User's Guide

10,000 psi Circulation System

Auto version

Version 1.02

Daedalus Innovations LLC

Aston, Pennsylvania

United States

TABLE OF CONTENTS

LIST OF FIGURES

WARNINGS / INFORMATION

This device can deliver a continuous flow of high pressure fluids. In some system configurations it can deliver pressures higher than the NMR cell can withstand. Therefore, it is critical that users have a thorough understanding of this manual and how this system operates before using this equipment.

When configured for thermal regulation of samples fluids exiting the hot side heat exchanger and returning to the circulator can be 150°C or more. Caution must be exercised when touching tubing or other components of the system as they may be hot enough to cause severe burns.

Under no circumstances should the circulator be operating at high temperatures without a refrigerated bath circulating water through the cool side heat exchanger. Failure to do so will cause damage to internal components.

If the **SYSTEM PRESSURE** suddenly drops immediately hit the **E-STOP** valve to cut the flow and operation of the circulator. It is important that the role of this component is understood before use.

The accumulator within the circulator requires a minimal gas charge before the fluid flow is started. Failure to do so can jam and render ineffective the functionality of the accumulator.

When operating the circulator there must be a 90 psi nitrogen gas supply to internal air-actuated valve. This opens the valve to allow external flow. Pressure will build in the system even with this valve closed which can cause an overpressure condition.

Under no circumstances should the circulation component be operated at the same time the gas booster is delivering the nitrogen gas charge to the accumulator.

The nitrogen gas charge on the accumulator should be between 25%-60% of the maximum operational pressure.

The overburden cell should be heated to thermal equilibrium prior to increasing the pressure to experimental pressure.

Always perform a leak test of the system prior to running the system to high temperature.

It is preferential to use the interface to smoothly decrease pressure in the system rather than using the **VENT** valve to perform a rapid depressurization.

Always be sure to **VENT** the system with the **E-STOP** valve opened to release any remaining pressure in the system/overburden cell before disconnecting any high pressure connections.

Whenever making high pressure connections always use an appropriate sized wrench to prevent counter rotation of the fitting hardware. Failure to do so could break loose downstream connections or loosen the bulkhead ports.

ITEMS TO BE SUPPLIED BY THE END-USER

Some additional tools may be required to complete the setup or maintenance of the instrument.

Metal tubing cutter: A 1/16" and 1/8" metal tubing cutter and deburring tool will be necessary to cut sections of tubing for making connections between the rock core cell and external pressure delivery devices.

Stainless steel anti-seize compound: Stainless steel is relatively soft. Depending on the applied torque it is possible to generate a condition where the parts seize. This is especially true for adapters to pipe fittings used in this system. The application of an anti-seize compound will extend the life of the components. A light grade machine oil may be used as a substitute, but is not as effective as a proper anti-seize compound.

Teflon tape: Some joints, such as those to the gas cylinders, may require Teflon tape for proper assembly. Higher density tape will perform better for higher pressures. When connections are made between stainless steel components a grade of Teflon tape specific to stainless steel is recommended.

CONVENTIONS USED IN THE MANUAL

To aid in the description of procedures and items in this manual when referring to specific components of the system all capital letters and bolded text will be used. For example the text **SYSTEM PRESSURE** box refers to the items within the silkscreen box bearing this title on the front panel of the circulator and corresponds to the Figure 1 block diagram. The text ***START*** refers to the start button on the main screen of the interface module. When other sections of the manual are referenced the section title will be in bold italics (e.g. ***Operating Instructions)***. When specific interface module menus are identified it will be referenced with italics (e.g. *Zero Sensors Page).*

The terms for describing tubing outer diameter is abbreviated OD, and the term inner diameter is abbreviated ID.

CONNECTION DETAILS

Fittings and adapters have been included for making connections to the rock core cell. These can be either with 1/16" tubing or 1/8" tubing. For both cases a compression sleeve is used to hold the tube while under pressure and must be assembled with sufficient torque to provide proper holding power. The tubing must also be properly prepared for optimal sealing performance.

For the 1/16" tubing, the high pressure 15-AM1 glands and 15-2A1 collars are used.
For the 1/8" tubing, the high pressure 15-AM2 glands and 15-2A2 collars are used.

Whenever assembling high pressure connections always use a wrench on the gland nut and secondary wrench on the attachment fitting to prevent counter rotation during tightening.

These steps should be followed when making a high pressure connection:

i) Cut and deburr the end of the tube section.

ii) Assemble the gland then sleeve onto the tube end.

iii) Insert the end of the tube into one of the stainless steels fittings until it bottoms.

iv) For the AM1/AF1 style fitting tighten the gland to 55 in·lbs. A "bottoming out" or "dead stop" should be felt when the connection is properly assembled. For the AM2/AF2 style fitting and initial application of 120 in-lb. will compress the sleeve part way. A second step requires application of torque to 300 in-lbs. to fully compress the sleeve. These are only guides and actual torque may be higher.

The AM2 style fittings are difficult to assemble and without use of anti-seize compound could damage the associated adapter that is being used for the assembly purposes. Should damage occur a 1/2"-20 tap and die can be used to reprofile the threads. Always use tapping fluid for these operations.

These connectors are all from High Pressure Equipment Company. Additional tech support is available on their website www.highpressure.com.

THEORY OF OPERATION

The block diagram of the 10,000 psi circulation system and the accessory equipment cart is shown in Figure 1. The circulation system is designed to provide near pulseless confining fluid to Daedalus overburden NMR cell. The primary working fluid is a fluorinated heat transfer fluid. The high pressure circulation is provided by an air-driven liquid pump that delivers 0.65 ml / pulse. With suitable air flow and pressure this system can deliver up to 210 ml/min at 10,000 psi.

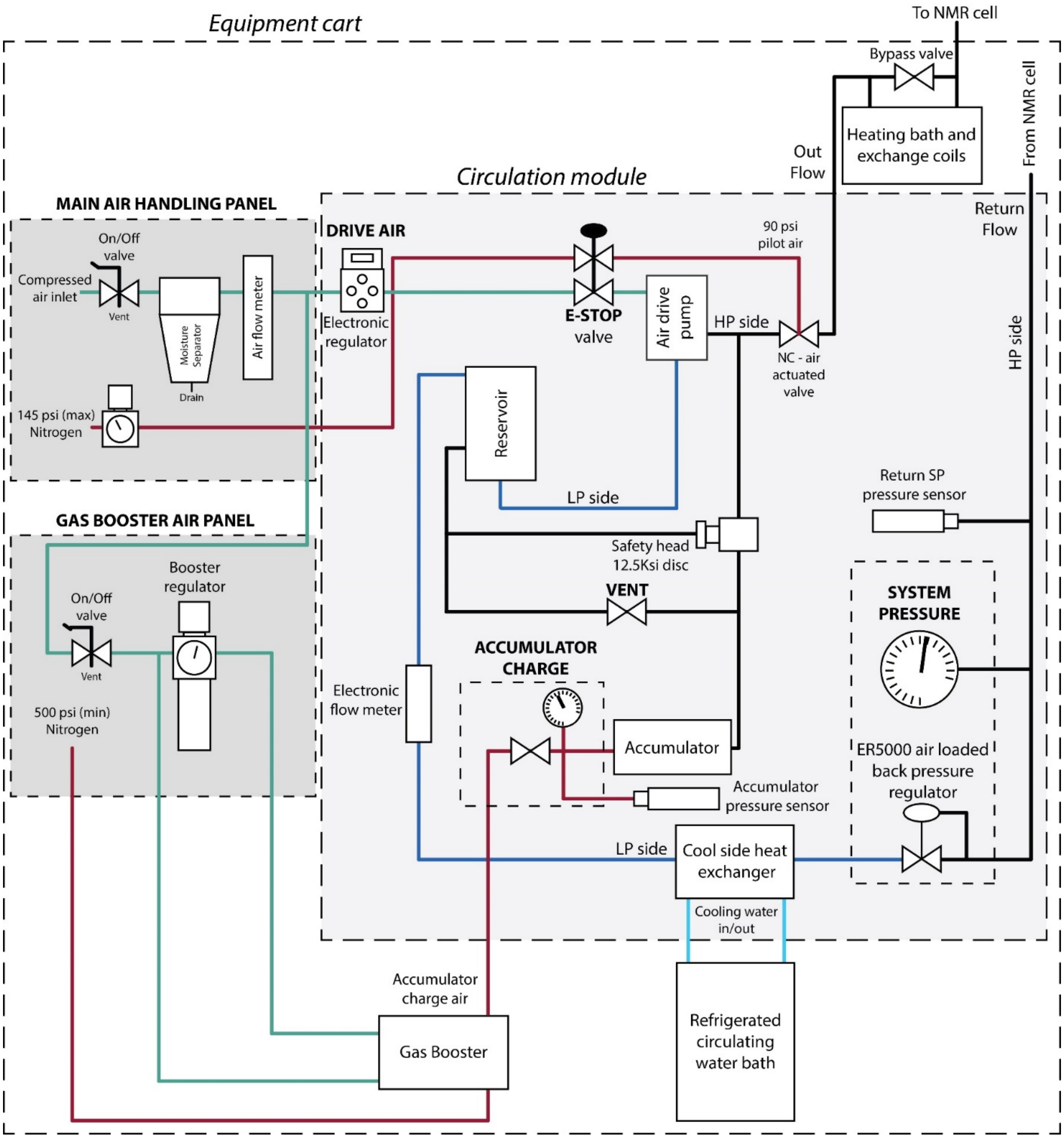

Figure 1: Recirculation system block diagram

It is assumed in this description that the 10,000 psi circulation system is mounted on the accessory equipment cart. The equipment cart provides a convenient focal point for all the necessary support hardware required for operation. The equipment cart also contains air handling sections for driving the various components of the system.

The circulator functions when compressed air pushes the internal pump piston into the suction mode to draw low pressure fluid into the pump from the reservoir. An internal air spool valve is also retracted until it reaches the discharge position at which time the pump piston is reversed into the fluid discharge direction. Hydraulic multiplication allows the pump to achieve pressures that are much higher than the driving compressed air pressure. In the case of this system, the pump has a 220:1 compression ratio with a maximum pressure of 31,900 psi. Such a high compression ratio allows the pump to driven by a lower compressed air pressure, and allows smaller air compressors to be used.

The compressed air to the full system is delivered from a single inlet connection point on the **MAIN AIR HANDLING PANEL**. The main inlet valve has a locking actuation lever on it which requires the user to physically move the locking mechanism to turn the valve into the open position. The valve can be closed without manipulating the lock. This same valve also has an auto venting feature such that when the valve is closed it will vent all upstream air from the system. The compressed air then flows through a moisture separator and through an air flow meter. From there it splits with one branch going to the circulator, and the other branch to a valve that feeds the gas booster.

The compressed air moves to **DRIVE AIR** port on the front of the circulator then to the internal electronic regulator. The air then passes to a manually controlled valve labelled **E-STOP.** This valve controls the flow of air to the air-driven pump as well as controlling the actuation of the NC (normally closed) valve. Operation of the pump requires a conscious act on the part of the user to pull the **E-STOP** knob out and start the flow. This **E-STOP** can also be pushed in to immediately cut the flow out of the circulator should any downstream problems occur.

Each pulse of the pump causes a significant spike in the system pressure which needs to be dampened. This is handled by the accumulator. This unit has an internal piston that moves to absorb the volume increase from each pulse and gradually releases it into the system between pulses. To accomplish this the piston must be backed by a suitable fluid, in this case nitrogen gas, that can readily compress more than the working fluid.

There are two important features within this region of the block diagram. Notice that there is a safety head device inline with the output of the air driven pump. This has a 12,500 psi burst disc installed. Should a situation occur where the system exceeds safe operating levels for the circulator this disc will burst and vent all fluid back to the reservoir. The system will not build pressure until this disc is replaced.

The other item of note is the **VENT** valve. This allows the system pressure to be vented directly to the reservoir provided the NC valve is open. Otherwise it will simply vent the pressure from the accumulator and associated pressure lines. The accumulator does hold a significant volume of fluid so rapidly opening this valve at high pressure will cause a noticeably audible discharge of fluid back into the reservoir.

In Figure 1 the charge on the accumulator is provided by the gas booster. This is also an air-driven pump, but with a much larger volume (though smaller compression ratio) and with check valves capable of handling gas media. This device, also driven by compressed air, converts relatively modest nitrogen inlet pressure to high pressure gas suitable for pulse absorption. Typically the accumulator should have a charge between 25% and 60% that of the maximum working pressure for the experiment. One consequence of this approach is that at or below the accumulator charge pressure there will be no pulse dampening.

The final stage before leaving the system is to pass through the air-actuated valve controlled by the **E-STOP** manual valve. This valve requires 90 psi nitrogen gas to actuate. With it closed pressure will build, and be reported, only on the **ACCUMULATOR CHARGE** gauge. This condition is only possible if the nitrogen feed line is not charged from an external cylinder as expected.

The fluid, now pulse dampened, will exit the system and to the coils suspended in the heating bath and on to the overburden cell. There is a bypass valve that when opened diverts fluid from the heating coils. After passage through the overburden cell it returns to the circulator and passes through an internal pneumaticaly controlled back pressure regulator. Ultimately, it is this back pressure regulator that controls the pressure in the system. The pressure reported on the **SYSTEM PRESSURE** analog gauge is not necessarily the pressure in the overburden cell, but does accurately report the pressure of the returning fluid. Note, however, that the pressure of the fluid exiting the circulator, as indicated on the gauge in the **ACCUMULATOR CHARGE** box may not be the same as the pressure reported by the **SYSTEM PRESSURE** gauge. A moving fluid will have an associated pressure drop across the system which is reflected in the difference between the two gauges.

Once past the back pressure regulator, the fluid is at atmospheric pressure. This fluid is also very hot and must be cooled prior to returning to the reservoir. This is to protect the air-driven pump, which has a maximum media temperature of 60°C. To do this the fluid passes through a cool-side heat exchanger which is flooded with thermally regulated water from a refrigerating cooling bath. The recommended flow volume is 17 L/min which can readily cool returning fluid at 150°C back to a proper temperature. The fluid then travels through a fluid electronic flow meter and back to the reservoir.

The feedback from the pressure sensors, flow meter, and thermocouples are all processed by the internal microcontroller and passed to the touchpad interface module. It is important to note that the interface module does not contain the control electronics such that if it is disconnected the circulator will continue to operate.

When the heat tracking routines are active the feedback from the thermocouples serve as the parameters for a PID routine that is used to set the heat bath temperature. Typically the heat bath temperature is set high to promote more rapid heating, but as the temperature approaches the cell temperature setpoint the heat bath setpoint is gradually lowered to deliver the proper themal output. The heat bath is not refrigerating, but due to the large heat transfer to the flowing confining fluid the bath temperature will rapidly decrease to level defined by the microcontroller.

Other parameters such as the flow can be monitored by the microcontroller and adjusted to a constant level using additional feedback from the flow sensor. There is also a mechanism that automatically steps the pressure to the new setpoint. This is important when the setpoint is above the accumulator charge pressure. Change in pressure under those conditions causes wild fluctuations in flow and to provide a smoother operation those changes can be handled by the microcontroller.

INITIAL SETUP

The following instructions are provided as a brief guide to the steps required to assemble the unpacked items into the functional circulator. The result is to have the circulator ready to function as an independent unit, but without thermocouples or overburden cell connected. The details of connecting to the overburden cell are not contained within this section.

