayed

The values on line **1)** are:

R The number of Repetitive washes for the selected solvent. The default value is 3, which represents three repetitions of this line of the program.

S Solvent position. The default value is position A.

Add Solvent delivery time in seconds. The default value is 60 (60 seconds).

Mix Agitator time in minutes and seconds. The default value is 5:00 (5 minutes and 0 seconds).

Pul Pulsed draining time in seconds. The default value is 25 (25 seconds). If **Drn** is displayed rather than **Pul**, the default value is 50.

➤ NOTE

If you want to wash with two solvents simultaneously, set the Drain time to zero (0) for the solvent that is delivered first of the two, and be sure to program the second solvent on the next following program line. Also remember to reduce the quantity of each solvent.

1. **Enter the values listed in line 1 of Figure 51 into the first line of the program. Use the FORWARD button (→) to align the cursor over the**

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value to be changed and press the VALUE SETTING (+ or –) buttons to increase or decrease the value.

#	R	S	Add	Mix	Pul
1	3	A	*	5	15
2	3	B	*	5	15
3	3	C	*	5	15
4	3	D	*	5	15

Figure 51: Automated Solvent Wash Program - Lines 1-4

* For the **Add** value, use the delivery times of the respective solvents as measured in the section "Delivery Times" on page 5-56.

2. **Press the FORWARD button (→) once to add a second line to the Automated Solvent Wash Program. The software creates a copy of the first line and enters it as the next line of the program.**
3. **Enter the values listed in line 2 of Figure 51 into the second line of the program.**
4. **Press the FORWARD button (→) once to add a third line to the Automated Solvent Wash Program. The software creates a copy of the second line and enters it as the next line of the program.**
5. **Enter the values listed in line 3 of Figure 51 into the third line of the program.**
6. **Press the FORWARD button (→) to add a fourth line to the end of the Automated Solvent Wash Program.**
7. **Enter the values listed in line 4 of Figure 51 into the fourth line of the program. The parenthesis ")" indicates that this is the last line of the program.**
8. **Press the MODE button two times to reach the Other Functions menu.**

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MODE Button Add and delete lines from the program by pressing the MODE button, which displays the menu shown in Figure 52.

```
Press MODE to Exit
< to Resume
> to Add New Line
+ to Insert - Delete
```

Figure 52: Pressing MODE to Exit Menu

The following functions are available on the Press MODE to Exit menu:

- Press the MODE button to exit and return to the Other Functions menu.
- Press the BACK (←) button to return to the previous location on the Auto Solvent Wash menu.
- Press the FORWARD button (→) to add a new line to the end of the program.
- Press the VALUE SETTING plus button (+) to insert a new line before the current line in the program.
- Press the VALUE SETTING minus button (–) to delete the current line in the program.

WASH TIMES

The Wash Time value menu provides the control over the solvent delivery line purge time and the pulse duration for pulsed draining.

1. **Press the MODE button until the OTHER FUNCTIONS menu is displayed.**
2. **Press the VALUE SETTING minus button (–) to move the cursor to the Wash Time Params selection.**
3. **Press the MODE button to display the WASH TIMING PARAMS menu (Figure 53).**

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```
--WASH TIMING PARAMS-
Purge---------- 12.5
DrnPulse Open---  2.0
DrnPulse Closed-  2.0
```

Figure 53: Wash Time Params Menu

4. **Use the FORWARD button (→) to move the cursor over the Purge (Time) value.**
5. **Use the VALUE SETTING buttons (+ or –) to increase or decrease the purge time. The default value is 12.5 seconds.**
6. **Use the FORWARD button (→) to move the cursor over the DrnPulse Open value.**
7. **Use the VALUE SETTING buttons (+ or –) to increase or decrease the time the Lower Manifold valve is held open. The default value is 2 seconds.**
8. **Use the FORWARD button (→) to move the cursor over the DrnPulse Closed value.**
9. **Use the VALUE SETTING buttons (+ or -) to increase or decrease the time the Lower Manifold valve is held closed. The default value is 2 seconds.**

WASHING

Before starting the program, ensure that the solvent bottles are pressurized:

1. **Turn the Bottle Cap Assembly valves toward the PRESSURE tubing.**
2. **Allow the bottles to stand for 10 minutes to pressurize.**

➤ NOTE

If the solvent bottles have been stored in a pressurized state, depressurize and sparge them to avoid uneven solvent delivery before re-pressurizing. See the section "Solvent Sparging" on page 8-103.

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Ensure that the Control Valves on Side B of the Reactor are both turned to CLOSED and that the **Upper Manifold Membrane** Valve is set to CLOSED.

1. **Rotate the Reactor to Side A.**
2. **Set the Bubbler valve to SOLVENT DELIVERY, set the Vent valve to Autowash, and set the Upper Manifold Membrane Valve to OPEN RVs.**
3. **Place the waste reservoir below the Lower Manifold.**
4. **Open the Lower Manifold Drain Valves. If there is liquid in the reaction vessels, drain them before starting the Automated Solvent Wash program.**

WARNING

Reaction Vessel draining should always be verified before running the Automated Solvent Wash program, by manually filling the RVs with solvent and then draining.

WARNING

Ensure that the solvent delivery times are accurate before operating the Automated Solvent Wash Option. Inappropriate solvent delivery times can cause overfilling of the RVs and create cross contamination between RVs, clogging of the upper manifold, and solvent overflow into the ASW.

5. **Press the MODE button until the Agitator menu is displayed.**
6. **Enter the Agitator values to be used. When the program is started, the only means of adjusting the speed of the agitator is by using the Mix Speed valve.**
7. **Press the MODE button three times to display the Auto Solvent Wash menu. A flashing cursor is aligned to the right of Pgm: (program).**
8. **Use the VALUE SETTING buttons (+ or –) to increase or decrease the program number until Pgm 1 is displayed.**
9. **Press the AGITATOR ON button to start the Automated Solvent Wash program. The program begins at line 1.**

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To start the Automated Solvent Wash program on a line other than line 1, use the FORWARD or BACK buttons (← →) to align the cursor over the desired line number. Press the AGITATOR ON button to display the menu shown in Figure 54.

```
Press : To Start At:
Mode  : Current Line
'+'   : Top of Pgm
'<'   : Don't Start
```

Figure 54: To Start At Menu

The following functions are available on the To Start At Menu.

- Press the MODE button to start at the Current Line.
- Press the VALUE SETTING plus button (+) to start at the top of the program.
- Press the BACK (←) button to return to the previous location on the Auto Solvent Wash menu.

MONITORING THE WASH

The Automated Solvent Wash program has several features that make it simple to edit a running program, to identify where the program is at any particular time, and to know how much longer the program has to run before it is complete.

When the ASW program is running, the flashing cursor aligns itself over the active value and counts down the remaining time. The one exception is during purging, when the flashing cursor remains in the Add location. The status of the program is displayed in the upper right corner of the display. Examples of screens displayed when the ASW program is running are shown in Figure 55.

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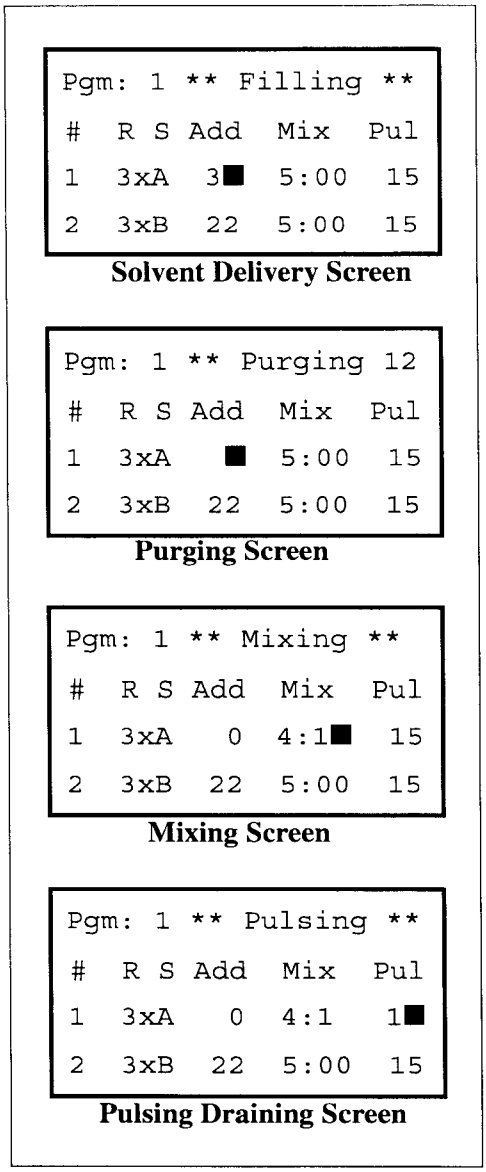


Figure 55: Four Screens Displayed During Operation

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EDITING ACTIVE VALUES

This section describes the functions that are available while an Auto Solvent Wash program is running.

Only the active value or the iterations of that particular program line may be edited while a program is running. The active value is indicated by the position of the flashing cursor. The value of the active value is increased or decreased by pressing the VALUE SETTING buttons (+ or –). The following examples with Figure 56 show how to do this.

Increase Delivery Time To increase delivery time for solvent B in the second line of program 1, press the VALUE SETTING plus button (+) when the cursor is over the Add value. A countdown timer is now displayed in the upper right corner while the number at the cursor position reflects the newly edited value.

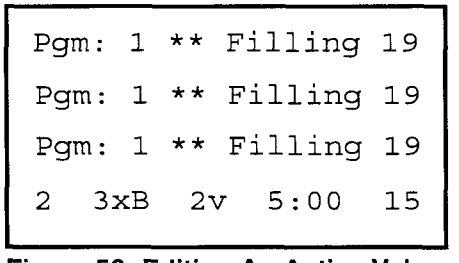


Figure 56: Editing An Active Value

Edit Line Repeats The Repeat (R) value shows the number of line repeats remaining to be executed. Press the BACK button (←) to scroll over to the Repeat value. Use the VALUE SETTING buttons (+ or -) to increase or decrease the number. Any alterations will be added to the total repeats value the next time the program runs.

Pause the Program An ASW program that is running can be paused by pressing either the AGITATOR ON/OFF or MODE buttons. The Program Pause Menu is displayed (Figure 57).

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```
-Wash Program Paused
Press MODE to STOP
< to Resume
> to Skip      + Hold
```

Figure 57: Program Pause Menu

NOTE

The Hold function is not available during solvent deliveries. "Hold" will not appear on the Program Pause menu if the program is paused at that time.

The following functions are available while the program is paused.

Stop (Abort) the Program You can stop the program and return the instrument to manual control by pressing the MODE button. If you stop the program during a delivery, a purge will be performed before the program is aborted.

NOTE

Be sure to manually close the Lower Manifold Drain Valves before aborting an Automated Solvent Wash Program during pulsing (or draining).

Resume the Program You can resume the program at the point at which it was paused by pressing the BACK button (←).

Hold the Program You can place the program on hold by pressing the VALUE SETTING plus button (+). The current process will continue indefinitely until the hold is released. You can release the hold, by re-entering the Program Pause menu and pressing the VALUE SETTING plus button (+). The Hold function is not available for solvent deliveries.

Skip a Program Value You can skip a value by pressing the FORWARD button (→) to jump to the next value in the program.

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For example, pressing the FORWARD button (→) during a solvent fill jumps the program to purging. Pressing the FORWARD button (→) again will jump the program to Start Mixing.

The Skip feature is always available. You do not need to access the Program Pause menu.

COMPLETION

When the Auto Solvent Wash program is completed:

- The screen displays the message "Auto Solvent Wash Run Finished".
- The buzzer sounds.
- The ASW Delivery valves close.
- The Lower Manifold Drain Valves open.
- The Upper Manifold is vented.

If the Reactor is not to be used again for several hours, we recommend that the solvent bottles in the ASW be depressurized so that the solvents do not become saturated with gas. A solvent saturated with gas at 9 psig (1.6 bar) may out-gas during a delivery, causing uneven RV filling. Storing solvents in depressurized bottles results in significantly reduced gas absorption and related uneven RV filling.

Depressurize the solvent bottles as follows:

1. **Turn the valves on the top of the Bottle Cap Assemblies towards VENT.**
2. **Allow the gas pressure to release.**
3. **Turn the valve to the closed position. The closed position is defined as any valve position other than towards the PRESSURE or VENT.**

If the solvent bottles have been stored in a pressurized state, you should sparge them before use, as described in the next section.

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SOLVENT SPARGING

Sparging a solvent requires forcing inert gas at 9 psig (1.6 bar) back into the bottle to out-gas the solvent. Perform the following steps to sparge a solvent.

1. **On the solvent bottle to be sparged, turn the valve on the Bottle Cap Assembly toward VENT.**
2. **On the Reactor, place both Upper Manifold Membrane Valves to the SEAL RVs position.**
3. **Open the toggle valve on ASW that corresponds to bottle position to be sparged (A, B, C, or D).**
4. **Open the PURGE toggle valve. Bubbles should be visible inside the sparging solvent bottle and coming out of the solvent pickup tube. Allow the sparging to continue for five minutes.**
5. **Return the PURGE and bottle position toggle valves to the OFF position.**
6. **On the Reactor, return both Upper Manifold Membrane Valves to the OPEN RVs position.**
7. **Return the valve on the Bottle Cap Assembly to the PRESSURE position to re-pressurize the sparged solvent.**

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9

CLEANING & MAINTENANCE

This chapter describes the steps required to clean the Quest 210 manually. Chapter 8 describes the procedure when the Automated Solvent Wash Option is installed.

Immediately after each synthesis, flush the Upper Manifold, all delivery lines, Luer Plugs, and the Lower Manifold with a solvent compatible with the synthesis. This will ensure that no hardened residue remains to contaminate the next synthesis. Dispose of the solvent waste in accordance with local laws and regulations.

1. **Place the empty Waste Reservoir under the Reactor. Move the tray to its upper most position using the Waste Tray Handles. The silver buttons will snap out to verify that the Waste Reservoir is locked into position.**
2. **Perform three solvent washes (solvent delivery, agitation, drain) on the reaction vessel bank(s) used for the synthesis. Select a solvent that will dissolve any solids or other residues generated during the synthesis.**
3. **Fill the vessels so they are about ¾ full, mix for one minute, and drain the solvent. Depending on the type of chemistry used, it may be desirable to flush the Reactor with a series of solvents. If a series of solvents is used, do at least three washes with each solvent. It is recommended that a volatile solvent, such as acetone, be flushed through the system last.**
4. **After the last solvent wash, let the system drain for a couple of minutes to flush residual solvent out of the Upper Manifold.**
5. **Be sure to vent the Upper Manifold by turning the Control Valves to CLOSED and VENT.**
6. **Remove each Luer Plug and clean with the same solvent as in step 2.**
7. **Rinse out the Luer Ports with an appropriate solvent and wipe clean with a cotton swab wetted with the same solvent as in step 2.**
8. **Replace the Luer Plugs.**
9. **Flush the Upper Manifold to waste using the following protocol:**
   - Open the Lower Manifold Drain Valves.

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- Turn the Control Valves to HIGH PRESSURE GAS and CLOSED and allow the delivery pathway to purge for about 1 minute.
- With the drain valves open, turn the Delivery valve to CLOSED and the Vent valve to LOW PRESSURE GAS. This will purge the other common delivery path of the Upper Manifold. Allow the system to purge for about 1minute.

10. **Vent the Upper Manifold by turning the Control Valves to CLOSED and VENT.**
11. **Close the Upper Manifold by turning each of the Control Valves to CLOSED.**
12. **Clean the Lower Manifold with a wash bottle containing either acetone or MeOH. If necessary use dichloromethane (DCM).**

⚠ CAUTION

The Lower Manifold drain path is not direct. DO NOT INSERT needles or other objects into the Lower Manifold. (See Figure 58 on page 9-107). Obstructions may be removed by connecting a syringe and backflushing with solvent.