Shipped components required:

- HCTB-3030 heater

- Heat bath (8-liter) with heating coils installed
- Heat bath to bulkhead panel tubing manifold
- Pressure sensor bypass bridge
- PM-125 silicone oil (2 gallons)
- Touch panel interface module
- 6-ft. DB9 cable
- 10-ft. DB9 cable

Step 1: Place the *8-liter heat bath* in the recess on the top of the circulator. The coils can remain in place. Remove the AM2 plugs in the bulkhead couplers using a 1/2" wrench on the gland and a 1" wrench on the bulkhead coupler to prevent rotation of this component. The AM2 plugs can also be removed from the bulkhead panel and the circulator outlet port at this time. There may be fluid in these lines so some leakage may occur.

Step 2: Loosely connect the *heat bath to bulkhead panel tubing manifold* to the heating coils, the bulkhead panel, and the outlet from the circulator. See Figure 2 for reference. Though shown in the figure the HCTB-3030 heater should not yet be connected to the bath.

Figure 2: Heat bath with tubing

Step 3: With the fit confirmed tighten the four connections to the bulkhead couplers using the 1/2" wrench on the gland and the 1" wrench on coupler to prevent rotation. The suggested torque is 160-200 in-lbs. When tightening the connections the tubing will also tend to rotate. This can be minimized if the tubing is held in a static position while tightening the gland. Check the other connections as well. Anchor the tubing to the circulator cart frame using the cable tie tubing holders.

Step 4: Connect the *pressure sensor bypass bridge* to the bulkhead panel at the right of the cart using the same procedure as Step 3. See Figure 12 for reference. The DB9 terminated cable can be connected to the **SENSOR CABLE** on the back of the circulator.

Step 5: Add the PM-125 silicone oil to the bath. This can be done through the opening where the heater will sit. Fill until the oil is about ~2.5 cm from the top of the bath. This oil will expand upon heating so do not overfill the bath. After the heater is in place the small panel on the top face held down by screws can be removed to add more oil. The cross head screw can be removed to make this movement of this panel easier.

Step 6: Place the heater in position on the bath. Follow the instructions for this device if needed. The shipped 6-ft DB9 cable can be connected to the port on the heater to the **HEAT BATH** port on the back of the circulator. The bath can be plugged into the power source. If the system is for 230V there is a transformer at the bottom of the cart into which the heater should be plugged. This converts 230V to 115V suitable for the heater. This transformer must be turned on as well. See the manual for this device for further details.

Step 7: Fill the cool side water bath with water. Once complete the bath should be plugged in and turned on and allowed to flow to fill up the water filter chamber and the heat exchanger inside the circulator. The bath is independent of the circulator so there is no need to have the circulator powered for this operation. Filling up the extra volume will likely trigger a low level alarm requiring additional water be added to the system. See the manual for this bath for details on operating the bath.

Step 8: Connect the touch panel LCD interface module to the circulator using the 10-ft. DB9 cable. Place this in a convenient location for use.

Step 9: Plug in the circulator using the shipped cable. The circulator uses auto-ranging power supplies so any voltage between 100-240V, 50/60Hz will work.

Step 10: Connect the compressed air lines and the nitrogen gas supply lines to the **MAIN AIR HANDLING PANEL.**

The gas booster must be filled with a charge prior to running fluid at pressure, but otherwise the system is ready for operation.

AIR DELIVERY PANELS

It is expected that filtered, dry air will be used to drive the system. However, the compressed air does flow through a moisture separator to remove any large droplets of water that remain. This is meant as a backup, and will not serve to fully dry the air. That must be performed upstream from the connection to the cart with an appropriate sized air dryer. If the moisture separator fills with water then the air is not suitably dry and this will decrease the life of the air-driven pumps.

> The compressed air supplied to the circulation system must be dry and filtered. Failure to do so will decrease the effectiveness and lifetime of the air-driven components.

Main Air Handling Panel:

The compressed air to the full system is delivered from a single connection point located on the **MAIN AIR HANDLING PANEL** shown in Figure 3. This connection is a push-to-connect tube fitting for 3/8” OD tubing with a 1/4” NPT male pipe fitting. This connection can be swapped out to accept larger compressed air tubing if necessary.

The main compressed air valve has a locking lever on it which requires the user physically move the locking mechanism to turn the valve into the open position. This is to assure that the system cannot be accidentally activated. The valve can be closed without manipulating the lock for quick shutoff. This same valve also has an

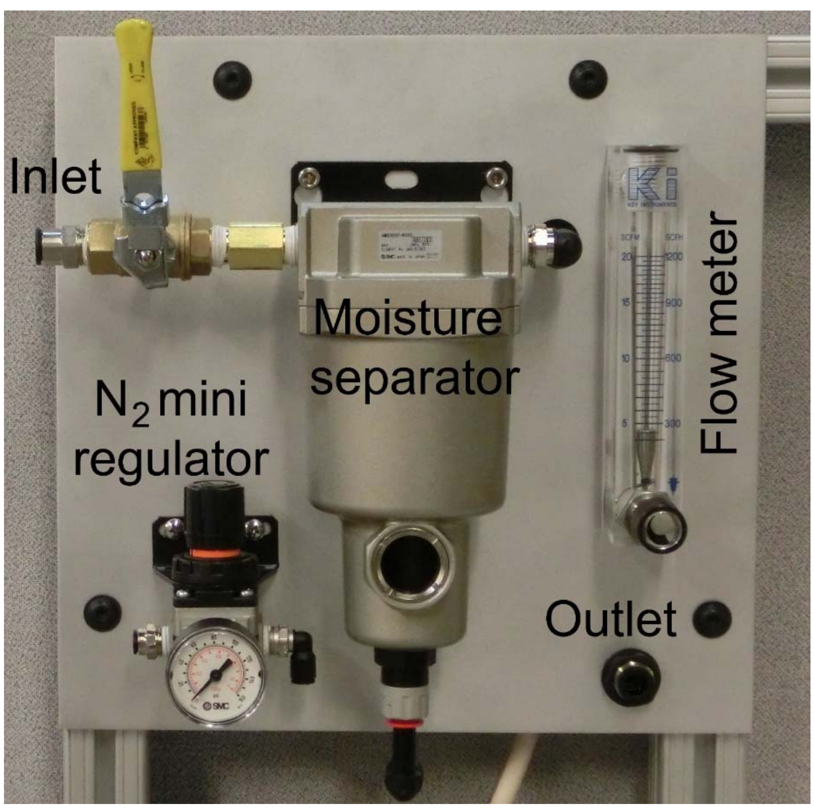

Figure 3: Main air handling panel

integral vent feature such that when the valve is closed it will vent all downstream air pressure from the system. This feature can also be used to vent all the upstream air as well so care should be taken to fully and rapidly open the valve when intended.

All air is piped through a moisture separator which will remove excess droplets of water from the air stream. This is meant only as a backup and is not intended to be the primary air dryer in the system. It has an automatic drain that actuates if the fluid level reaches a threshold. There is a 3/8” OD tubing push-to-connect fitting at the bottom that allows routing of the excess fluid to a suitable receptacle.

The inline air flow meter can measure 4-20 SCFM. It will not accurately report air flows below 4 SCFM. During operation the pump will draw air at high rates followed by a pump pulse during which time the air flow requirements will drop. Thus it is expected that the indicator will be fluctuating during operation. The air flow is split after the air flow meter with one branch going to the regulator on the circulator, and the other branch to the **GAS BOOSTER AIR PANEL**.

The **MAIN AIR HANDLING PANEL** also has a mini regulator for metering nitrogen gas pressure to the air-actuated valve within the circulator. The inlet port is a push-to-connect fitting for 1/4” OD tubing. The maximum inlet pressure is 150 psi. The outlet from this regulator connects directly to the nitrogen inlet on the back of the circulator. The output should be set to 90 psi for proper actuation of the valve. This regulator will vent downstream air by lowering the setpoint pressure with the knob, but otherwise will not vent. Therefore, care should be used when disconnecting the tubing from this regulator as it could still be under pressure.

The mini regulator on the main air handling panel is for nitrogen. The maximum inlet pressure is 150 psi. This should be set to 90 psi outlet for optimal actuation of the air controlled valve in the circulator.

Gas Booster Air Panel:

The specifics on the operation of the gas booster is described elsewhere in this manual. In this section the air handling panel for the gas booster is described. The compressed air supply for this panel comes from a branch off the main air handling panel. It is the same line that supplies the circulator and under no circumstances should both the circulator and the gas booster be driven at the same time. However, in order to operate the gas booster the valve on the main panel must be open. The gas booster panel is shown in Figure 4.

The compressed air valve on this panel has a locking mechanism that is engaged when in the closed position. This lever must be physically disengaged in order to turn the lever into the open position. This valve also has the same integral vent feature as the main panel compressed air valve. When switched from open to close it will vent all downstream pressure. The reverse is also true so it is important to rapidly turn from closed to open to prevent significant upstream pressure loss.

The compressed air line then branches from the valve with one line passing to the regulator, and the other 1/4" OD tubing running direct and unregulated to Port X (see booster manual) on the gas booster. The panel regulator has an integrated filter and also acts as a moisture separator. The drain is automatic so if there is a build-up of

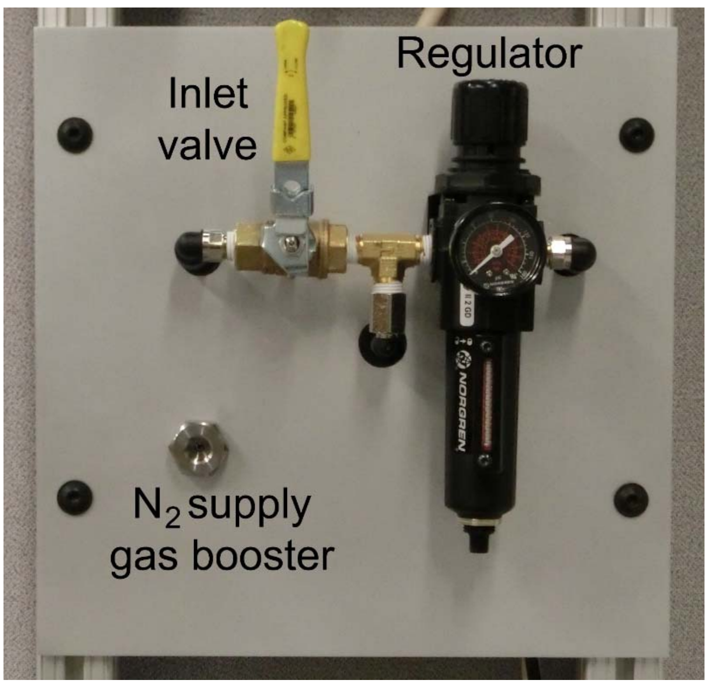

Figure 4: Gas booster air handling panel

water it will leak out the bottom of the regulator. This condition should not occur if the compressed air supply is adequately dry. Pressure to the gas booster is achieved by turning the control knob on the top of the regulator clockwise. During operation the pressure indicator will fluctuate with the pulsing of the gas booster.

The other component on the panel is the inlet port for nitrogen gas, which connects directly to the inlet (Port A) on the gas booster. This port requires the standard AF2 style fitting for 1/8” tubing. It is recommended that the 1/8” tubing have a minimum inner diameter (ID) of 0.06”. The larger the ID of the tubing, the more complete the filling of the gas booster with nitrogen between cycles. The tubing must be rated to withstand the inlet nitrogen pressure.

GAS BOOSTER SPECIFICS AND OPERATION

The accumulator within the circulator requires a nitrogen pressure charge to absorb the pulses from the air-driven pump. This pressure charge is typically between 25%-60% of the projected working pressure. Standard cylinders cannot supply pressure over that entire range so a separate device is required to build up to that pressure from lower pressure nitrogen supply. This is accomplished by the gas booster. The charging action only needs to be done periodically as the charge circuit will hold the pressure for an extended period.

Under no circumstances should the gas booster and the circulation system be operating at the same time.

The gas booster is a model DLE75-1 from MAXPRO Technologies. This is designed to work specifically with gases only. It has a 20:1 compression ratio with a maximum outlet pressure rating of 10,875 psi. It is driven by compressed air with a maximum drive air pressure of 145 psi. Relative to the drive air pressure the maximum outlet pressure of the booster is 1:75.

The specifications for this unit indicate that using 550 psi inlet pressure will allow the unit to reach maximum outlet pressure at a drive air pressure of 145 psi. However, this requires a compressor than can deliver a very large volume of air. This may not be something is present in a laboratory setting. Instead, higher inlet pressures can be used to more rapidly reach the necessary accumulator charge pressure even with a smaller air compressor.

Shown in Figure 5A are the compressed air connections. The 3/8” OD tubing on the left of the image comes from the regulator on the **GAS BOOSTER AIR PANEL**. Also from that panel is the black 1/4” OD tubing which is an unregulated line that pushes the internal spool valve back into position between cycles. In Figure 5B the 1/8” tubing from the **GAS BOOSTER AIR PANEL** connects on the left side of the booster head (Port A) with the 1/16” tubing on the right (Port B) connecting to the bulkhead port within the **ACCUMULATOR CHARGE** box on the circulator.

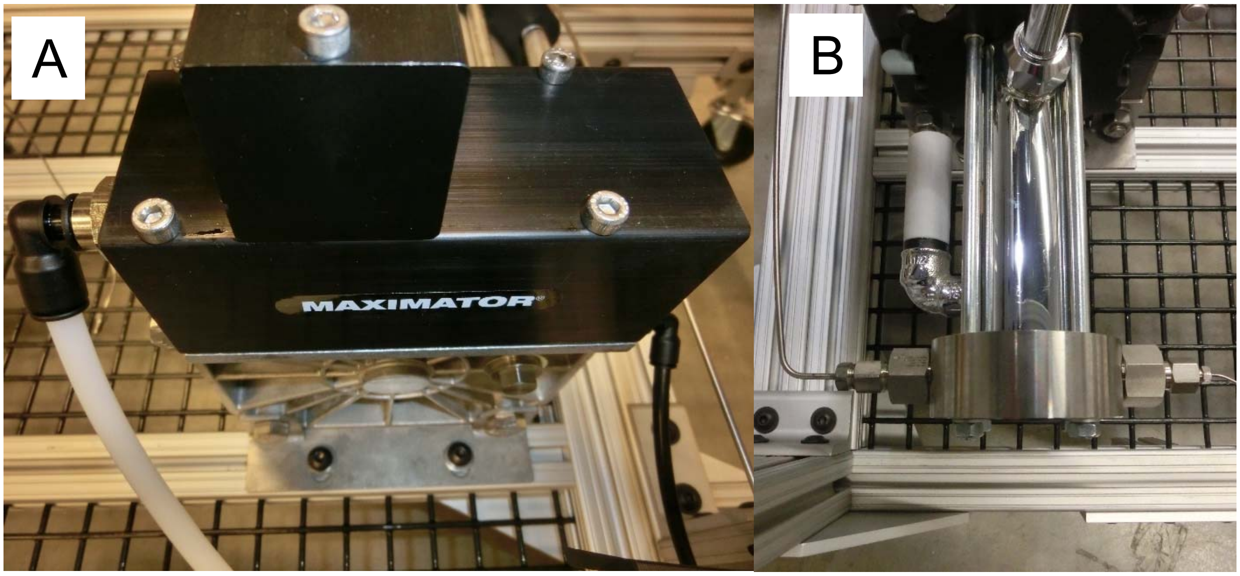

Figure 5: Gas booster inlet/outlet connections

The nitrogen charge on the accumulator can be as low as 25% of the maximum operating pressure and as high as 60% of the operating pressure. There is little difference in the pulse dampening effect over this broad range. This also allows for a range of confinement pressures to be performed without the requirement to correspondingly match a specific accumulator charge pressure. The accumulator charge will hold for several weeks, and if the system is operated only at a single pressure the recharge procedure will not need to be performed often.