13. **Close the Lower Manifold Drain Valves.**
14. **Raise and swing out the Upper Manifold and use the pliers provided to remove the reaction vessels.**
15. **Remove and clean the agitator magnets from the reaction vessels. Discard the reaction vessels and resin.**
16. **Examine the male fittings that hold the reaction vessels in place on the Upper Manifold and the Lower Drain Manifold. Wipe clean with a cotton swab or soft cloth wetted with the same solvent as in step 2. Do not allow any particulates to enter the drain ports of the Lower Manifold male fittings.**
17. **Empty the Waste Reservoir and rinse clean. Do not leave the reservoir full of waste liquid when the system is not in use. Examine the plastic plate located above the Waste Reservoir. Wipe clean of any remaining residue.**

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WARNING

Dispose of all hazardous waste in accordance with all local laws and regulations.

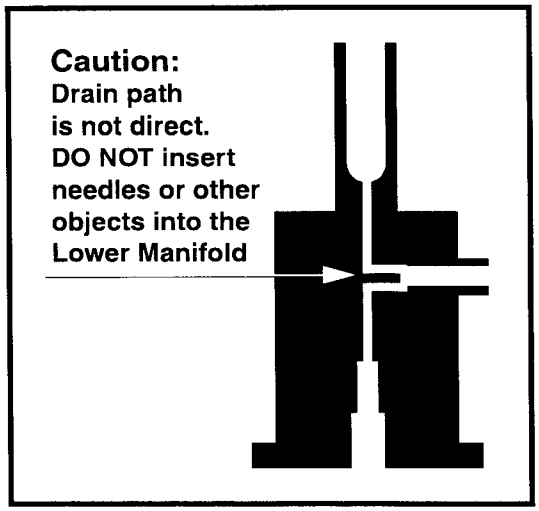


Figure 58: Drain Path of Lower Manifold

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10

TUTORIAL

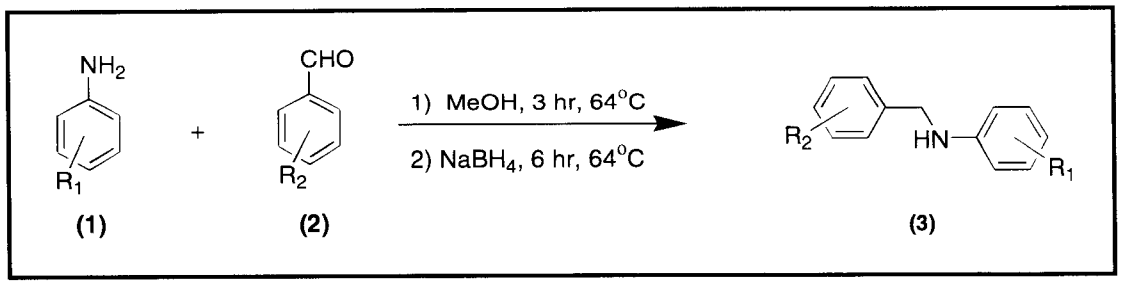


Figure 59: Reductive Amination to form Corresponding Secondary Amine

OVERVIEW

The objective of this Quest 210 Organic Synthesizer User Tutorial is to introduce users to the instrument by performing a parallel solution phase reaction, work-up, and purification. This synthesis is a reductive amination to form the corresponding secondary amine, and the reaction scheme is shown in Figure 59. Equipment required is listed in Figure 60, and the reagents required for the synthesis and purification are listed in Figure 61.

The tutorial is divided into the following sections:

- **Preparation**
- **Reductive Amination**
- **Product Work-Up and Collection**
- **Cleaning and Maintenance**
- **Product Analysis**

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SUMMARY OF THE PROCEDURE

NOTE

This is only a summary. When carrying out the synthesis, follow the detailed procedure beginning on page 10-111.

This synthesis is performed on a single bank of ten 10 mL reaction vessels (RVs) on the Reactor.

Prior to starting the synthesis, thoroughly clean the instrument and install new RVs. Rinse the RVs with methanol (MeOH).

Add the R_1-aniline (0.5 M in MeOH) and R_2-aldehyde (0.5 M in MeOH) solutions to the corresponding RVs, and mix for three hours at 64°C under reflux conditions.

Cool the reaction mixture to room temperature and add the reducing agent (0.5 M $NaBH_4$/EtOH) to each RV in two 1 mL aliquots over a thirty minute period. After the evolution of gas ceases, reflux the reaction for six hours at 64°C.

Work up the reaction mixtures with de-ionized water (diH_2O) and CH_2Cl_2.

Dry and purify the organic layer: *Either...*

in parallel on-line using a Quest SPE Rack and solid phase extraction (SPE) cartridges packed with neutral alumina containing $MgSO_4$. Drain the organic layer directly into the SPE cartridges and then into tared scintillation vials for product collection.

Or...

drain the organic layer directly into scintillation vials and dry the samples with $MgSO_4$. Decant the dried solution into neutral aluminum oxide SPE cartridges and then drain into tared scintillation vials.

Concentrate the products using a rotary evaporator or concentration unit and calculate the mass yields. Reconstitute the samples in 1 mL of CH_2Cl_2, and analyze by HPLC and NMR analysis.

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PREPARATION

- Read this Quest 210 User Manual.
- Verify that the Reactor was installed according to the Installation Procedure in Chapter 4, "Quest 210 Installation."l.
- If your Quest is not fitted with the Automated Solvent Wash (ASW) Option, verify that the inert gas input pressure is between 55-60 psig (4.8-5.2 bar).
- If your system is using both compressed air and inert gas, verify that Controller agitator gas (e.g., compressed air) input pressure is 55-60 psig (4.8-5.2 bar) and the inert gas input pressure is 40-60 psig (3.8-5.2 bar).
- Familiarize yourself with the nomenclature and various components of the Reactor.
- Install the Bubbler. This allows you to visualize the gas flow rate on the Reactor. Follow the installation procedure provided with the Bubbler.
- Install the RVs as described in Chapter 6.


WARNING

Always wear gloves, eye protection, and proper clothing when working with the Quest 210.

EQUIPMENT REQUIREMENTS

In addition to the Quest 210 synthesizer, the following equipment is required:

Item	*Source*	*Comment*	*Total amount used*
10 mL Reaction Vessels	Argonaut	P/N 900087	10
Bubbler	Argonaut	P/N 900125	1
Scintillation Vials	VWR		20
15 mL Filtration Columns	Jones Chromatography	120-1025-D	10
SPE Rack (Optional)	Argonaut	P/N 900182	1

Figure 60: Equipment Required

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REAGENT REQUIREMENTS

The reagent requirements are listed in Figure 61 below and their structures are illustrated in Figure 62.

NOTE

The numbers in parentheses refer to the structures shown in Figure 62.

Material	*Source*	*Comment*	*Total amount used*
Aniline (4)	Aldrich	FW=93.13 d=1.022	501.2 µl (in 11 mL MeOH)
3,5-dimethylaniline (5)	Aldrich	FW=121.18 d=0.972	685.7 µl (in 11 mL MeOH)
Benzaldehyde (6)	Aldrich	FW=106.12 d=1.044	254.1 µl (in 5 mL MeOH)
p-Anisaldehyde (7)	Aldrich	FW=136.15 d=1.119	304.2 µl (in 5 mL MeOH)
p-Tolualdehyde (8)	Aldrich	FW=120.15 d=1.019	294.8 µl (in 5 mL MeOH)
Salicylaldehyde (9)	Aldrich	FW=122.12 d=1.146	266.4 µl (in 5 mL MeOH)
m-Tolualdehyde (10)	Aldrich	FW=120.15 d=1.019	294.8 µl (in 5 mL MeOH)
Sodium Borohydride	Aldrich	FW=37.83	198.6 mg (2x 10.5 mL EtOH)
Methanol	Aldrich	FW=32.04	50 mL
Ethanol	Aldrich	FW=46.07	25 mL
di H_20		FW=20.03	60 mL
Dichloromethane	Aldrich	FW=84.93	240 mL
Aluminum Oxide, activated, neutral	Aldrich		20 g total or 2 g/cartridge
Magnesium Sulfate	Aldrich	FW=120.37	10 g total or 1 g/cartridge

Figure 61: Reagents Required

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REAGENT PREPARATION

Prepare the following stock solutions as described in Figure 61.

- R_1-substituted aniline solutions in MeOH (0.5 M)
- R_2-substituted benzaldehyde solutions in MeOH (0.5 M)
- No more than five minutes before addition, two 10 mL stock solutions of $NaBH_4$ solution in EtOH (0.5 M)

⚠ CAUTION

It is critical that the $NaBH_4$ solution added to each RV is freshly made.

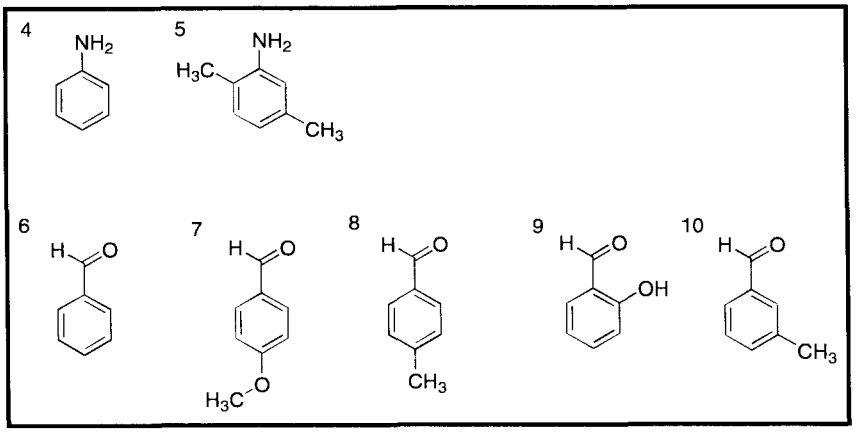


Figure 62: Structures of Aniline and Benzaldehyde Reagents

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REDUCTIVE AMINATION

ADDING REAGENT

Before adding reagent, fill the dead volume between the bottom of the RV frit and the Lower Manifold Drain Valve with solvent. This is necessary to eliminate air from the system which might otherwise affect the synthesis. The procedure is described on page 7-72.

NOTE

The numbers in parentheses refer to the reagent list in Figure 61.

RV	R_1-C_6H_4-NH_2	R_2-C_6H_4-CHO
1	Aniline (4)	Benzaldehyde (6)
2	Aniline (4)	p-Anisaldehyde (7)
3	Aniline (4)	p-Tolualdehyde (8)
4	Aniline (4)	Salicylaldehyde (9)
5	Aniline (4)	m-Tolualdehyde (10)
6	3,5-dimethylaniline (5)	Benzaldehyde (6)
7	3,5-dimethylaniline (5)	p-Anisaldehyde (7)
8	3,5-dimethylaniline (5)	p-Tolualdehyde (8)
9	3,5-dimethylaniline (5)	Salicylaldehyde (9)
10	3,5-dimethylaniline (5)	m-Tolualdehyde (10)

Figure 63: Reagent Addition Scheme

1. **To RVs 1 through 5, add 2 mL of aniline/MeOH via syringe.**
2. **To RVs 6 though 10, add 2 mL of 3,5-dimethylaniline/MeOH via syringe.**
3. **Add 2 mL of the appropriate 0.5 M R2-substituted benzaldehydes/MeOH to the corresponding RVs given in Figure 63. in the same manner.**
4. **Into each RV, snugly insert and twist the Luer Plug to ensure a tight seal.**

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5. **Close the Upper Manifold Membrane by toggling the switch to SEAL RVs. Rotate both Control Valves to CLOSED.**

⚠ CAUTION

While refluxing it is vital to seal the reaction vessels using the above procedure or solvent loss will occur.

6. **Using a 7/64 inch hex wrench, lower or raise the agitator stops so that the magnets in the RVs are approximately 1 cm below the solvent level.**
7. **Program the agitator values by pressing the MODE button on the front panel of the Controller until the LCD displays the AGITATOR menu. Enter the following agitator values:**

MixEvery:	2.0 sec
UpStroke:	1.0 sec
% Upwards:	50%

To enter the MIXEVERY and % UPWARDS values, move the cursor to the desired position by using the FORWARD and BACK buttons (← →) below the LCD screen. To change the value settings, use the VALUE SETTING (+ and –) buttons to the left of the LCD screen.

8. **Start the agitator by pressing the AGITATOR ON/OFF button on the Controller.**
9. **Program the heater values by pressing the MODE button until the LCD displays the SET TEMPERATURE menu. Enter the following values for Bank A:**

A:	64°C	3:00	OFF
B:	100°C	0:00	OFF
RV Size:	A: 5 mL	B: 5 mL	

The heating duration and temperature can be adjusted by moving the cursor with the FORWARD and BACK buttons (← →) below the LCD screen. To change the heating values, use the VALUE SETTING (+ and –) buttons to the left of the LCD screen.

NOTE

For accurate measurement of the RV temperature, advance the cursor to the RV Size value and enter the volume of solvent that is in the RVs. The firmware accepts volumes from 3 to 12 mL in 1 mL increments.

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10. Move the cursor to the line for Bank A and press the HEAT ON/OFF button. This will only turn on the heater for bank A.
11. Monitor the reaction temperature by pressing the MODE button until the TEMPERATURE MONITOR screen is shown. The set and actual temperature of each reaction vessel bank, heater status, and remaining time in the heater program will be displayed. With the current values, the screen should show the following:

A: (ON)	B: (OFF)
Set 64°C	100°C
Act 45°C	33°C
3:00:00	0:00:00

The timer for the heating program will not start until the RV temperature reaches the set temperature. When the programed temperature is achieved and the timer is counting down, the temperature can be adjusted by going back to the Set Temperature menu and adjusting the temperature and time values.

12. Reflux the reaction for three hours at 64 °C. The heater will turn off when the heater program is complete.
13. Allow the reaction bank to cool to room temperature.

ADDING SODIUM BOROHYDRIDE

1. Open the Upper Manifold Membrane by toggling the switch to OPEN RVs.
2. Remove the Luer Plug from each RV, and slowly add 1 mL of freshly prepared 0.5 M sodium borohydride/EtOH solution.
3. Snugly insert and twist the Luer Plugs to ensure a good seal.
4. Rotate the Control Valves to BUBBLER and CLOSED to vent the gas evolved during the $NaBH_4$ addition.
5. Adjust the agitator stops so that the magnets in the RVs are approximately 1 cm below the solvent level.
6. Agitate for 30 minutes at room temperature.
7. After 30 minutes, stop the agitator and add the second addition (1 mL) of 0.5 M sodium borohydride/EtOH to the RVs in the same manner as the first.
8. Snugly insert and twist the Luer Plugs to ensure a proper seal.
9. Rotate the Control Valves to BUBBLER and CLOSED to vent the gas evolved during this addition.
10. When gas evolution ceases, toggle the Upper Manifold Membrane Switch to SEAL RVs and rotate both Control Valves to CLOSED.

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⚠ CAUTION

While refluxing it is vital to seal the reaction vessels using the above procedure or solvent loss will occur.

11. Adjust the agitator stops so that the magnets in the RVs are approximately 1 cm below the solvent level and turn the agitator on.

12. Program the heater values by pressing the Controller MODE button until the LCD displays the SET TEMPERATURE menu. Enter the following values for Bank A:

A: 64 C	6:00	OFF
B: 100 C	0:00	OFF
RV Size	A: 5 mL	B: 5 mL

➤ NOTE

For accurate measurement of the RV temperature, advance the cursor to the RV Size value and enter the volume of solvent that is in the RVs. The firmware accepts volumes from 3 to 12 mL in 1 mL increments.

13. When gas evolution has stopped, turn on the heater for Bank A.

14. Reflux the reaction for six hours at 64°C. The heater will turn off after the temperature program is complete.

15. Allow the RVs to cool to room temperature.

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PRODUCT WORK-UP AND COLLECTION

Two alternative methods of solid-phase extraction are described here: On-line using the Quest SPE Rack, and off-line.