The nitrogen charge on the accumulator should be between 25% and 60% of the maximum operating pressure of the circulation system.

A lower charge pressure relative to the confinement pressure target makes the response to pressure changes by the back pressure regulator slower to realize. The initial build up to pressure will take longer which also means more fluid is stored in the accumulator with high release potential. Ultimately though there is no significant performance consequences to using lower accumulator pressures under normal operation.

Delivering the Accumulator Charge:

With the appropriate compressed air connections made to the air handling panels the accumulator charge can be generated according to the following steps.

Step 1: Open the nitrogen cylinder valve and set the outlet pressure to between 500 – 2000 psi.
Step 2: Connect the tubing from Port B of the gas booster to the bulkhead port within the **ACCUMULATOR CHARGE** box.
Step 3: Open the valve between the nitrogen cylinder and the **GAS BOOSTER AIR PANEL**. See *Nitrogen Manifold Instructions* for an example of such regulator.
Step 4: Open the valve on the circulator within the **ACCUMULATOR CHARGE** box. If the pressure in the accumulator is less than the outlet pressure from the cylinder it will first fill the accumulator with nitrogen. This will take time depending on the current fill state. Do not continue to the next step until the gauge within the **ACCUMULATOR CHARGE** box equals the cylinder outlet pressure.
Step 5: Open the main compressed air valve. Be certain the circulator does not start.
Step 6: Open the compressed air valve on the **GAS BOOSTER AIR PANEL**.
Step 7: Set the regulator to 90 psi. This setting is selected to allow for maximum air flow even for lower accumulator charge targets. It can be changed as needed.
Step 8: Depending on the air flow capacity of the compressor used the gas booster will likely only pulse every couple of seconds. Watch the build up of pressure on the gauge within the **ACCUMULATOR CHARGE** box. Continue to allow the gas booster to function until the desired target is reached.
Step 9: To stop the gas booster close the compressed air valve on the **GAS BOOSTER AIR PANEL** panel to vent the downstream pressure contained within the system. Reduce the regulator setpoint to zero to assure the booster does not start prematurely the next time the accumulator charge is set.
Step 10: Close the valve within the **ACCUMULATER CHARGE** box on the circulator.
Step 11: Disconnect the 1/16” tubing from the circulator and stow for later use.
Step 12: If necessary change the accumulator pressure to the new value in the *Other Functions* menu. Alternatively toggle the power on the circulator so the new reading is made by the system.

It is possible the air compressor that is used for this purpose may not be capable of delivering the required air flow to build up the charge on the first attempt. In this condition the unregulated compressed air line equals the drive air pressure such that the spool valve (described previously) is not able to be fully retracted for the next cycle. The gas booster will make a markedly different sound similar to venting of air, but will not have the characteristic knock of a full pulse. If that happens the compressed air valve on the **GAS BOOSTER AIR PANEL** should be closed to allow the compressor to build back up to full pressure. The procedure can then be continued from Step 5 from above.

CIRCULATION SYSTEM FRONT PANEL

Shown in Figure 6 is the front panel of the circulator. Many of the visible items are described in the ***Theory of Operation*** section of this manual. Further description of the various components and how they interact in the complete operation of the unit will follow in this section.

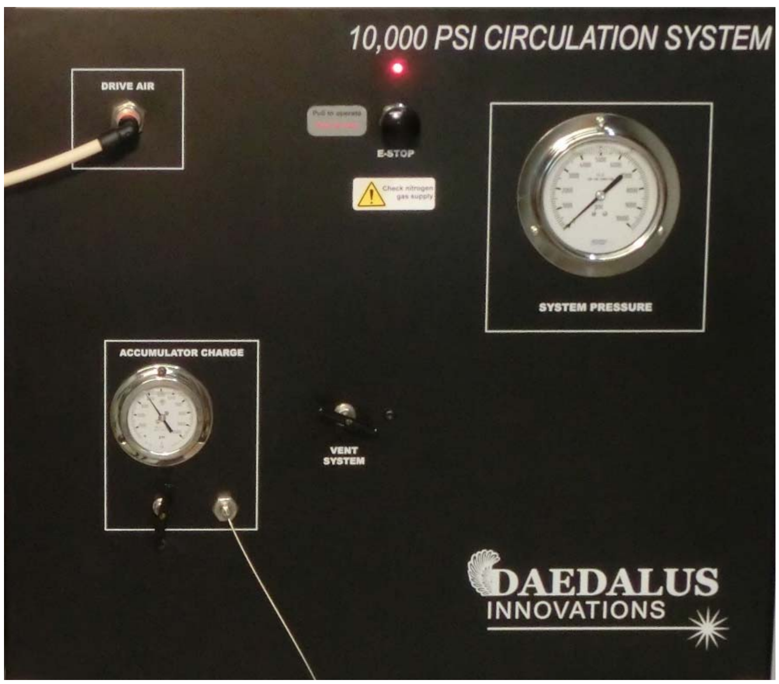

Figure 6: Circulator front panel

The **DRIVE AIR** port of the circulator panel is where the compressed air from the **MAIN AIR HANDLING PANEL** enters the circulator and feeds to an internal electronic regulator. The drive air controls both the maximum pressure that can be obtained by the system as well as the liquid flow delivery of the system. As was indicated in the ***Theory of Operation*** section the maximum pressure the air drive pump can deliver is 31,900 psi, but due to a number of limiting devices it cannot reach that pressure. Therefore, any excess air flow above the current pressure setting serves to drive the pump faster and thus greater flow of fluid.

Do not operate the circulator (open the **E-STOP** valve) without checking first to assure the low pressure nitrogen gas is flowing to the circulator. Failure to do so could cause an overpressure condition and require maintenance to return the system to operational.

The compressed air to the pump is restricted by the **E-STOP** valve. Actuating this valve is the final physical step a user must take in order to start the flow of fluid. All the necessary high pressure tubing connections must be done prior to actuating this valve to prevent a rapid discharge of fluid. The **E-STOP** valve also controls the flow of nitrogen gas to the internal air-actuated valve. Without this low pressure nitrogen charge to the valve the pathway will not be open to external circulation of the fluid. However, pressure will build on the other side of the valve which can be monitored by the **ACCUMULATOR CHARGE** gauge.

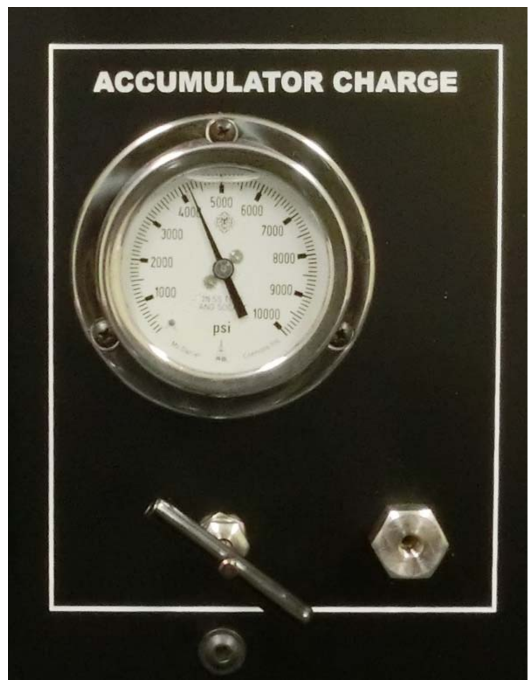

Figure 7: Accumulator charge section

The condition where the air drive pump is operating and no actuating nitrogen pressure is present should be avoided. There is a possibility of overpressure in this condition, which will be relieved by the 12,500 psi rupture disc, but this result will require replacing the disc before the system can be used. This is time consuming and easily avoided by assuring the nitrogen gas is flowing to the system.

In Figure 7 is the **ACCUMULATOR CHARGE** section of the panel. The open bulkhead port in this section is for 1/16” tubing terminated by an AM1 fitting and connects directly to the outlet (Port B) of the gas booster. When making a connection to this port always use a 3/4” wrench to prevent counter rotation of the coupler. The valve in this section opens the path from the bulkhead coupler to the internal accumulator described in the ***Theory of Operation***. The gauge in this portion of the panel reports both on the pressure in the accumulator, and reports on the pressure of the fluid leaving the circulator (when it is above the charge pressure), or in the improper case where the air-actuated valve is closed it reports on the pressure in the system up to the valve.

It is possible, and certainly likely that the pressure reported by this gauge is significantly higher than the pressure reported by the **SYSTEM PRESSURE** gauge. This is due to the fluid dynamics of the flowing fluid within the tubing of the system causing a pressure drop from the start to the end of the circuit. At room temperature this pressure drop can be quite significant, but as the fluid is heated the friction becomes less and this gauge and the **SYSTEM PRESSURE** gauge will be in better agreement.

The **SYSTEM PRESSURE** box contains the analog four inch pressure gauge. This gauge reports on the pressure of the fluid returning to the circulator. This is not the pressure in the overburden cell. Instead it will read lower than the pressure in the cell. It is present to provide a quick reference of the current working pressure without needing to look at the **INTERFACE MODULE**.

Figure 8: System pressure gauge

This portion of the fluid circuit is on the other side of the **E-STOP** valve from the pump so it is important to recognize that if this valve is closed due to a lack of nitrogen actuation pressure this gauge will not provide a reading on building pressure even if the air drive pump is running.

The final component on the front panel is the **VENT** valve. As can be noted from the diagram of the circulator in Figure 1 the **VENT** valve when opened will release the pressure from the upstream side of the internal air-actuated valve. If that valve is open it will vent all pressure in the system. Actuation can cause a rapid decompression of the system and will allow any fluid stored in the accumulator to discharge into the reservoir. While this is not problematic for the system it is an uncontrolled release of pressure. It is better to use the **INTERFACE MODULE** to release the system pressure in a more measured fashion.

CIRCULATOR BACK PANEL AND FILL PORT

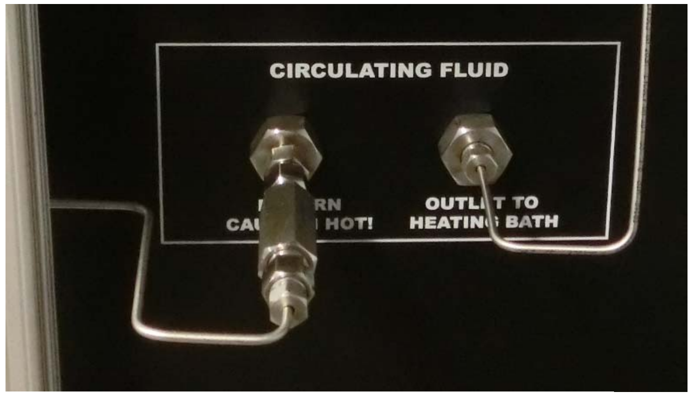

Figure 9: Circulating fluid ports

The circulating fluid outlet and return ports are located on the back of the circulator as shown in Figure 9. Both ports are bulkhead couplers for AM2 style fittings for 1/8” tubing. The figure shows the setup with the line to the heat bath connected and the 100 micron line filter in place on the return line. It is important to recognize that fluid returning to the circulator can potentially be very hot so caution should be used when manipulating these ports. That is the primary reason these ports are located on the back of the circulator. Always use a 1” wrench on the coupler body to prevent rotation of the coupler in the circulator case wall.

Shown in are Figure 10 the remainder of the operational liquid/gas ports on the back panel. The cool side heat exchange water connections are shown in this figure. These are for 3/8” ID tubing held in place by a screw-drive tubing clamps. The outlet from the mini regulator on the **MAIN AIR HANDLING PANEL** connects to the nitrogen port on the back. This supplies the required air to the internal air actuated valve controlled by the **E-STOP** valve.

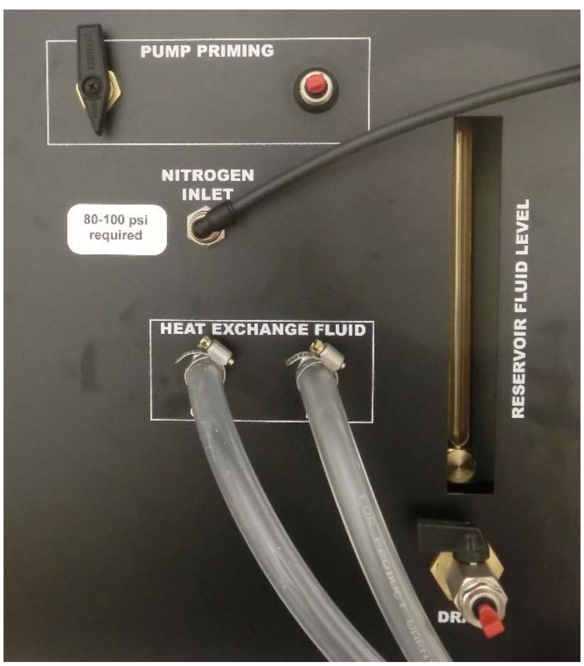

Figure 10: Back panel connections

The fluid level in the reservoir can be checked from the back panel of the circulator. There is no specific mark for being full. If the fluid level can be seen on the indicator there is likely enough fluid to run the circulator. However, there are a number of factors such as whether the circulator is used to fill the overburden cell that can impact this. There is also likely going to be a certain amount of fluid loss from disconnection of the overburden cell that will eventually drain the reservoir. Therefore, it is good practice to keep sufficient fluid in the reservoir to account for these eventual losses. Below the

RESERVOIR FLUID LEVEL is a valve that allows the reservoir to be drained.

The **PUMP PRIMING** contains a quarter turn valve and port that can be used to prime the pump. This usually does not need to be done unless the system has been fully drained. However, should this operation need to be performed there is a port for 1/4” tubing that can be connected to a syringe to be used to draw fluid up from the reservoir closer to the pump inlet to assist in initial priming of the pump. Once complete the valve can be closed and the port plug reinserted to assure fluid is not pumped out of the circulator.