ON-LINE SOLID PHASE EXTRACTION

The samples are worked up directly in the reaction vessels (using the Quest SPE Rack) with di H_20 and extracted with CH_2Cl_2 three times as follows:

1. **To each of ten 15 mL SPE cartridges, add 2 g of neutral aluminum oxide, followed by 1 g of $MgSO_4$.**
2. **Insert a small amount of glass wool to secure the matrix. install the packed cartridges onto the SPE Rack in positions 1-10 and close the polypropylene valves.**
3. **Lower the Waste Tray to the lowest setting and remove the Waste Reservoir. Place the SPE Rack with cartridges onto the Waste Tray. Push in the buttons on the Waste Tray Handles and raise the tray to its uppermost position so that the Teflon drain lines from the Lower Manifold are inside the cartridges. If the Waste Tray will not lock into place, adjust the height of the SPE Rack using the adjustable legs.**
4. **Add 3 mL of di H_20 and 3 mL of CH_2Cl_2 to each RV via the luer port.**
5. **Agitate the RVs for three cycles and stop the Agitator by pressing the AGITATOR DOWN button.**
6. **Allow the organic and aqueous layers to separate in the RVs, then turn the Low Pressure Gas Valve completely clockwise to close the valve and rotate the Control Valves to CLOSED and VENT.**
7. **Rotate the Control Valves to CLOSED and LOW PRESSURE GAS.**
8. **Open the drain valve for RV1 only, and increase the inert gas flow by slowly turning the Low Pressure Gas Valve counter-clockwise to drain the bottom layer.**
9. **Watch for the end of the phase separation. When the organic layer has drained into the cartridge on the SPE rack, close the drain valve.**
10. **Turn the Low Pressure Gas Valve completely clockwise to close the valve and rotate the Control Valves to CLOSED and VENT.**
11. **Repeat step 7 through 10 for each RV in turn.**
12. **Add an additional 3 mL of CH_2Cl_2 to each RV via the luer port.**
13. **Agitate the RVs for three cycles and stop the Agitator by pressing the AGITATOR DOWN button.**

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14. **When the layers have separated, drain the bottom layer into the cartridges as described in steps 7 through 10 above.**
15. **Repeat steps 7 through 10 two more times with CH_2Cl_2.**
16. **Remove the SPE rack from the Waste Tray and place it directly on top of the Scintillation Vial Rack containing ten tared scintillation vials. Open the polypropylene valves and gravity drain the product into the scintillation vials.**

NOTE

If the SPE cartridges fill to capacity before all the extractions are complete, allow the organic layer to sit for ten minutes. Then remove the SPE rack from the Waste Tray and place it directly on top of the Scintillation Vial Rack containing ten tared scintillation vials. After draining the SPE cartridges into the vials, place the SPE Rack underneath the Reactor to collect the additional extractions.

17. **Concentrate the products to dryness using a rotary evaporator or concentrator. Weigh the scintillation vials and calculate the product mass yields. Reconstitute the samples in 1 mL of CH_2Cl_2, and characterize each sample by HPLC and NMR for product purity.**

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OFF-LINE SOLID PHASE EXTRACTION

The samples are worked up with di H_20 and extracted with CH_2Cl_2 three times as follows:

1. **To each of ten 15 mL SPE cartridges, add 2 g of neutral aluminum oxide followed by 1 g of $MgSO_4$. Insert a small amount of glass wool to secure the matrix.**
2. **Lower the Waste Tray to the lowest position and remove the Waste Reservoir. Place the Scintillation Vial Rack with ten scintillation vials onto the Waste Tray. Push in the buttons on the Waste Tray Handles and raise the tray to its uppermost position.**
3. **To each RV in turn, add via the luer port 3 mL of di H_20 and 3 mL of CH_2Cl_2.**
4. **Agitate the RVs for three cycles and stop the Agitator by pressing the AGITATOR DOWN button. Allow the layers to separate in the RVs.**
5. **Turn the Low Pressure Gas Valve completely clockwise to close the valve and rotate the Control Valves to CLOSED and VENT.**
6. **Rotate the Control Valves to CLOSED and LOW PRESSURE GAS. Open the drain valve for RV1 only and increase the inert gas flow by slowly turning the Low Pressure Gas valve counterclockwise to drain the bottom layer.**
7. **Watch for the end of the phase separation. When the organic layer has drained into the scintillation vial, close the drain valve.**
8. **Turn the Low Pressure Gas Valve completely clockwise to close the valve and rotate the Control Valves to CLOSED and VENT.**
9. **Repeat steps 6 through 8 for each RV.**
10. **Add 3 mL of CH_2Cl_2 to each RV via the luer port.**
11. **Agitate the RVs for three cycles and stop the Agitator by pressing the AGITATOR DOWN button.**
12. **When the layers have separated, drain the bottom layer into the scintillation vials as described in steps 5 and 6 above.**
13. **Repeat steps 10 through 12 two more times with CH_2Cl_2.**
14. **Remove the Scintillation Vial Rack from the Waste Tray and add $MgSO_4$ to the collected organic layer in the scintillation vial. Dry the organic layer for ten minutes.**
15. **Insert ten tared scintillation vials in the second row of the Scintillation Vial Rack and place an alumina SPE cartridge (prepared in step 1 above) on top of each vial**

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16. **Decant the dried organic layers into the cartridges, and allow each cartridge to gravity drain directly into the corresponding tared scintillation vial.**

17. **Concentrate the products to dryness using a rotary evaporator or concentrator. Weigh the scintillation vials and calculate the product mass yields. Reconstitute the samples in 1 mL of CH_2Cl_2, and characterize each sample by HPLC and/or NMR for product purity.**

CLEANING AND MAINTENANCE

After each synthesis the Reactor should be thoroughly cleaned to avoid contaminating the next reaction. This is achieved by flushing the Reactor liquid delivery system and cleaning the Upper Manifold Luers and Ports to remove residual solvent or reagent from the previous synthesis.

Two procedures are described here; the first for systems that do not have the automated solvent wash module, and the second for those that do.

NOTE

Routinely cleaning the Reactor after each synthesis will ensure that the system is fully functional prior to each synthesis.

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MANUAL CLEANING

1. **After completing the synthesis, rinse the RVs bank by delivering the last solvent used for the reaction until the RV's are approximately 30% full.**
2. **Agitate for approximately 5 minutes and then drain the RVs. Repeat this rinse three times.**
3. **After the last drain, leave the Lower Manifold Drain Valves open with the High Pressure Gas on for 10 minutes to allow the RVs to drain thoroughly and to remove any residual solvent.**
4. **Remove the Luer Plugs and clean them with acetone to remove any particulates.**
5. **Rinse the Luer Ports with acetone and then gently wipe them clean with a cotton swab wetted with acetone to remove any residue from the reaction.**
6. **Replace the Luer Plugs and dry the RVs by rotating the control valves to HIGH PRESSURE GAS and CLOSED. Allow the RVs to dry thoroughly to remove any residual solvent.**
7. **Turn both Control Valves to CLOSED.**
8. **Flush the Upper Manifold by turning the Control Valves to CLOSED and LOW PRESSURE GAS. After 10 minutes turn the Control Valves to CLOSED and CLOSED.**

➤ NOTE

If the Reactor is not going to be used immediately, leave the RVs on the Reactor. If the Reactor is going to be used immediately, continue with the RV removal and installation procedure.

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CLEANING WITH THE AUTOMATED SOLVENT WASH MODULE

(When fitted)

1. **After completing the synthesis, program the Automated Solvent Wash (ASW) module to rinse the reaction bank by delivering the last solvent used for the reaction until the RV's are approximately 30% full.**
2. **Agitate for approximately 5 minutes and then drain the RVs. Repeat this rinse three times. Leave the Lower Manifold Drain Valves open for all RVs.**
3. **Use the following ASW program to rinse the reaction bank:**

R S	ADD	MIX	PUL
3xD	78*	5:00	20

D=Dichloromethane

*Actual fill time may be different from the above.

4. **After the last drain, leave the Lower Manifold Drain Valves open with the High Pressure Gas on for 10 minutes to allow the RVs to drain thoroughly and to remove any residual solvent.**
5. **Remove the Luer Plugs and clean them with acetone to remove any particulates. Rinse the Luer Ports with acetone and then gently wipe them clean with a cotton swab wetted with acetone to remove any residue from the reaction.**
6. **Replace the Luer Plugs and dry the RVs by rotating the Control Valves to HIGH PRESSURE GAS and CLOSED. Allow the RVs to dry thoroughly to remove any residual solvent.**
7. **Turn both Control Valves to CLOSED.**
8. **Flush the Upper Manifold by turning the Control Valves to CLOSED and LOW PRESSURE GAS. After 10 minutes turn both Control Valves to CLOSED.**

NOTE

If the Reactor is not going to be used immediately, leave the RVs on the Reactor. If the Reactor is going to be used immediately, continue with the RV removal and installation procedure. For detailed instructions on RV removal and installation, refer to "Removing Reaction Vessels" on page 6-60 and "Installing Reaction Vessels" on page 6-63.