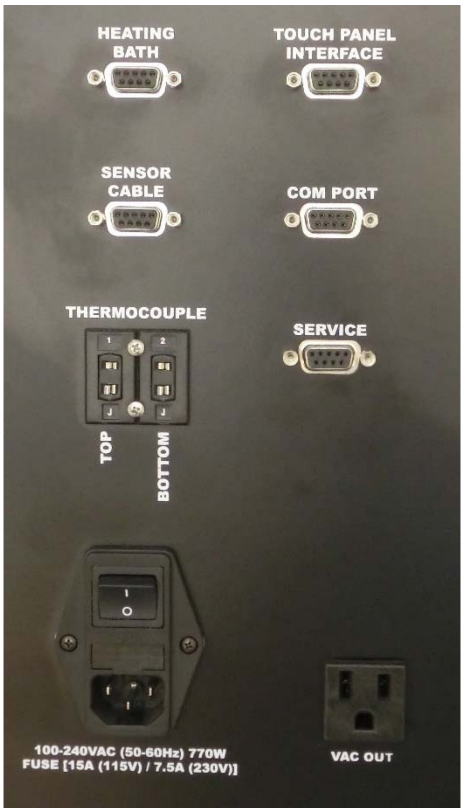

Figure 11: Electrical connections

Shown in Figure 11 are the various electrical connections located on the back of the circulator. The **HEATING BATH** port connects to the port on the HCTB-3030 heater. There is an included 6-ft. cable included for this purpose. The **INTERFACE MODULE** connect **TOUCH PANEL INTERFACE** port using the included 10-ft. DB-9 cable. The **SENSOR CABLE** connects to the pressure sensor that will provide the feedback to the system regarding the pressure of the confining fluid going to the overburden cell. The provided sensor has a cable attached with an appropriate DB9 connector for this purpose.

The **SERVICE** port is available to allow the microcontroller to be reprogrammed in the field. A programming cable was included with the shipment. During normal operation this cable is not connected. However, this port may be used for grounding purposes when monitoring the thermocouple temperatures using an external computer. See the ***Thermocouple Setup*** section for more details.

The thermocouple connection panel is where the feedback thermocouples are to be connected. The **TOP** and **BOTTOM** label below the panel correspond to the output reported on the **INTERFACE MODULE.**

The power module at the lower left has a C13 receptacle. The AC power is transmitted to the **VAC OUT** port. The heat bath should not be connected to this port as it will draw too much current. Rather it is provided to allow a convenient location to plug in a peripheral device such as a computer.

The brass elbow that protrudes from the left side of the circulator is the fill port for the circulator. The gray and black vented cap connected to this elbow allows for pressure equilibration both when the pump is drawing fluid and when fluid is vented back to the reservoir. This cap must be removed to add fluid to the reservoir. Use the included funnel for this purpose. Do not add fluid too fast or it will spill out of the elbow.

BULKHEAD PANEL AND SENSOR

Shown in Figure 12 is the pressure sensor connected to the bulkhead panel. This panel is on the right side of the circulator cart. It is rigid point on the cart to which the return line to the circulator and the outlet from the heating coils from the heat bath can be connected. The bulkhead couplers are isolated from the panel using Teflon inserts and washers. This minimizes heat transfer from the lines to the panel itself. However, it should be recognized that the tubing and bulkhead couplers themselves could be hot during operation.

Figure 12: Sensor connected to bulkhead panel

It should be noted that these bulkhead couplers, while tight against the panel, will not resist turning when making high pressure connections to the coupler. A second wrench must be used to restrain rotation of the coupler. In this case the 1” wrench should be on the outside portion, as seen in the image, of the coupler.

The circulator is shipped with the pressure sensor connected via a tubing tether to a high pressure tee. To that tee are two tubing sections bent to allow the sensor to be connected to the bulkhead panel. With the heating bath tubing manifold also connected the circulator can be operated in a self-contained mode. A plain section of tubing can also be connected in place of this bypass tubing, but the ER5000 controller will not receive feedback so pressure changes will not work. It is recommended instead to have the sensor in the loop when operating the circulator in a self-contained mode.

In this configuration the circulator can be operated to maximum pressure and with the heat bath operating. However, with heat, the cooling bath must also be switched on and the heat bath temperature should not exceed 150°C.

Whenever making high pressure connections always use an appropriate sized wrench to prevent counter rotation of the fitting hardware. Failure to do so could break loose downstream connections or loosen the bulkhead ports.

TOUCH PANEL INTERFACE MODULE

The user interface for the circulator is provided by the **INTERFACE MODULE.** This is a touch panel LCD device. Along with a module is a 10-ft. DB9 terminated cable that provides power and serial communication between the LCD and the microcontroller within the circulator. The module does not provide direct control of any of the relevant components of the circulator. It serves only as an input device. Therefore, the module can be disconnected from the circulator and it will continue to operate. This is an advantage if the cable were to become disconnected one need only plug it back in to resume operation. The disadvantage is the ability to stop the system will not be available when disconnected. Therefore, it is recommended that the DB9 cable be properly secured to the module.

Figure 13: Touch panel interface module

Main Screen:

Upon switching on the circulation system the touch panel interface box will boot and display the screen shown in Figure 14 without the data. After a few seconds data will start to populate the fields with the remainder of the data populating after a few more seconds. The interface will be unresponsive during this bootup phase. The screen is split into sections for which the description is provided below.

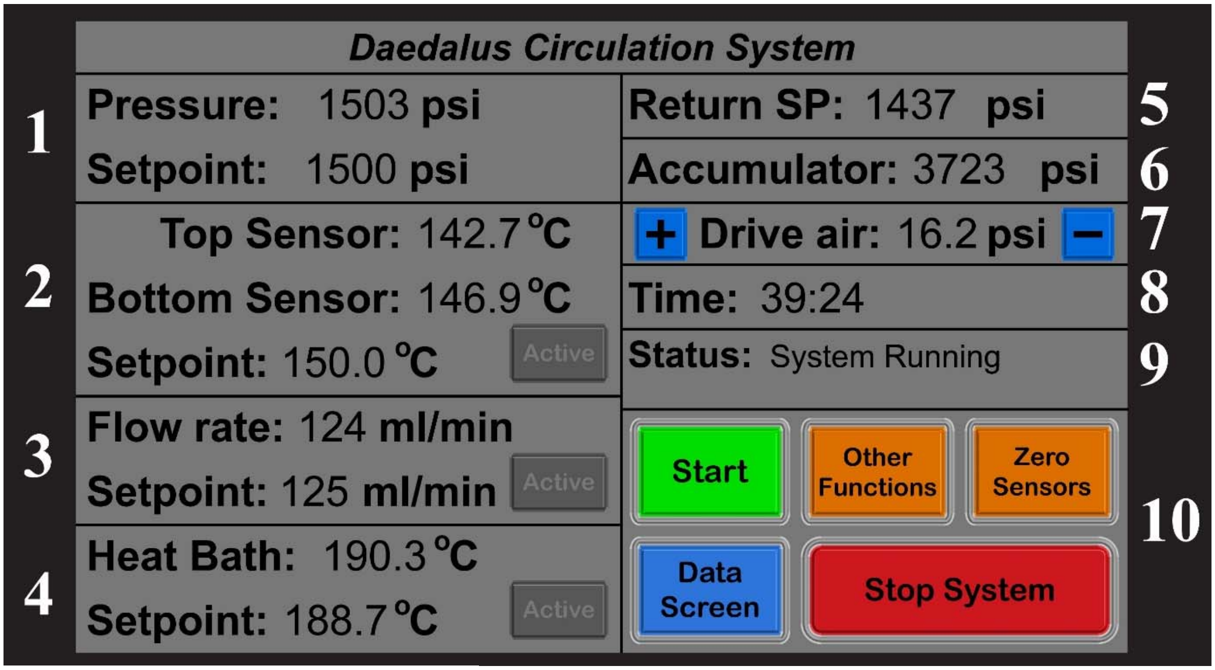

Figure 14: Main screen

Section 1: The pressure value reported here is the reading of the external pressure sensor placed near the overburden cell. This pressure is set by the internal ER5000 controller with back pressure regulator. It is the primary pressure reference for the cell. The setpoint can be altered by touching this section.

Section 2: The readings of the connected thermocouples are reported in this section. The top sensor value is for the thermocouple plugged into the circulator in the **TOP** port on the back of the unit while the bottom reading corresponds to the **BOTTOM** port. The setpoint in this section, set by touching the section, is the value to which the system will attempt to regulate the cell temperature.

Section 3: The flow rate through the internal sensor is reported here. The setpoint to which the system will regulate the flow can be set in this section.

Section 4: The current temperature of the heat bath is reported in this section. The setpoint of the heat bath can be set manually by touching this section. When the system is first turned on the heat bath will go to the previous setpoint. This may not be the optimal setpoint for the current experiment. Therefore it is a good idea to set the bath to an appropriate temperature soon after switching on the instrument. The setpoint will initially read zero to remind the user to make the required input.

Section 5: The pressure of the returning fluid is reported here. This value is always lower than the pressure in Section 1 due to the pressure loss of a flowing fluid. The actual pressure in the cell is between the pressure in Section 1 and that reported here. At low fluid temperatures this value can be quite large. The return pressure is also reported on the analog **SYSTEM PRESSURE** gauge on the front of the circulator.

Section 6: The pressure value shown is the charge pressure on the accumulator. This value is also reported on the analog gauge in the **ACCUMULATOR CHARGE** section. When the circulator is operating at pressures below the charge pressure there will be no significant change to this value. It is only when the pressure climbs above the original charge pressure will this value begin to change. Refer to the ***Theory of Operation*** section for more details.

Section 7: The current drive pressure is reported in this section. There is an increase and decrease button to adjust the drive air manually. The drive air pressure controls both the flow rate and the maximum pressure that can be obtained by the system.

Section 8: When the ***START*** button is pressed it start a timer reported in this section. It turns off by pressing ***STOP*** or other deactivation of the routines in Section 2.

Section 9: This reports any status messages. Usually this will just be a run and stop indicator. Other messages might be indications of the heat bath or thermocouples not being connected to the system.

Section 10: These are the stand alone action buttons. The operation of each is described in the next section.

Main Screen Touch Sections:

The screen is split into a number of touch regions, shown in Figure 15, that trigger specific functions as described below:

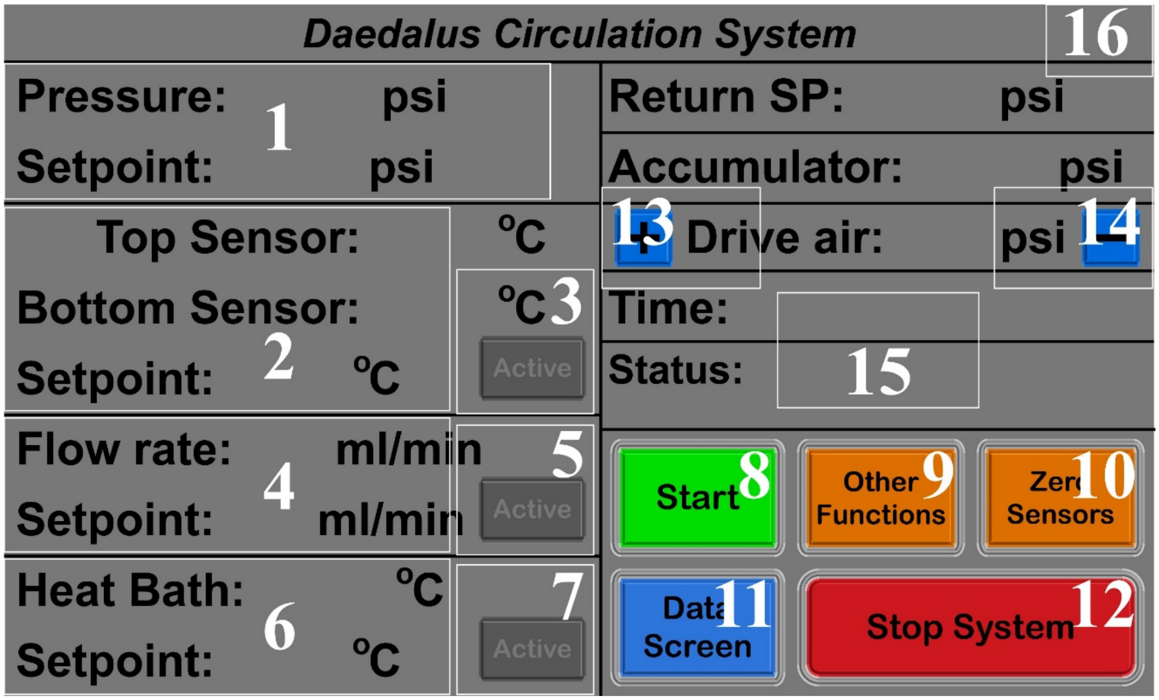

Figure 15: Main screen touch regions

Region 1: Change the pressure setpoint for the overburden cell. A keypad will be displayed for data entry. The change will be immediate.

NOTE: When the keypad is displayed, the operational parameters will not update.

Region 2: Change the temperature setpoint for the overburden cell. A keypad will be displayed for data entry. Implementing the change requires other steps.

Region 3: See the ***START*** button description as well. Touching this region will change the greyed ***ACTIVE*** button to a green active indicator. This serves to turn on the temperature adjustment routines. At this point the system will attempt to adjust the heat bath temperature bring the overburden cell temperature to the setpoint defined in Region 2. It is preferable to use the ***START*** button to initiate the temperature adjustment routines.

Region 4: Change the fluid flow setpoint. A keypad will be displayed for data entry. A flow of 125 ml/min is typically sufficient for thermal maintenance.

Region 5: Changing the setpoint (Region 4) does not cause any change in the operation. Instead it must be activated by touching this region to switch on the constant flow routines. This can be activated independent of other routines, and can be useful for maintaining a target flow rate as other activities are performed. In situations where the air pressure is not sufficient the flow may not reach the setpoint when at elevated pressures.

Section 6: Manually change the heat bath setpoint. A keypad will be displayed for data entry. The change is immediate.

Region 7: The greyed out ***ACTIVE*** controls routines that attempt to compensate for the large heat transfer to the confining fluid flowing through the heating coils. The bath controller often cannot keep the temperature at the setpoint due to this loss. Therefore, the circulator will adjust the setpoint behind the scenes to bring the actual bath temperature closer to the setpoint. The routines can be active during manual use, but it generally yields strange results. Instead the routines are turned on automatically when the temperature adjustment routines are activated using the ***START*** button. When activated in this manner the greyed indicator will not turn green until the cell temperature is near the setpoint and the heat bath temperature is more stable.

Region 8: The ***START*** button simultaneously turns on the cell temperature adjustment routines, the constant flow routines, the heat bath adjustment routines, and runtime timer. This is the recommended method for initiating the heating of the overburden cell as it establishes at the same time the parameters required to reach the target. It should be noted the heat bath ***ACTIVE*** indicator will not immediately turn on at this point; rather it will become active at an appropriate time in the routine.

Region 9: The ***OTHER FUNCTIONS*** button switches away from the main screen to the other functions windows described in a later section.

Region 10: The ***ZERO SENSORS*** button switches to a screen where the pressure sensors, thermocouples, and flow sensor can be adjusted.

Region 11: The ***DATA SCREEN*** button switches to a screen where the output from the thermocouples can be displayed.

Region 12: Hitting the ***STOP SYSTEM*** button once will turn off the temperature adjustment, flow, and heat bath routines and turn the drive air value to zero. This is meant to function as an emergency stop button should anything go wrong. A single press will not, however, turn the cell pressure setpoint to zero. This is a failsafe in case the button is inadvertently hit. Depending on the system configuration the pressure may still fall, but ideally the pressure will be maintained. To force the pressure setpoint to zero the ***STOP SYSTEM*** button should be hit three times in 2.5 seconds, bearing in mind there is a debounce of the button between presses. This will immediately adjust the cell setpoint to zero. Bear in mind that it is not a recommended method for reducing the pressure to zero since it could discharge a large amount of fluid through the back pressure regulator and flow meter. However, in an emergency it may be the most appropriate method for dropping the pressure quickly. The **VENT** valve can also be opened manually to achieve the same result.