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PRODUCT ANALYSIS

This section gives the results of the solution-phase reductive amination experiment carried out as described above. These results are representative and intended solely for purposes of illustration. Actual results may vary according to the purity of the reagents used for the synthesis and other factors.

TABULATED RESULTS AND STRUCTURES

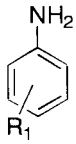
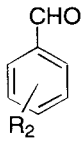
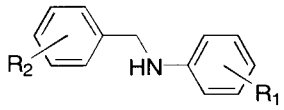
RV	 (1)	 (2)	 (3)	Yield (%)	HPLC Purity (%)
1	4	6	12	>99	100%
2	4	7	13	82.0	76%
3	4	8	14	77.1	98%
4	4	9	15	79.9	89%
5	4	10	16	82.9	95%
6	5	6	17	77.0	94%
7	5	7	18	84.2	79%
8	5	8	19	88.1	95%
9	5	9	20	89.7	81%
10	5	10	21	80.0	97%

Figure 64: Results of Solution Phase Reductive Amination

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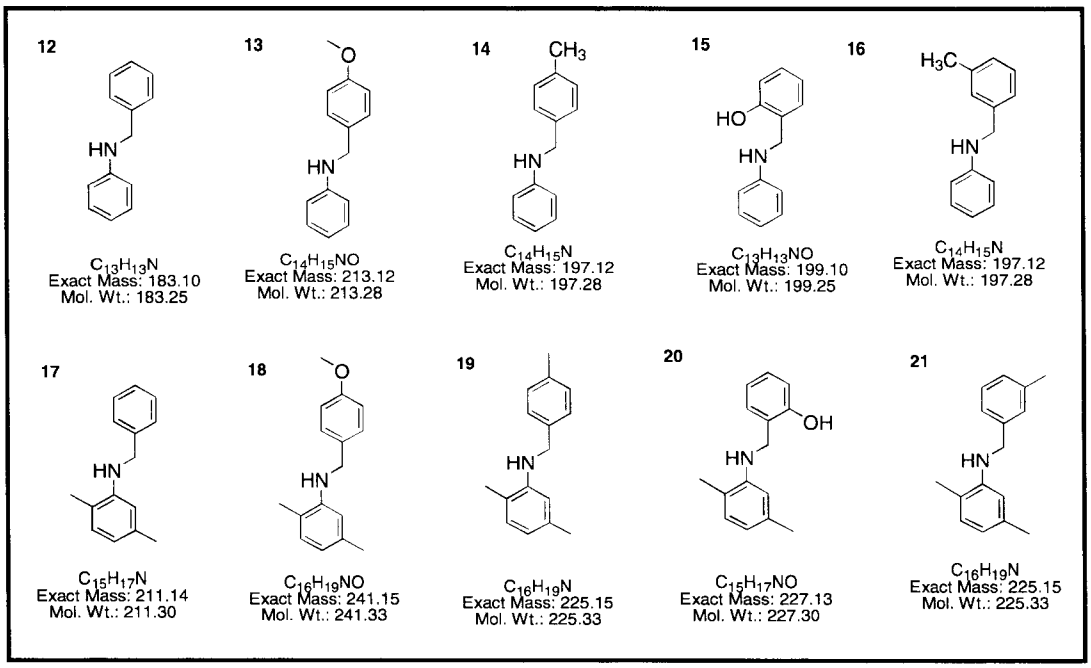


Figure 65: Structures of Amine Products

SPECTROSCOPIC DATA

Figure 64 illustrates the percent yield and HPLC purities for each amine product. Purities were determined by high-pressure liquid chromatography (area %) using a 3 mm C18 column (Microsorb-MV, 100 Å). The HPLC method used was a 2-90% ACN/H_20 with 0.1% TFA run over 9 minutes for quantitative analysis of reaction products, UV detection at 223 nm. HPLC retention time and ^1H NMR of representative products are provided below:

Product 12: HPLC retention time: 4.48 min.^1H NMR ($CDCl_3$, 300 MHz):δ 7.40-7.16, 6.75-6.63 (m, 10 H, Ar-H), 4.34 (s, 2 H, CH_2), 4.03 (br s, 1 H, NH) ppm.

Product 13: HPLC retention time: 4.47 min.^1H NMR ($CDCl_3$, 300 MHz): δ 7.38-7.14, 6.88-6.86, 6.74-6.63 (m, 9 H, Ar-H), 4.25 (s, 2 H, CH_2), 3.79 (s, 3 H, CH_3), ppm.

Product 14: HPLC retention time: 4.74 min. ^1H NMR ($CDCl_3$, 300 MHz): δ 7.29-7.11, 6.75-6.63 (m, 9 H, Ar-H), 4.29 (s, 2 H, CH_2), 3.98 (br s, 1 H, NH), 2.36 (s, 3 H, CH_3) ppm.

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Product 15: HPLC retention time: 4.12 min. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.26-7.15, 6.92-6.84 (m, 9 H, Ar-H), 4.42 (s, 2 H, CH2), 3.96 (br s, ^{1}H , NH), 1.55-1.54 (s, 1 H, OH) ppm.

Product 16: HPLC retention time: 4.81 min. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.27-7.09, 6.75-6.64 (m, 9 H, Ar-H), 4.29 (s, 2 H, CH_2), 4.00 (br s, 1 H, NH), 2.37-2.36 (s, 3 H, CH_3) ppm.

Product 17: HPLC retention time: 5.02 min. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.38-7.25, 6.40-6.30 (m, 8 H, Ar-H), 4.31 (s, 2 H, CH_2), 3.89 (br s, 1 H, NH), 2.23 (s, 6 H, 2(-CH_3)) ppm.

Product 18: HPLC retention time: 5.01 min. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.39-7.25, 6.91-6.82, 6.42-6.6.22 (m, 7 H, Ar-H), 4.22 (s, 2 H, CH_2), 3.81(s, 3 H, CH_3), 2.34-2.23 (s, 6 H, 2(-CH_3)) ppm.

Product 19: HPLC retention time: 5.25 min. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.26-7.09, 6.38-6.21 (m, 7 H, Ar-H), 4.24 (s, 2 H, CH_2), 3.83 (br s, 1 H, NH), 2.41-2.22 (s, 6 H, 3(-CH_3)) ppm.

Product 20: HPLC retention time: 4.73 min. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.26-7.13, 6.93-6.83, 6.58-6.49(m, 7 H, Ar-H), 4.38 (s, 2 H, CH_2), 3.81 (br s, 1 H, NH), 2.36-2.19 (s, 6 H, 2(-CH_3)), 1.56 (s, 1 H, OH) ppm.

Product 21: HPLC retention time: 5.27 min. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.26-7.08, 6.39-6.30(m, 7 H, Ar-H), 4.26 (s, 2 H, CH_2), 3.86 (br s, 1 H, NH), 2.43-2.17 (s, 6 H, 3(-CH_3)) ppm.

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APPENDIX A: GLOSSARY

ADJ. LOW PRESSURE GAS FLOW	See **LOW PRESSURE GAS Valve**
Agitator	The pneumatically actuated magnetic Agitator Bar moves up and down between the two banks of reaction vessels. Small Teflon encapsulated magnets placed in the reaction vessels follow the movement of the Agitator Bar causing mixing to occur.
Agitator Valve	See Mix Speed Valve.
Bulkhead Fitting	Female threaded ports for the connection of tubing. These ports can be found on the front panel of the Controller, and on various places of the Reactor.
Collection Racks	Racks that can hold up to 20 test tubes or scintillation vials for product collection.
Control Valves	Each side of the reactor assembly has two Control Valves. These valves control the delivery of gas and solvent to the reaction vessels.
Controller	The unit containing the LCD screen and buttons for controlling agitation and temperature.
Cp	Centipoise, a measure of viscosity.
di H_20	De-ionized water.
Ferrule	The yellow conical shaped Tefzel sleeve used for Teflon tubing connections
Fitting	Tan colored threaded fittings (PEEK) used for Teflon tubing connections.
Frit	Either a 30 µm or 7 µm Teflon filter installed in the bottom of each reaction vessel.