Region 13: Manually increase the drive air pressure by 1 psi.

Region 14: Manually decrease the drive air pressure by 1 psi.

Region 15: Touching this region will change the time/status window to a small data graph. This graph will display 300 seconds of data, and constantly updates in a trace mode. This is a convenient method for visualizing the temperature of the cell. While in graph mode pressing the same region will toggle back to the time/status window. Each time the window is toggled the data collection will start over.

Region 16: In the upper corner of all windows is a section that can be used to reset the screen. The **INTERFACE MODULE** is connected to the circulator via a long cable. This was done so the controls could be relocated to a more convenient location such as near the computer operating the NMR. There is no data flow control to the module so under certain conditions errant data may be displayed. This is not harmful to the operation. However, should it be desired touching this region will redraw the main screen. This is true for any of the windows. When the data entry keypad is displayed this region moves to the opposite side of the screen.

Zero Sensor Page:

The zero sensors page provides a mechanism for zeroing out the pressure sensors, flow sensor, and manually adjusting the room temperature reading of the

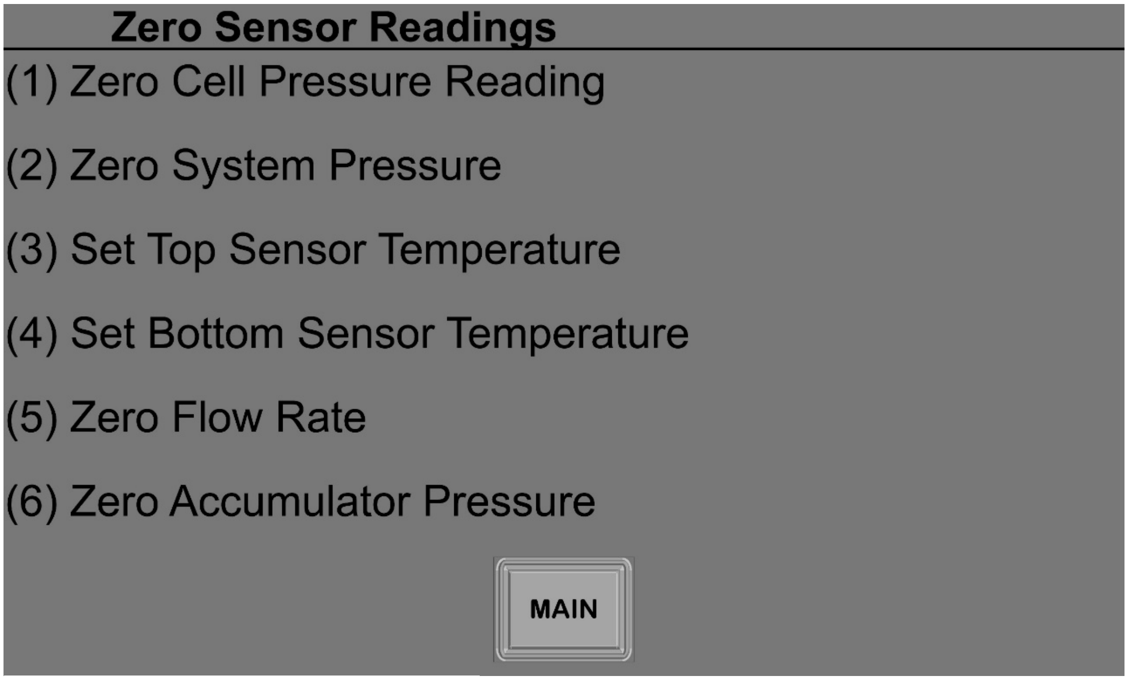

Figure 16: Zero sensor page

thermocouples. As shown in Figure 16 there are six options available on this page. Each option line of text represents a touch region. Options 1, 2, and 6 will zero the pressure reading of the corresponding sensors. For Option 6, this is usually only done when the system is initially setup and the charge on the accumulator is zero. The accumulator pressure can be changed manually through the *Other Functions/Additional Operational Parameters* menu. If the flow reading is not zero upon startup this too can be zeroed on this page.

Options 3 and 4 allow the thermocouple reading to be set to a specific value. Selecting these options brings up the keypad display for entering the appropriate data. The new value might from be a high accuracy thermometer reporting the room temperature or perhaps an alternate system such as the Omega TC-08 thermocouple device which can be used as a supplement data recording device. See the ***Thermocouple Setup*** section for more details. Once the data has been entered the controller will generate an offset value for the base reading. These offsets can also be changed using the *Other Functions/Thermocouple Parameters* menus. The ***MAIN*** button will return to the main screen.

Data Display Screen Page:

The data display screen page provides a mechanism for visualizing the temperature trajectory during operation. The screen is shown in Figure 17. The system has the capacity to store 10,000 data points for both thermocouples collected at a default one second intervals. After that period the data will revert to the start of the data array and begin to overwrite the data. If longer data collection is required the data interval can be changed using the *Other Functions/Additional Operational Parameters* menu.

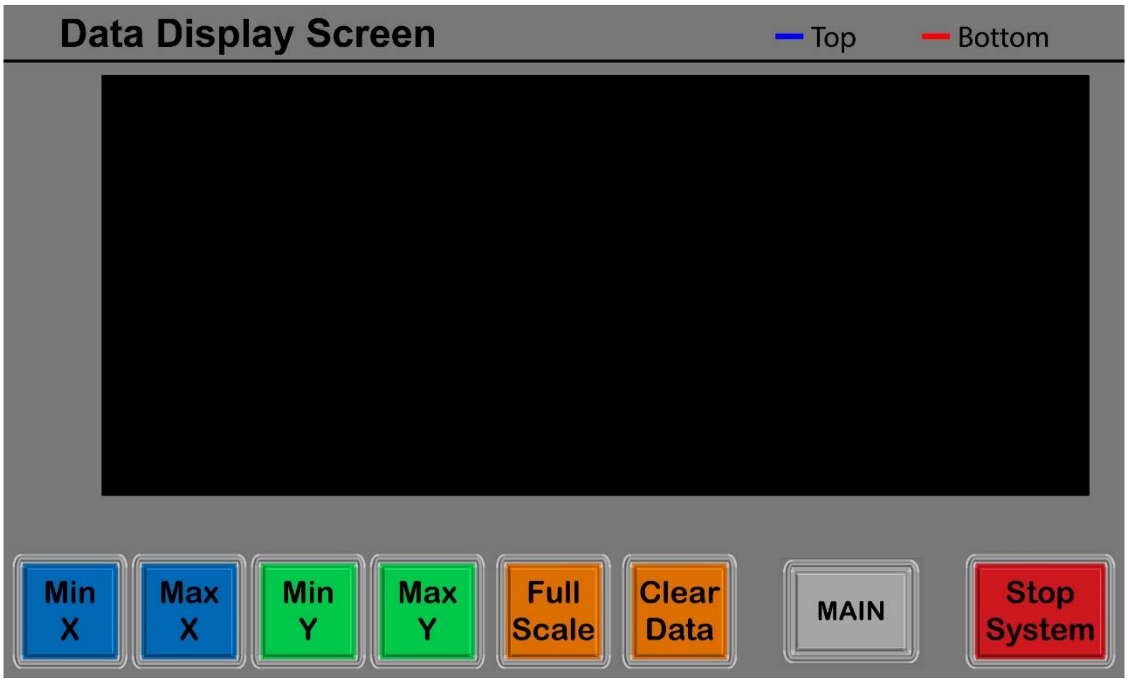

Figure 17: Data display screen

The first four buttons on this screen allow the minimum and maximum X and Y values to be redefined. This is useful for zooming in on the region to look at the variance in temperature once equilibrium is reached. The ***FULL SCALE*** button will redraw the full data set. The ***CLEAR DATA*** button will promptly zero the array and return to the main screen. There will be no confirmation message give. The ***MAIN*** button will return to the main screen.

The ***STOP SYSTEM*** button on this screen has the same functionality as the button on the main screen, in that it will stop the system. This is the only menu that has this additional option.

Other Functions Page:

The other functions menu page contains or provides options to set all the other parameters available to the user. Two of the most used parameters are available directly on the screen.

Option 1 allows the user to change the maximum system pressure. This can be useful to limit the maximum pressure of the system when using overburden cells rated to pressures below the maximum capacity of the system. It can also be used to allow the system to operate a slightly higher pressures on the accumulator side to achieve pressure in the overburden cell closer to the 10,000 psi maximum. This is allowable. However, it should be noted that at low temperatures where the accumulator pressure might be 1,000 psi or more higher than the overburden cell pressure, operating under these conditions might rupture the internal safety disc. Therefore, caution should be used whenever this parameter is set higher than 10,000 psi.

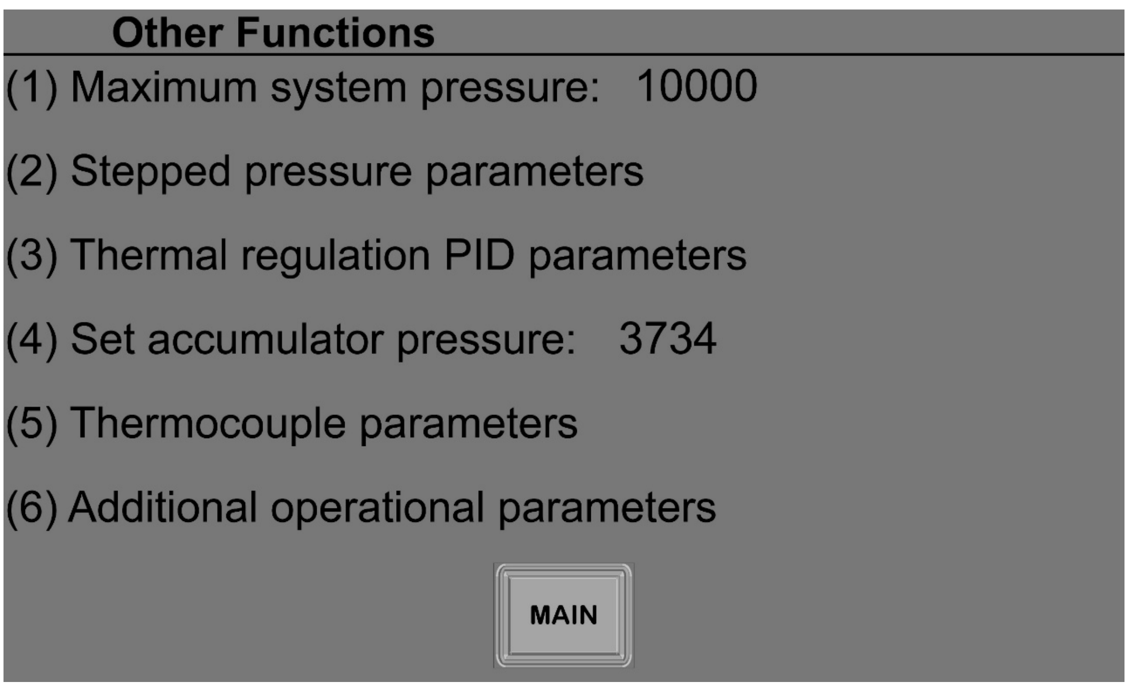

Figure 18: Other functions screen

Option 4, Setting the accumulator pressure, is the other commonly set parameter. When the system is first turned on the reading of the accumulator is recorded. The system operates differently when below versus above the accumulator charge pressure. Therefore, this value is recorded as the threshold when certain routines are called to govern the operation. However, if the accumulator charge is altered after the system is turned on this reading will not be accurate. Therefore, it should be changed manually by selection this option. Use the analog gauge in the **ACCUMULATOR CHARGE** section or the reading on the main screen for setting the parameter. The alternative is to toggle the system power to allow the system to record the accumulator pressure normally.

The other available options lead to additional menus which are described separately.

Stepped Pressure Parameters Page:

The stepped pressure feature provides a mechanism whereby controlled pressure steps are performed for increasing and decreasing pressure while maintaining reasonable flow characteristics. Primarily this is meant to assist for pressure changes above the accumulator charge, but it also provides some assistance below that threshold.

When the system first reaches the accumulator charge pressure the fluid that is ordinarily running through the heating coils and on to the overburden cell gets diverted to fill the accumulator. This causes the flow to drop to very low levels. With each increase the flow drops and slowly builds back as the accumulator fills. Moving in the other direction, when the pressure is decreased this built-up fluid discharges into the system. It can be especially pronounced for large jumps where the flow might drop to zero for several minutes when moving to higher pressures or might jump from 100 ml/min to over 500 ml/min for a short period upon a large decrease. This is not an optimal situation and could potentially damage the flow meter. Such large momentary changes in flow will likely cause problems with the constant flow routines leading to unusual performance until the system again stabilizes.

Therefore, to ease the burden on the user this feature can be used to gradually step the pressure, wait for the flow to return to some defined threshold, then perform the next pressure step. All the user needs to do is enter the target setpoint and the system will handle the rest, apart from possibly adjusting the drive air level periodically.

The screen shot for this menu is shown in Figure 19. Selecting **Option 1** will toggle the stepped pressure control on and off. The control provided by this section can be handled manually, and there may be situations where this is more appropriate thus the ability to switch it off.

Option 2: This is the step size for each pressure increment. When the pressure is below the accumulator pressure this parameter does not apply since changes in that range are fast. The default parameter of 250 psi works well for most system configurations.

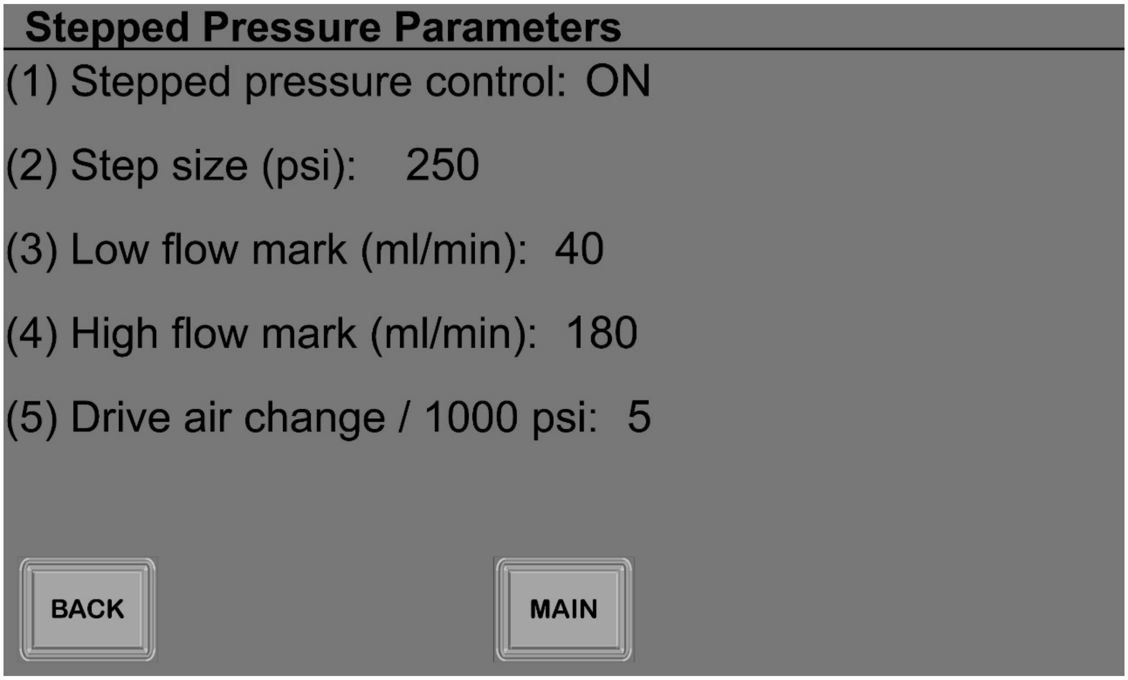

Figure 19: Stepped pressure parameters page

Option 3: This parameter defines the flow rate that must be reached before the next pressure step is made. This applies when the pressure step is an increase.