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GRCM	Gaseous Reaction and Concentration Manifold Accessory for the Quest 210 Manual Synthesizer.
Heating Block	Aluminum plate mounted behind the Reaction vessel banks.
High Pressure Gas	30 psig (3.1 bar) gas used to drain the reaction vessels.
Interface Harness	The Interface Harness connects between the Controller and Reactor. It contains the Teflon tubing for gas and solvent delivery and wiring for reaction vessel bank heating and temperature sensing.
Low Pressure Gas	9 psig (1.6 bar) gas used together with the Low Pressure Gas Valve for metered draining and purging of the reaction vessels.
LOW PRESSURE GAS Valve	Used to increase or decrease the flow of 9 psig (1.6 bar) gas into the reaction vessels.
Lower Manifold Drain Valves	Reaction vessels are emptied using the Lower Manifold Drain Valves. Each valve has a lever to open or close the valve.
Luer Port Adaptor	Teflon piece that screws into each luer port at the top of the Upper Manifolds. The luer plug is inserted into the adaptor.
Luer Ports	Bevelled holes with corresponding plugs. Solvents, reagents, solids and gases may be added through the luer ports.
Magnet Bar	Aluminum assembly with cylindrical magnets mounted along its length. The magnet bar is driven up and down, moving Teflon-coated magnets in each of the RVs which mix the contents
Mix Speed Valve	Controls the Agitator Bar speed.
Ports	External accessories are attached to the Reactor through the two utility ports - the Bubbler Port and Autowash Port. Access to each port is controlled by the Control Valves.
Reaction Vessels	Clear Teflon vessels used on the Reactor for compound synthesis. Abbreviated "RV".

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Reactor	The Reactor has two banks of 10 reaction vessels for parallel organic synthesis. Heating, cooling, mixing and solvent additions are performed on the Reactor.
Rinse Solvent	A solvent which is effective in rinsing its associated reagent. Usually the solvent in which the associated reagent is dissolved.
RV	See Reaction Vessel.
Solvent Bottle Assembly	Consists of the Solvent Bottle Cap Assembly and a solvent bottle.
Sparge	1. Bubbling inert gas through the Reaction Vessels for mixing. 2. Pressurizing the solvent bottle with inert gas to remove dissolved air.
Teflon Tubing	The semi-transparent tubing (1/8" OD) used for gas and liquid connections.
Upper Manifold	The Upper Manifold is an aluminum and Teflon block that has two common delivery pathways for gas and solvents: one for parallel solvent delivery drain gas, the other for venting and metered gas.
Upper Manifold Membrane	Teflon membrane valve that seals against the common delivery paths of the Upper Manifold to prevent cross contamination between reaction vessels.
Upper Manifold Membrane Switch	Pressurizes a Teflon membrane which seals against the common pathways of the manifold to prevent crossover of the contents of RVs.
Waste Reservoir	Container for waste collection. The Waste Reservoir has a valve which can be used to drain the reservoir into a suitable external container.
Waste Tray	The Waste Reservoir sits on the Waste Tray. The tray can be lowered using the buttons on its handles for removal or installation of the Waste Reservoir.

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APPENDIX B: CONTROLLER SCREENS

The User interface of the Controller is a liquid crystal display (LCD) (Figure 66). The different screens and menus and an explanation of how to use the Controller buttons is provided in this appendix.

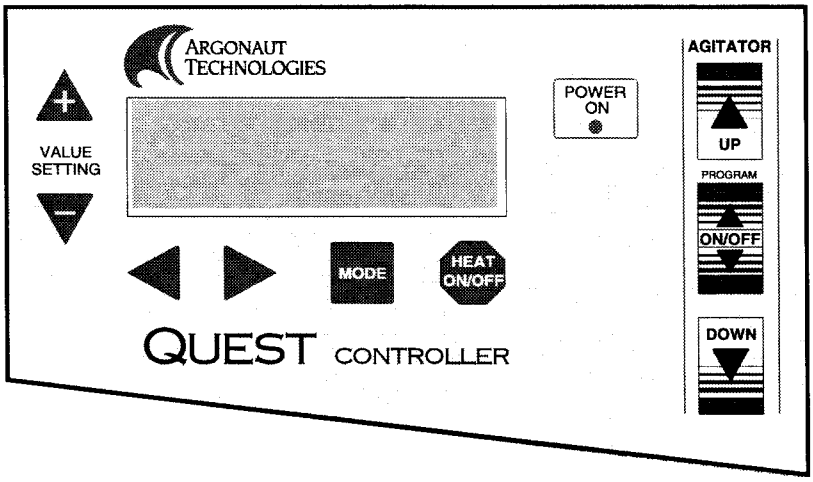


Figure 66: Quest Control Panel and LCD Screen

START-UP SCREEN

Whenever the power is turned on the Controller displays the start-up screen (Figure 67)

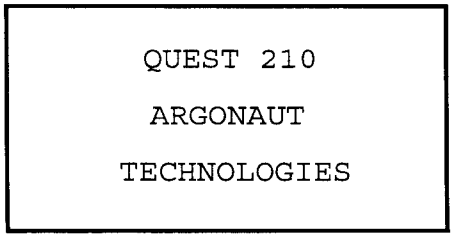


Figure 67: Quest 210 Controller Start-Up Screen

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Figure 68 lists the functions of the buttons on the front panel of the Controller.

Button	Screen
MODE	Displays firmware version and date
← →	N/A
VALUE SETTING	N/A
HEAT ON/OFF	Displays Heaters menu
AGITATOR ON/OFF	Starts or stops the agitator.

Figure 68: Start-Up Screen and Button Functions

FIRMWARE VERSION SCREEN

The Firmware Version screen (Figure 69) shows the Quest 210 software revision and the revision date.

```
-----QUEST 210-----

REV   : 1.1

DATE  : JAN 16,1998
```

Figure 69: Firmware Version Screen

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MENUS

AGITATOR VALUES

The Agitator Value Menu (Figure 70) allows the user to program the agitator values.

```
MixEvery   :    4.0 sec
UpStroke    :    3.8 sec
% Upward   :      95 %

- - Not Agitating - - -
```

Figure 70: Agitator Value Menu

Using the buttons the user inputs the following agitator values:

- MIX EVERY: Time between consecutive down strokes of the Agitator Bar (agitator frequency)
- UPSTROKE: Amount of time in seconds that the agitator bar remains at its highest position (the entered time must be less than the MIX EVERY entry)
- % UPWARD: Same as the UPSTROKE value but expressed as a percentage of the MIX EVERY value

Changing the UpStroke value will automatically change the % Upward value to corresponding value. Agitator values apply to both reaction vessel banks A and B.

Figure 71 lists the functions of the buttons on the front panel of the Controller.

Button	Screen
MODE	Displays SET TEMPERATURE Menu
← →	Moves cursor to agitator values
VALUE SETTING	Increases or decreases the value of the selected value
HEAT ON/OFF	Displays Heaters menu
AGITATOR ON/OFF	Starts/Stops agitator.

Figure 71: Agitator Value Menu and Button Functions

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SET TEMPERATURE

Reaction temperature and heating duration are programmed using the Set Temperature menu (Figure 72). The reaction temperature and heating duration for reaction vessel Bank A and B are programmed independently.

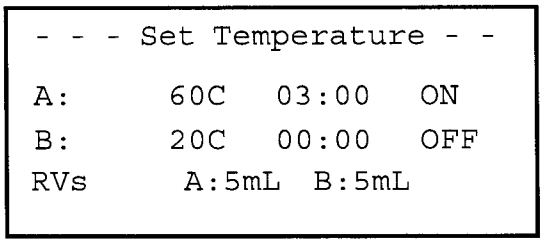


Figure 72: Set Temperature Menu

Figure 73 lists the functions of the buttons on the front panel of the Controller.

Button	Screen
MODE	Displays Monitor Temperature display
← →	Moves cursor to heater temperature values
VALUE SETTING	Increases or decreases the value of the selected value
HEAT ON/OFF	If heaters are off and the cursor is next to A:, pressing ON/OFF turns the heaters on for bank A. If the heaters are on, pressing ON/OFF will turn the heaters off. If heaters are off and the cursor is next to B:, pressing ON/OFF turns the heaters on for bank B. If the heaters are on pressing ON/OFF will turn the heaters off.
AGITATOR ON/OFF	Starts/Stops agitator.