Option 4: This parameter is what the flow rate must be below before the next pressure step is performed. This parameter applies when the fluid is discharging from the accumulator during a pressure decrease.

Option 5: This parameter defines the rate of change made to the drive air with each increase. The drive air is coupled both to the flow rate and the maximum pressure the system can generate. Thus as the pressure is increased it is expected that the drive air pressure must also increase to maintain the same flow. The default parameter of 5 psi / 1,000 psi change means the drive air would change 1.25 psi / 250 psi step. This is a coarse parameter in that at the higher pressures the step size might be larger, but it provides a reasonable approximation of the needed increase that when the flow monitoring is turned on it helps limit the unexpected rapid changes that can occur.

As a special note, when the pressure is being decreased it is usually better to turn of the constant flow rate routine. The large flow rate changes that accompany the accumulator discharge can sometimes lead to over compensation. It is better to allow the stepped pressure routines to handle the drive air changes along with manual adjustments to keep the flow close to the expected levels.

Thermal Regulation PID Parameters Page:

The system uses an ideal, textbook version of a continuous-time PID controller to adjust the heat bath setpoint to lead to a stable sample temperature within the overburden cell. Parameters for the PID routine have been established that provides for rapid heating with a small overshoot in temperature before returning to the proscribed setpoint. The default parameters are shown in Figure 20.

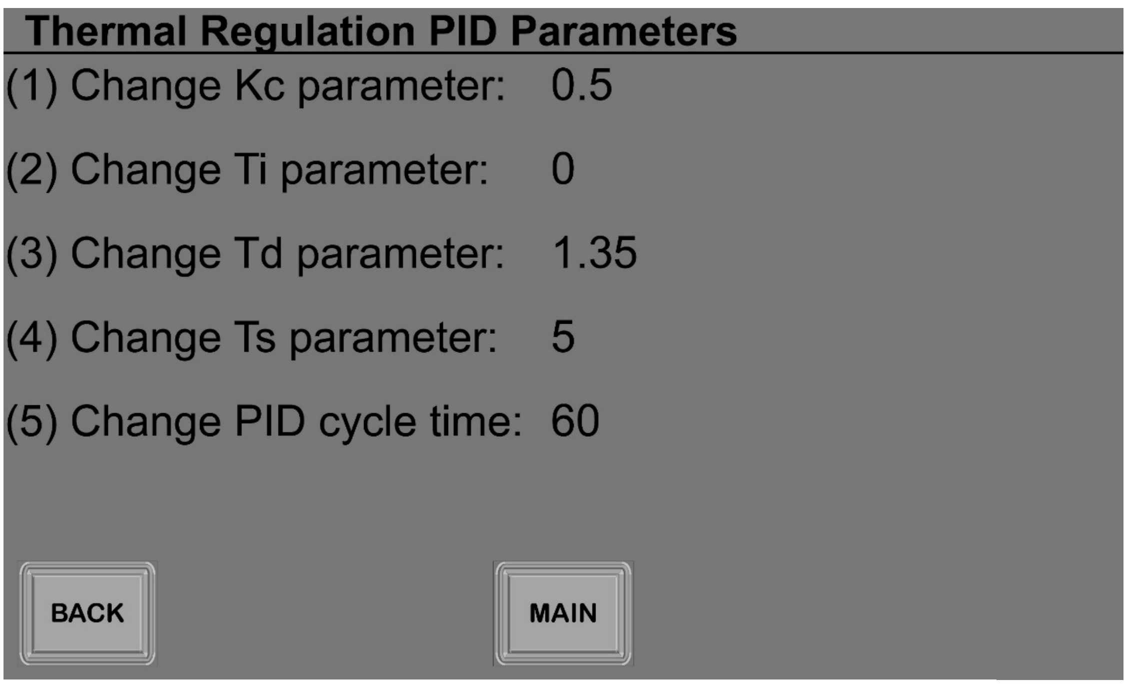

Figure 20: Default PID parameters for thermal regulation routine

Option 1, 2, and 3 are the proportional, integral, and derivative constants respectively. Option 4 is the sample period in seconds, and option 5 is the cycle time in seconds, or the period between changes to the heat bath setpoint.

A higher proportional constant will induce a more rapid increase in temperature within the limits of the ability of the heat bath to reach the target setpoint and the time required to transfer heat to the sample. It is thought that the proportional constant is already near the maximum that will yield a marked difference in performance due to the lag in heat bath response. The derivative constant governs the overshoot of the system. A higher number may decrease the magnitude of this overshoot, but may correspondingly increase the time required to reach the target. There is a large lag in changes to heat bath setpoint relative to changes in the sample temperature such that the integral portion of the PID routine has little impact. Therefore, the integral parameter is zero by default.

Thermocouple Parameters Page:

The thermocouples used in the circulator are J-type referenced to the NIST lookup tables. Once calibrated the output should be accurate to 1°C. This menu provides the mechanism to enter the calibrated slope for each thermocouple as well as adjust the offset established from the *Zero Sensors* menu. The current offset is displayed on this page. See the ***Thermocouple Setup*** for information on how to generate this information. The thermocouples can also be toggled on and off from this page. Toggling off serves the same purpose as unplugging the thermocouple, but using this route will also display the “off” message on the main screen. The system will use the output of one or two thermocouples for sample temperature maintenance.

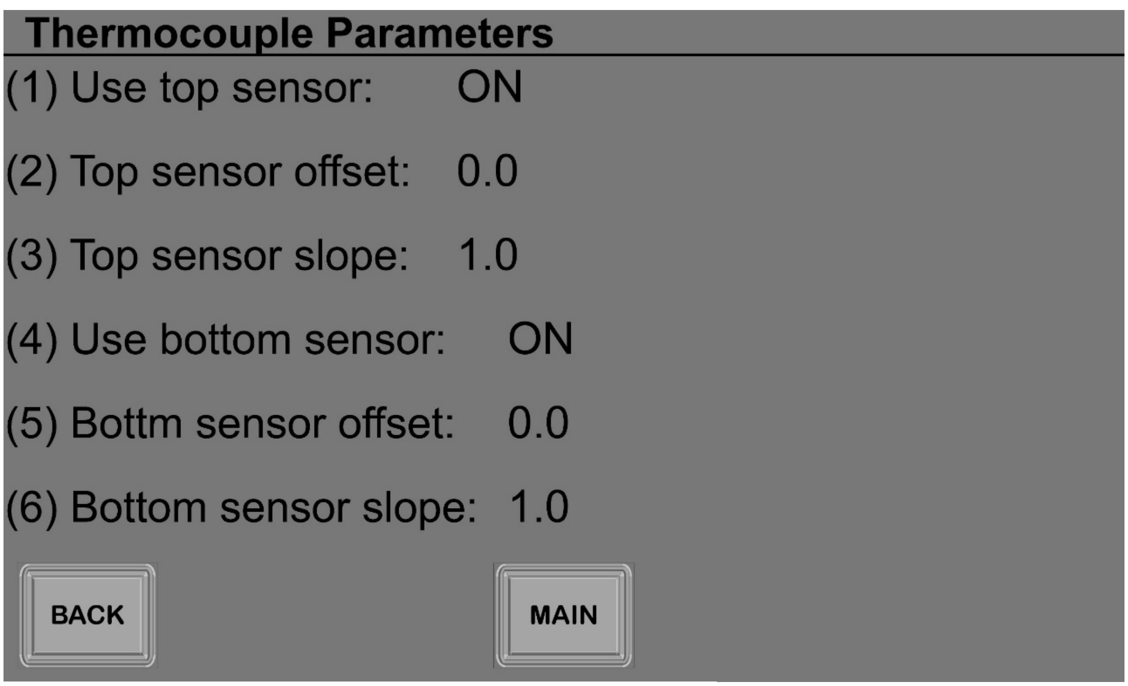

Figure 21: Thermocouple parameters page

Additional Operational Parameters Page:

The last menu option from the *Other Functions* menu is the *Additional Operational Parameters* menu shown in Figure 22. This menu contains a few additional parameters that are useful for defining the function of the circulator.

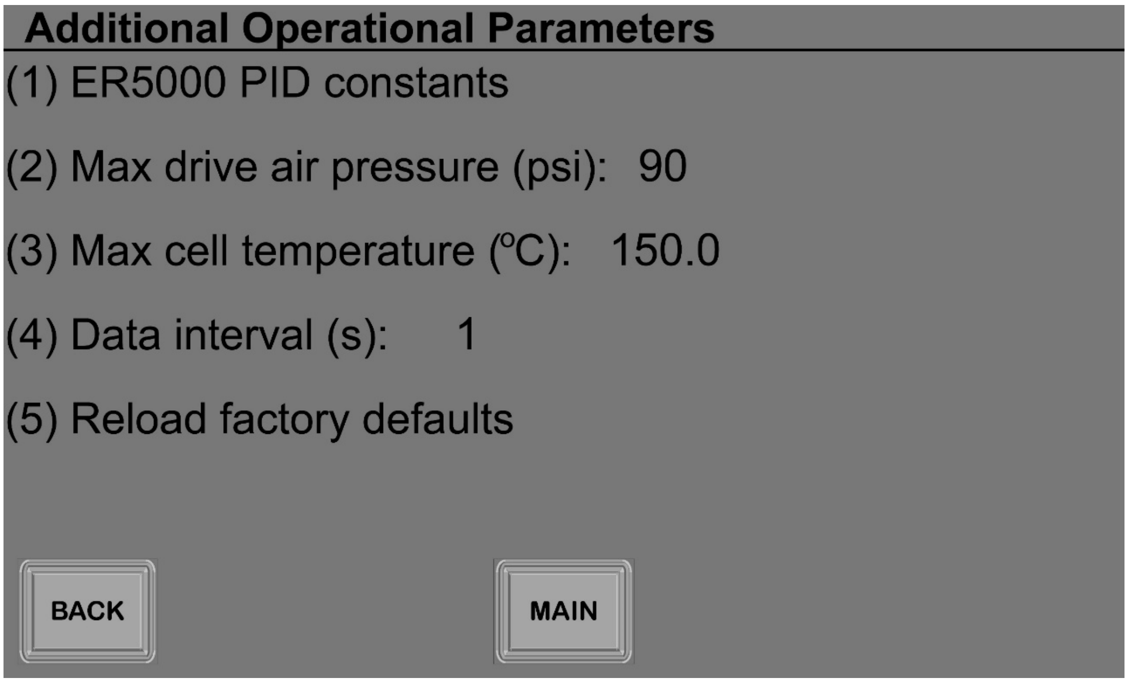

Figure 22: Additional operational parameters page

Option 1: This leads to *ER5000 PID Constants* menu that allows the user to change the PID constants used by the ER5000 controller to set the back pressure regulator. That menu is described elsewhere.

Option 2: This sets the maximum drive air pressure to put a ceiling on the flow regulation routines. The internal electronic regulator can handle pressure to 145 psi. However, most compressed air systems will not deliver pressure at that level or do so only for a very short time. It the circulator is attempting to reach a certain flow rate at high pressures it will continue adjusting the drive air pressure higher even though it is not altering the flow performance. Limiting the maximum pressure to that point where no further changes in flow can be detected minimize unnecessary air consumption and can lower the duty cycle of the air compressor.

Option 3: This allows the maximum cell temperature to be set higher than the rated maximum of the cell. There is typically a temperature gradient across the sample with the thermocouple closest to the confining fluid inlet being higher than the exit side. When both thermocouples are present the circulator will use the average as the current temperature. As an example the inlet hot side thermocouple might read 151.5°C with the outlet cool side reading 148.5°C for an average of 150°C. There may be reason to run with only a single thermocouple such as when flow experiments are performed. If it was the hot side thermocouple only in use the setpoint could then be set to 151.5°C to achieve the desired sample temperature of 150.0°C. This assumes that all other characteristics are equal.

Option 4: This parameter changes the interval between data points for the thermocouple data displayed on the *Data Display Screen.* This does not alter the data interval for the trace graph that can be accessed from the *Main Screen.*

Option 5: If there is need to reset the circulator to the factory settings that can be done with this option. It will overwrite all the stored offset parameters, including those for the thermocouples, so it should not be used unless operation has become unstable.

ER5000 PID Constants Page:

The Tescom ER5000 controller uses a PID routine, coupled with the feedback from

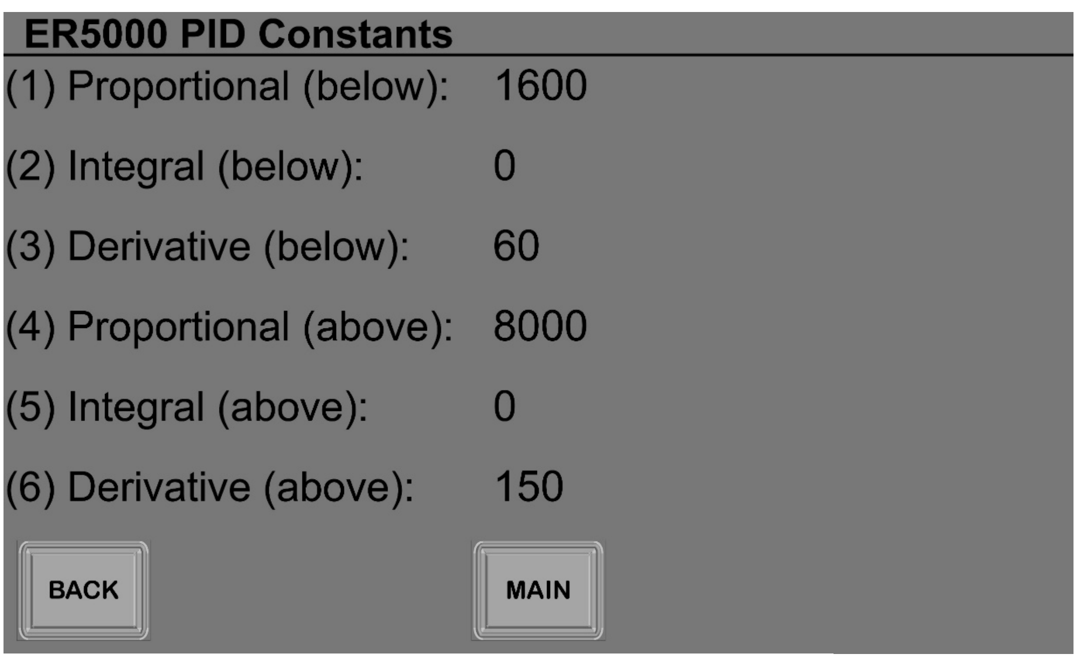

Figure 23: ER5000 PID constants page

the pressure sensor, to set the supply air pressure to the back pressure regulator for maintenance of the pressure setpoint. These parameters are made available in the menu page shown in Figure 23. Due to the presence of the accumulator absorbing fluid from the circulation circuit there are two decidedly different profiles used. One is for pressure points below the accumulator charge and the other set for pressures above the accumulator charge. Both sections can be changed within this menu. As is the case with the thermal regulation routines there is a significant lag that renders the integral component largely ineffective in this routine so is zero by default.

In general these parameters should not be changed. The one exception might be when the system fills the overburden cell and air is driven through the back pressure regulator. This loss of flow can create and unusual phenomenon that may require changing these parameters to correct. See the *Troubleshooting Pressure Regulator* section.

CELL PRESSURE SENSOR SETUP

The cell pressure sensor is external to the circulator, but is essential for the proper function of the circulator. It provides the feedback at a specific point in the circulation loop that feeds back to the ER5000 controller to set the pressure at that specific point in the circuit. The accumulator pressure reading, when above the charge pressure reports on the pressure of the fluid leaving the circulator. This fluid then travels through the heating coils and other tubing to reach the overburden cell. The heating coils contain around 40 feet of tubing so there is a considerable pressure drop across the coils from the point where the fluid leaves the circulator to the external pressure sensor. The return system pressure is that of the fluid returning to the circulator. The pressure in the overburden cell will lies somewhere in between the external sensor and the return pressure.

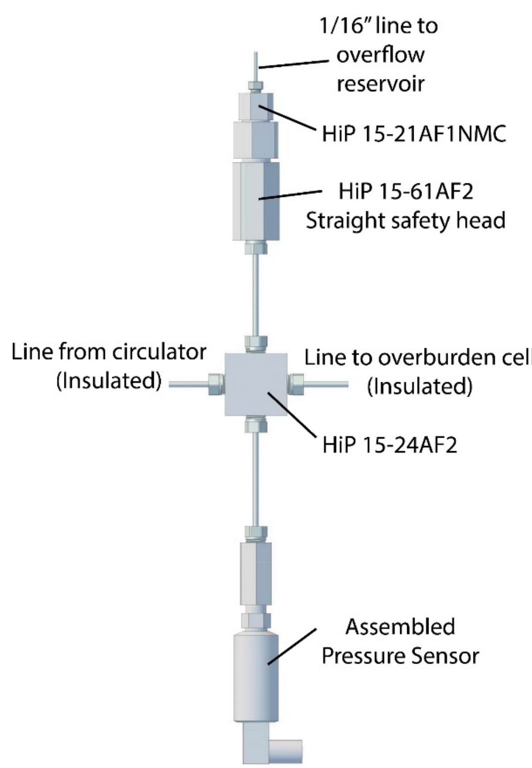

Figure 24: Pressure sensor cross configuration

The idea then is to place the cell pressure sensor as close to the overburden cell as is practical. The sensor can be a couple of feet away without much pressure drop expected from the reading to the actual cell pressure, but closer is going to provide a more accurate reading. In addition to the pressure sensor a safety head component with suitable rupture disc should be placed on the inlet side of the overburden cell. The rupture disc should be rated at 125% of the rating of the overburden cell. When used with the 10,000 psi cell the safety head is not strictly necessary since the circulator itself has a 12,500 psi rupture disc inside that will fail before a rupture disc placed at this point in the confining fluid loop.

One example configuration is shown in Figure 24 with both the sensor and the safety head split from a cross in the confining fluid loop. It is important to provide a minimum 6” connector tube between the cross and connection to the sensor or safety head. This is especially important for the safety head where the rupture disc rating is highly dependent on the temperature. Due to the heat transfer to the safety head body a direct connection to the cross would cause a premature failure of the rupture disc. The tubing to both can be bent if necessary.

The outlet from the safety head should be piped into an overflow reservoir capable of holding high temperature fluid with a capacity to accept the full contents of the circulator. A large 5 liter glass bottle would be appropriate.

The 1/8” tubing to the cross and away from the cross to the overburden cell should be insulated. Included with the circulator are lengths of 1/4” ID Viton insulation for this purpose. An additional supply of 1/8” ID x 1/4” OD silicone tubing can be over the tubing first followed by the Viton tubing. This can be difficult to do especially if there are bends in the tubing. It is likely just the Viton insulation will be sufficient for most applications. There is also a small supply of adhesive backed silicone rubber that can be cut and fixed to the cross for some insulating effect and protection to the user from very hot parts. The lines to the safety head and pressure sensor do not need to be insulated.

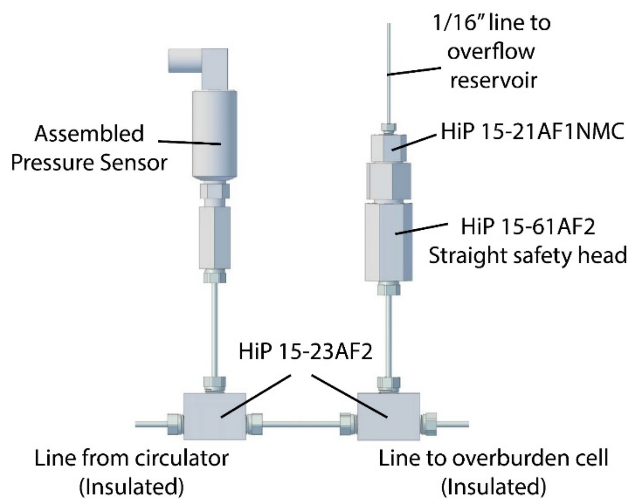

Figure 25: Pressure sensor tee configuration

A second option for connecting the

pressure sensor is shown in Figure 25. Instead of using a cross to connect both, separate Tee junctions are used. This may provide a bit more flexibility in the placement of components. Though it is drawn with the safety head closer to the overburden cell, it can in fact be between placed somewhere between the bulkhead panel and the overburden cell without significantly affecting the protection offered by the device.

As was the case for the first configuration insulation should be used on the confining fluid lines and silicone sheeting used to cover the T-junctions. The lines to the sensor and safety head should not be insulated.

Connections of the confining fluid lines to the overburden cell can be made with 90° bent 1/8” tubing connected to straight couplers or using high pressure elbows included with the shipment. The hot inlet side components will be very hot so some measure of protection or indication to the user should be present as a warning.

OPERATING INSTRUCTIONS

The following instructions assume that the circulator is being used with the equipment cart equipped with the gas booster. It provides guidelines for using it to the system to regulate the pressure and temperature of the overburden sample.

A guiding principle for building up the temperature and pressure of the overburden cell is to first raise the temperature of the cell, with minimal pressure, then ramp up the pressure to the target experimental conditions.

The ***DRIVE AIR*** supplied to the electronic regulator controls both the maximum pressure that can be generated by the air-drive pump, but also controls the rate of flow. The maximum pressure obtainable is simply the regulator setpoint multiplied by 220, but the back pressure regulator sets the actual pressure in the system. Any air flow delivered above the level required to meet operating pressure of the system will increase the cycle rate and thus flow in the system. Decreasing the setpoint of back pressure regulator will also increase the rate of flow unless the ***DRIVE AIR*** pressure is reduced to match the desired flow. Therefore, the ***DRIVE AIR*** and back pressure regulator settings are coupled and often require iterative changes to reach a stable operating condition when manually controlled.

The operation should proceed as follows:

Step 1: Upon turning on the circulator and support equipment the system will start with the operational parameters at zero. The heat bath setpoint will also read zero. This indicates the heat bath has not yet received a new setpoint so will automatically move to the setpoint last used. This may not be appropriate for the current experimental conditions so it should be changed upon power-up. Having the heat bath start at a higher temperature will also decrease the time required to reach the target sample temperature. If the target sample temperature is below 100°C the heat bath should be initially set 15°C above the target sample temperature. When the target is around 125°C it should be 20°C higher and at 150°C the setpoint should be 25°C higher. Once the temperature adjustment routines are activated the appropriate temperature will be set, but it will save time if the heat bath is already close to that point.

Check that the **VENT** valve is open to allow the flow to initially divert back to the reservoir. Also confirm that the bypass valve on the heat bath tubing manifold is open. This diverts fluid away from the heating coils. The cool side water bath should be switched on at set to 30°C.

> Do not operate the circulator at high temperature without the water bath supplying the cool side heat exchanger running. High temperature fluids will damage the air-drive pump.

Check **RESERVOIR FLUID LEVEL** to be sure there is sufficient fluid in the system for the experiment. Finally be sure the **E-STOP** valve is closed (pushed-in). The electronic regulator being set to zero will also block the flow, but it is good practice to keep that valve closed to prevent compressed air from unexpectedly starting the flow when it is not expected.

Step 2: Check the nitrogen gas charge on the accumulator. The gauge within the **ACCUMULATOR CHARGE** box reports on this value. The nitrogen gas charge should be at the very minimum of 25% of the maximum pressure of the experiment to a maximum of 60% of the maximum working pressure for the experiment. If this is not the case see the *Gas Booster Specifics and Operation* section for instructions for building up the appropriate charge.

Step 3: Check to be sure the nitrogen gas supplying the internal air-actuated valve is set to 90 psi. Open the main compressed air valve to start the flow of air to the system. At this point the system is primed to operate so all external high pressure connections should be verified.

Step 4: Open / Pull the **E-STOP** knob to open the flow of compressed air and nitrogen to the system. Assuming the ***DRIVE AIR*** value is still zero the system should not yet be operating.

Step 5: Slowly increase the ***DRIVE AIR*** pressure until the pump start to operate. The pump may initially need 15-20 psi to start operation, but it should then be dialed back to 8-10 psi.

Step 6: Close the **VENT** valve. The ER5000 controller does not perform well with no flow. When the **VENT** valve is closed it forces fluid to the back pressure regulator and will cause the ER5000 to resume control. This action will cause a momentary spike in the pressure. It may rise as high as 2,500 psi before settling back to a few hundred psi which is the back pressure in the system. It will not fully return to zero pressure when there is flow through the system. Perform an initial survey of the system to assure there are no leaks.

Step 7: Manually increase the ***DRIVE AIR*** until the flow is close to the desired rate. Good heating occurs when with the flow around 125 ml/min. It can be higher and perhaps improve the thermal gradient across the sample. At very high pressures the circulator will consume a large volume of air so it may not be possible to run at higher flow rates without a suitable air compressor delivering sufficient air.

Once the flow is close to the target activate the constant flow routine by touching the greyed out ***ACTIVE*** button within the flow rate section. It will turn green and start adjusting the ***DRIVE AIR*** to hold the flow constant.

During this process monitor the gauge in the **ACCUMULATOR CHARGE** box to assure pressure is not just building in the accumulator. This can happen if the nitrogen gas is not supplied. The ER5000 will still allow flow if no air is provided, however the internal air actuated stop valve will not be open. Thus the all the fluid will be diverted to the accumulator. With such low air draw at this point it will probably not be able to reach very high pressure. However, it is possible to rupture the safety disc if the system is started and it is not noticed that the pressure is building only in the accumulator.

Once flow of the confining fluid is initiated, but still at low pressure and temperature, validate all the connections in the system to assure there are no leaks.

Step 8: The integrity of the system can be checked prior to increasing the fluid temperature of the confining fluid. This step is good practice to minimize problems that might occur later in the procedure, but it may not be permissible for certain samples. It does not require the pressure of the fluid to the core be increased. The confining fluid can be at maximum with the core fluid at ambient pressure provided the sample permits it and the system setup is sound.

Do not enter a new pressure setpoint and walk away from the instrument. Always monitor the response of the system.

Therefore, this step is optional. As confidence in the setup of the overburden cell and system connections develops it may not be necessary to perform a full pressure tests. However, increasing the system pressure to something below the accumulator charge pressure does not take long and will help find potential leaks while the fluid is still close to room temperature.

To increase the pressure enter the new setpoint in the cell pressure window and assuming the stepped pressure function is active, the system will, by itself, run the pressure to the desired setpoint. It may be necessary to occasionally modify the ***DRIVE AIR*** pressure to remain consistent with the flow. It is suggested that the steps be on the order of 2,000 psi to allow pauses for system integrity checks. If pressure does not build see the *Troubleshooting Pressure Regulator* section.

Step 9: Once the system connections have been validated the pressure can be reduced. Due to the large discharges from the accumulator during depressurization it is suggested the constant flow routines be switched off. When properly established the stepped pressure routines handle the necessary reduction in ***DRIVE AIR*** pressure with system pressure decreases better than the constant flow routines which are better at constant pressures.

Reduce the pressure in the system to 25% (or lower) of the maximum overburden cell rating to prepare the system for the temperature increase. The pressure at which the thermal regulation operations are performed can be one of user preference. The overburden cell can withstand heating over the full range while at 25% of maximum. It can be lower, but probably should not be much below 1,000 psi. There should be some pressure in the system to force closed any voids remaining in the cell so there is equal distribution of heat to the cell housing. It is also likely that the fluid to the core will be introduced at this point to immerse the thermocouples in the working fluid. This will likely involve some pressure increase so there needs to be some pressure in the confining fluid to maintain the integrity of the seals on the core sample assembly. The manual for the overburden cell should be consulted for the proper pressure differential between the confining fluid and fluid in the core sample.

The confining fluid pressure should be at least 300 psi higher than the pressure of the fluid delivered to the core. This allows a positive pressure on the core assembly sealing rings to prevent leakage.

Step 10: Configure the system for the thermal regulation routine. Enter the temperature setpoint on the main screen. Be sure the thermocouples are connected or toggled off if not in use. There must be one thermocouple in use for the system to work.

The initial heat bath setpoint should have been set in Step 1. It is assumed the bath temperature is close to or at that setpoint. It is not required that the temperature be at that level, but the heat up of the sample will be faster if there is less wait time for the bath to reach temperature.

The data collection routines are running from the moment the circulator is turned on. Therefore, there may be a significant amount of data that has no relevance to the current experiment. Hit the ***DATA SCREEN*** button from the main screen and then hit the ***CLEAR DATA*** to reset the data collection to time zero.

The heating coil bypass valve on the heat bath manifold can be closed to force fluid through the heat transfer coils immersed in the bath.

The cool side bath must be turned on prior to sending high temperature fluid back into the circulator. Failure to do so could damage components within the system and potentially render it inoperative.

Step 11: Heating of the sample is initiated with the close of the bypass valve. To initiate thermal regulation of the sample simply hit the ***START*** button. The indicator in the temperature section will turn indicator green. If the constant flow is not already active this too will activate and attempt to maintain the flow at the current setpoint. Certain heat bath routines will be activated, but the indicator within that section will not necessarily turn green until much later in the process when the system becomes more stable.

Generally it will take about 30-40 minutes to heat the sample from room temperature to near 150°C. As expressed elsewhere it is recommended that the overburden cell be heated first at low pressure, followed by increasing to operational pressures. This is to allow the system to achieve a sort of thermal equilibrium, and allow all the associated changes that occur with heating to happen, prior to subjecting the cell to additional forces.

However, it is not necessary to wait until the cell reaches stable equilibrium before pressure changes be initiated. It is sufficient merely to wait until the temperature is close to the target. For example, with a setpoint of 150°C, pressure changes can be initiated starting when the temperature reaches 135-140°C. There is still about the same period of time remaining as took to reach that point before the temperature is stable. Increasing the pressure of the confining fluid will have little impact on the time to equilibrium.