Figure 73: Set Temperature Menu and Button Functions

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TEMPERATURE MONITOR SCREEN

The Temperature Monitor screen (Figure 74) shows the programmed and actual temperature for reaction vessel banks A and B. The screen also shows the heater status (ON or OFF) and remaining program time.

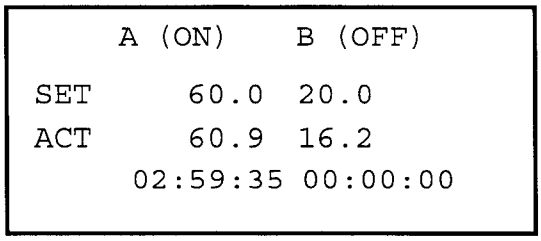


Figure 74: Temperature Monitor Screen

Figure 75 lists the functions of the buttons on the front panel of the Temperature Monitor screen.

Button	Screen
MODE	OTHER FUNCTIONS screen
←	N/A
→	Displays Set Temperature display
VALUE SETTING (+)	Displays Set Temperature menu
VALUE SETTING (–)	N/A
HEAT ON/OFF	Displays Heaters menu
AGITATOR ON/OFF	Starts/Stops agitator.

Figure 75: Temperature Monitor Screen and Button Functions

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OTHER FUNCTIONS MENU

The Other Functions menu allows the User to select from the following:

1. **Proceed to Main**
2. **Adjust LCD: Adjust the LCD contrast**
3. **Show Fmwr Version: Displays firmware version and date**
4. **Perform Self Test: Initiates self test of Controller**
5. **Restore Defaults: Restores default values to menus**

Use the VALUE SETTING buttons (↑ ↓) to move the cursor to the desired menu. Press the MODE button to select the menu. Figure 76 lists the functions of the buttons on the front panel of the Controller.

Button	Screen
MODE	Selects the menu from the Other Functions list
← →	Move cursor up or down to the different menu choices
VALUE SETTING	Moves cursor up or down to the different menu choices
HEAT ON/OFF	Displays Heaters menu
AGITATOR ON/OFF	Starts/Stops agitator.

Figure 76: Other Functions Menu – Button Functions

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SET CONTRAST MENU

The SET CONTRAST menu (Figure 77) allows the User to adjust the Controllers display. Use the VALUE SETTING buttons (↑ ↓) to adjust the screen.

```
      A (ON)    B (OFF)
SET     60.0    20.0
ACT     60.9    16.2
      02:59:35 00:00:00
```

Figure 77: Set Contrast Menu

The table in Figure 78 lists the functions of the buttons on the front panel of the Controller.

Button	Display
MODE	Return to the OTHER FUNCTIONS menu
← →	Increases or decreases the screen contrast
VALUE SETTING	Increases or decreases the screen contrast
HEAT ON/OFF	Displays Heaters menu
AGITATOR ON/OFF	Starts/Stops agitator.

Figure 78: Set Contrast Menu - Button Functions

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SELF-TEST SCREEN

When Perform Self Test is selected from the OTHER FUNCTIONS menu the Controller initiates a self test of the Controller hardware. The following are checked:

- A/D: Analog to digital converters for temperature control
- TC: Thermocouples for reaction vessel bank temperature sensing
- TFMR: Transformer sensor
- HTR: Two reaction vessel bank heaters
- V: Status of the 16 valves

When the test is complete, the Self Test Screen (Figure 79) is displayed. The results for the A/D, thermocouple (TC), transformer (XFMR), heaters (HTR) self test are as follows:

- OK: Pass
- ERR: Fail
- ??: Test skipped

Valve status is shown by two 8-character strings, which should be "--OOOOOO OOOOOOOO" if all is well. Each character represents one valve. '–' means that the valve functions properly. 'O' means it is open. 'S' means there is a short. In the current Quest, only two of the 16 valves are actually used. That is why the screen normally shows two '--' characters, followed by 'O's.

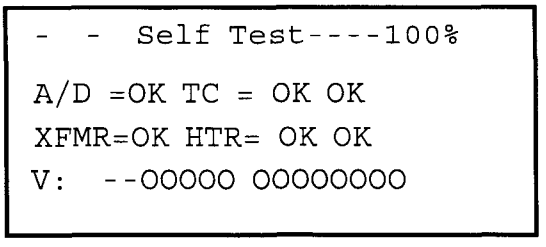


Figure 79: Self-Test Screen

Table Figure 80 lists the functions of the buttons on the front panel of the Controller when the Self Test Screen is showing.

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Button	Screen
MODE	Returns to OTHER FUNCTIONS screen
← →	N/A
VALUE SETTING	N/A
HEAT ON/OFF	Displays Heaters menu
AGITATOR ON/OFF	Starts/Stops agitator.

Figure 80: Self-Test Screen Button Functions

RESTORE DEFAULTS SCREEN

The Restore Defaults (Figure 81) screen is shown momentarily after Restore Defaults is selected from the OTHER FUNCTIONS menu.

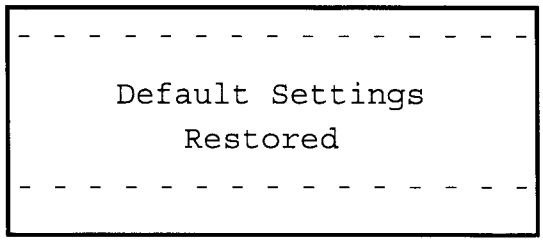


Figure 81: Restore Defaults Screen

Selecting Restore Defaults resets all programmable values (such as the agitator frequency values, LCD screen contrast, etc.) to their factory default values

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START/STOP HEATERS SCREEN

The Stop Heater screen (Figure 82) appears when the ON/OFF button is pressed (except if the Set Temperature menu is displayed). Use the FORWARD and BACK buttons (← →) to move the cursor to the desired operation and press MODE to activate.

```
-Start/Stop Heaters-
Stop      Both A and B
Start A   A (is now OFF)
Start B   B (is now OFF)
```

Figure 82: Start/Stop Heater Menu Screen

Figure 83 lists the functions of the buttons on the front panel of the Controller when the Start/Stop Heater menu is showing.

Button	Screen
MODE	Returns to previous screen
←→	Moves cursor
VALUE SETTING	Moves cursor
HEAT ON/OFF	Starts or stops the heaters on Side A and/or B according to the position of the cursor
AGITATOR ON/OFF	Starts/Stops agitator.

Figure 83: Self-Test Screen Button Functions

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APPENDIX C: SPECIFICATIONS

QUEST 210

Reaction Vessels (20 total)	Disposable with 7 µm or 30 µm frit 5 or 10 ml transparent Teflon
Agitator	Proprietary vertical oscillation
Temperature Range	-40°C to 130°C (Recirculating chiller required for temperatures below ambient)
Dimensions Reactor Assembly (W×D×H) Weight Controller (W×D×H) Weight	 18" (45 cm) x 14" (36 cm) x 29" (74 cm) 75 lbs. 10" (25 cm) x 16.5" (42 cm) x 12" (30 cm) 20 lbs
Electrical Ratings Fuse	100-120VAC/200-240VAC 50/60 Hz 8.3 A T10AH/250V
Environmental Conditions	15°C to 35° C Ambient Temperature 30-70% Relative Humidity
Collection Racks	Part number 900098: Twenty 20 ml or 40 ml scintillation vials (included with system) Part number 900110: Twenty 20 mm x 125 mm test tubes Part number 900111: Twenty 20 mm x 125 mm test tubes

AUTOMATED SOLVENT WASH OPTION

Dimensions ASW module (W×D×H) Weight	 17" (43 cm) x 22" (56 cm) x 18" (46 cm) 58 lbs (26 kg)

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APPENDIX D: PARTS & ACCESSORIES

QUEST 210 SYNTHESIZER

The Quest 210 Organic Synthesizer is shipped from Argonaut Technologies with the parts required to successfully install the system. Some of the items (e.g., reaction vessels, polyethylene solvent/reagent filters, etc.) are considered consumables, and replacements can be ordered from Argonaut Technologies.

Visit our website *www.argotech.com* for more information about the Quest, including:

- Photographs and ordering information for Quest replacement parts and accessories on Argostore page
- Scientific Resources page which lists scientific and technical information in the form of :

  Application Notes

  Scientific Literature References

  Synthesis and Purification letters

- Quest Tips, which are designed to assist users with common instrument operation and synthesis procedures.

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