It is important to note that when fluid is injected into the core this fluid will likely not be at the same temperature as the core. This will cause the corresponding thermocouple to experience momentary temperature fluctuations. This will influence the temperature adjustment routines to a degree. Fortunately, the long cycle period of the PID routine means the output will not be duly impacted. Executing the confining pressure increases while simultaneously increasing the injection fluid pressure during the later phases of thermal regulation will improve the efficiency of the process such that about the time the cell reaches thermal equilibrium the pressure will also be close to or at the intended target.

Step 12: Once the experiment is complete the overburden cell needs to be cooled and the system pressure brought back to zero. Related to those is the necessity to reduce the fluid pressure in the core sample itself, but that is not addressed in these instructions since the circulator is not part of that circuit.

The toggle button with the temperature section can be hit turn switch off the tracking routines. **DO NOT HIT THE STOP SYSTEM BUTTON.** Doing this will stop all the regulating routines, but will also cut the ***DRIVE AIR***. This can be manually restored if necessary, but it has the potential to cause a rapid drop in pressure which may cause an internal rupture of the core sample assembly inside the overburden cell due the now unfavorable pressure differential with that of the fluid in the core. The process of setting up the core sample in the overburden cell would likely need to be performed to restore integrity of the seals in the core sample assembly.

The heat bath ***ACTIVE*** button, can also be toggled to turn off those routines as they are no longer needed. The heat bath setpoint can be reset to room temperature or simply turned off.

To more rapidly cool the overburden cell the bypass valve on the heat bath manifold can be opened to divert fluid away from the heating coils. The confining fluid will now be used to draw heat from the overburden cell and cooled by the cool-side bath.

While the cooling of the cell is ongoing, a certain level of pressure should be maintained in the overburden cell. This need not be more than 1,000 psi. The suggested method for reducing the pressure is to do so with the constant flow rate routines turned off. The stepped pressure routines have the capacity to keep the ***DRIVE AIR*** close to the appropriate level to roughly maintain the same flow. Therefore, toggle off this feature. Enter the new pressure setpoint and upon hitting ***ENTER*** the pressure will programmatically be dropped to the desired point.

The flow should be allowed to continue until the reported cell temperature is around 50°C. This is low enough that the cell can be touched without consequences. Once at this point the pressure can be further reduced to the zero point.

The **STOP SYSTEM** button can be pressed to cut off the **DRIVE AIR** to the pump. Any remaining pressure will slowly dissipate. Open the **VENT** valve to fully depressurize the system.

To finish, push in the **E-STOP** valve to cut the air and nitrogen flows to the circulator. The gas valves can be turned to the closed position, and the cooling bath and any other powered devices can be switched off.

TWO PAGE OPERATION INSTRUCTIONS

These instructions assume the system has just been turned on and it is connected to the cell. It also assumes the default operation routines such as the stepped pressure routines are active.

Step 1: Return system to low pressure state and check operational status.

Perform the following:

a) **VENT** valve open
b) **E-STOP** valve in stop position (pushed-in)
c) Confirm the pressure setpoint and drive air are set to zero
d) Confirm that the bypass valve on the heat bath manifold is open
e) Check that the thermocouples are connected
f) Heat bath should be set to ~20°C above the desired cell temperature
g) Turn on the cool side bath and set to 30°C

Step 2: Verify accumulator charge is appropriate for the maximum experimental pressure of the system (25-60% of the maximum experimental pressure).

Step 3: Initiate flow of air/ nitrogen gas to circulator

Step 4: Open **E-STOP** valve

Step 5: Increase ***DRIVE AIR*** pressure until pump starts. Set to around 8-10 psi.

Step 6: Close the **VENT** valve. The pressure will momentarily spike then fall back.

Step 7: Increase the ***DRIVE AIR*** pressure until the flow is close to the desired setpoint (125 ml/min is appropriate) then activate the constant flow routine. The button should turn green.

Step 8: Change the setpoint to the maximum operating pressure and allow the system to run to pressure. Check for leaks. It may be necessary at times to manually adjust the ***DRIVE AIR*** to maintain flow. If the pressure does not build see the *Troubleshooting Pressure Regulator* section.

Step 9: Reduce the setpoint to 25% of the overburden cell maximum rating (lower if the experiment requires) to prepare the system for the temperature increase.

Step 10: Configure the system for thermal regulation. It is assumed that the bath has reached the initial setpoint from Step 1.

Perform the following:

a) Change the temperature setpoint to the desired target.
b) Clear the data on the *Display Data Screen*
c) Close the bypass valve on the heat bath manifold to divert flow through the heating coils which will initiate sample heating.

Step 11: Hit the ***START*** button. This will activate the indicator button in the thermocouple section, and will also activate the heat bath routines. The latter will not turn green until later in the process. Allow the temperature to get within 10-15°C of the target temperature before increasing the pressure setpoint to the experimental level.

Step 12: After completion of the experiment return the system to the low pressure, low temperature condition.

a) Hit the ***ACTIVE*** button in the thermocouple section to turn off the temperature adjustment routines. **DO NOT HIT STOP.**
b) Open the bypass valve on the heat bath manifold to bypass the heat coils.
c) The heat bath can be switched off at this point. The ***ACTIVE*** button within this section can be toggled off.
d) Reduce the pressure setpoint to 1,000 psi for the cooling phase. The core pressure should be reduced correspondingly. This pressure reduction should be done in increments of 2,000 psi to allow all fluid discharging from the accumulator to escape at the end of each increment. The ***DRIVE AIR*** can be manually adjusted or the constant flow routine can be allowed to work. Often it is better to switch off this feature when decreasing pressure.
e) Allow the sample to cool to 50°C
f) Further reduce the setpoint to zero. Reduce the ***DRIVE AIR*** if necessary.
g) Hit ***STOP SYSTEM*** to fully cut the ***DRIVE AIR*** to the pump.
h) Open the **VENT** valve to eliminate any pressure remaining in the system
i) Push in the **E-STOP** knob to cut air flow
j) Turn off cooling bath
k) Switch off air/gas supplies

THERMOCOUPLE SETUP

The thermocouples used with this system are ungrounded J-type thermocouples. The output is amplified and converted to a voltage suitable to be read by the system. The output from thermocouples is not linear so to enhance the accuracy of the output it is compared against NIST tables to yield an accuracy of 1°C.

To achieve this level of accuracy the offset and slope of the internal amplifier must be determined. The offset of the thermocouples can be set using the ***ZERO SENSORS*** page. On this page the actual temperature from a reliable thermometer can be entered as the baseline point. The slope can be calculated by placing the thermocouples in the heat bath and collecting a series of data points where the actual versus reported measurement is recorded using the baseline value to establish the intercept. These values can then be used to obtain and average variance to yield a slope of the amplifier. The slope values can be enter on the menu *Other Functions/Thermocouple Parameters.*

One method that has been employed for providing high accuracy feedback is to use an external data acquisition module. The circulator was shipped with thermocouple tees that can be used to simultaneously plug the thermocouples from the overburden cell into the circulator and an external device. One possible device is the Omega TC-08 USB Thermocouple Data Acquisition Module. This device connects to a PC and can provide a high accuracy readout of the thermocouple outputs which can be used to set the baseline as well as the slope for the thermocouple. An added benefit is that because it is plugged into a computer with sizeable storage capacity larger data sets can be recorded by the computer and stored for later analysis.

NOTE: It has been found that some laptops use a USB controller that may cause grounding issues leading to erroneous results. To correct for this use the **USB Grounding Cable** connected to another PC USB port with the DB9 end plugged into the **SERVICE** port on the back of the circulator. This eliminates any grounding loops and will provide more accurate results.

TROUBLESHOOTING PRESSURE REGULATOR

When the circulator is first powered up the internal ER5000 controller starts in a no flow condition. In cases where the system lines are mostly filled with fluid the controller will resume proper operation once measurable fluid flow restored. However, when the system lines are filled with a large amount of air, or when the system is used to fill the overburden cell large volumes of air are forced through the regulator. This is not an ideal condition for the regulator and may induce a condition where the ER5000 will not behave as expected in that it will not build pressure even though it appears there is sufficient nitrogen air and liquid flow. This is not a failed component and careful analysis of all the operational variables governing the ER5000 have revealed nothing unusual such as a trapped PID routine yielding errant results.

This condition can be readily cleared by rapidly forcing a large volume of fluid through the regulator to clear any voids present in the pressure regulator as well as clear any debris that may be present. This can be achieved by building fluid pressure within the accumulator without allowing the fluid to discharge through the internal normally-closed air-valve. Then by actuating the internal valve the fluid rapidly discharges from the accumulator and through the pressure regulator. It may be useful to refer to the block diagram shown in Figure 1.

Read through the entire procedure first to understand the steps required.

Step 1: Reduce the **DRIVE AIR** and **SETPOINT** to zero.

Step 2: Turn off the nitrogen gas supply. This condition has been previously noted as having the potential to create an overpressure condition. That remains true for typical operation. It is important that once this procedure is initiated that it be followed to the end so the system is not left in an unexpected configuration.

Step 3: Close the **E-STOP** valve (push-in). This will allow nitrogen gas in the system to discharge. It is necessary to reduce the nitrogen gas pressure to zero to allow the internal valve to close. This may require toggling the E-STEP four or five times (more if the length of nitrogen gas tubing feeding the circulator is long). The alternative is to vent the pressure from the cylinder side. The objective is to be sure there is no nitrogen pressure available to actuate the internal valve.

Step 4: Increase the **DRIVE AIR** so the pump is operating. The objective is to build an additional few hundred PSI of pressure in the accumulator so keep a close watch on the pressure reading either on the analog gauge or touch panel module. The pressure build-up is gradual so there is not much danger of going too far. However, remember that hitting the **STOP SYSTEM** button will immediately cut the **DRIVE AIR** to zero stopping further build-up. Also, opening the **VENT** valve will allow the accumulator to vent to the reservoir if immediate pressure release is required.

Step 5: Once the pressure is a few hundred PSI above the starting accumulator charge, restart the flow of nitrogen gas. This will immediately actuate the internal air-valve and allow the excess fluid in the accumulator will discharge through the regulator.

Step 6: The flow reported on the touch pad module may exceed 500 ml/min. This will gradually drop to a level corresponding to the current **DRIVE AIR** setting. At this point the **DRIVE AIR** can be reduced to a more appropriate level. The ER5000 should now respond to pressure setpoint changes.

As noted as the start of this section, the unresponsive ER5000 condition normally occurs when the system contains a large volume of air. This often happens when the system fills the overburden cell or when primed. The frequency of this occurring may be reduced by placing a T-junction in-line with the returning line from the overburden cell. The open trunk of the junction is terminated with a valve and tubing leading to a collection vessel. During the fill process the setpoint is set to an elevated pressure which will induce the ER5000 to generate a load on the pressure regulator. This will effectively block flow into the circulator and it will divert through the open valve into the collection vessel.

NITROGEN MANIFOLD INSTRUCTIONS

The circulation system requires the delivery of low pressure as well as periodic high pressure nitrogen gas. This can be done with two nitrogen cylinders or a special manifold can be used to allow a single cylinder to be used for this purpose. However, the nitrogen gas pressure that is supplied to the gas booster is typically much higher than is allowed for the mini nitrogen gas regulator on the **MAIN AIR HANDLING PANEL**. If gas at this pressure is supplied to this regulator it could destroy that manifold as well as the internal air-actuated valve in the circulator. Shown in Figure 26 is a block diagram and parts for one manifold design that can be used for both low and high pressure nitrogen gas connections.

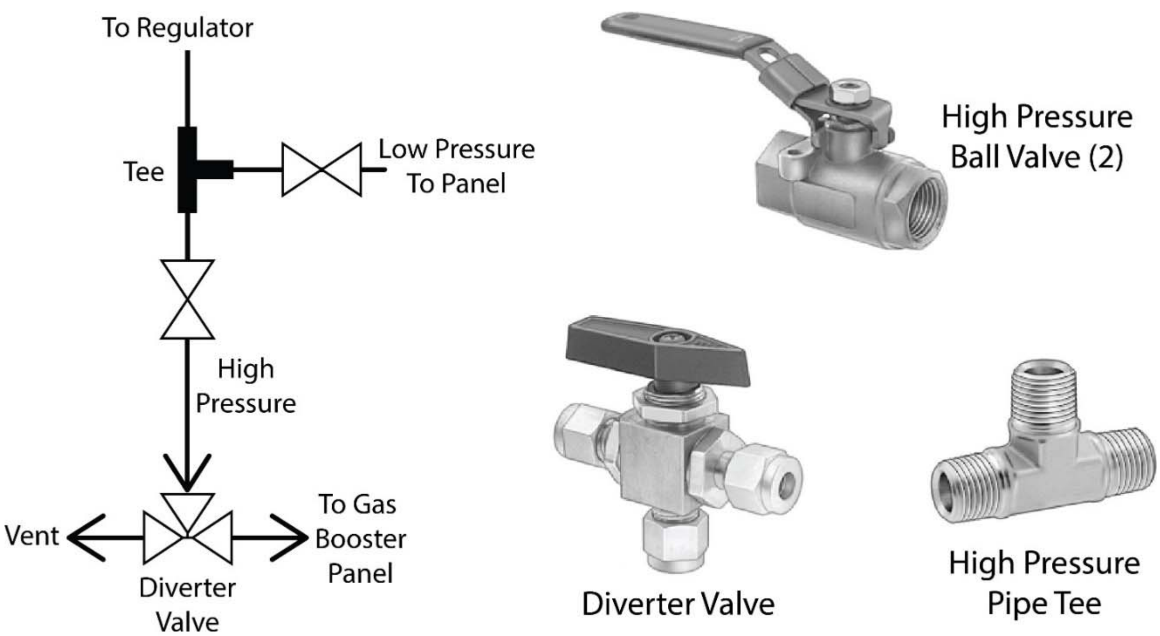

Figure 26: Nitrogen gas manifold block diagram and parts

Nitrogen gas is supplied from the regulator to a high pressure pipe tee which feeds to two high pressure ball valves. Both valves must be rated to the maximum outlet pressure supplied to the system. When the valve is opened to the low pressure side the regulator setpoint must not exceed 150 psi. This is not sufficient for the gas booster. Therefore, when charging the gas booster this low pressure valve must be closed.

> Never open the valve to the low pressure nitrogen line with the regulator pressure above 150 psi. This will destroy the downstream components.

The pressure for charging the gas booster can be 500 – 2000 psi. This is much too high for the low pressure side so the pressure must be vented after completion of the charging operation. This can be achieved by the diverter valve connected in-line from the manifold to the **GAS BOOSTER AIR PANEL**. Rotating the knob to one extreme allows passage to the **GAS BOOSTER AIR PANEL** and the other extreme will vent the pressure. The high pressure must be vented and the nitrogen regulator reset to 150 psi prior to opening the valve to the low pressure line.

REPLACING THE INTERNAL RUPTURE DISC

If the pressure in the system increases to 12,500 psi the rupture disc in the internal safety head will fail and vent fluid to the reservoir. Typically the confining fluid line also has a lower rated rupture disc in-line with the cell that will break before this disc. For the circulator rupture disc to fail there needs to be an artificial block in the line. Either a valve was installed downstream from the outlet of the circulator restricting flow or the internal air-actuated valve was not open. This condition can occur if the nitrogen gas supply to this valve was not on or at a pressure sufficient to open the valve. Once this failure has occurred the system will not build pressure until it is replaced.

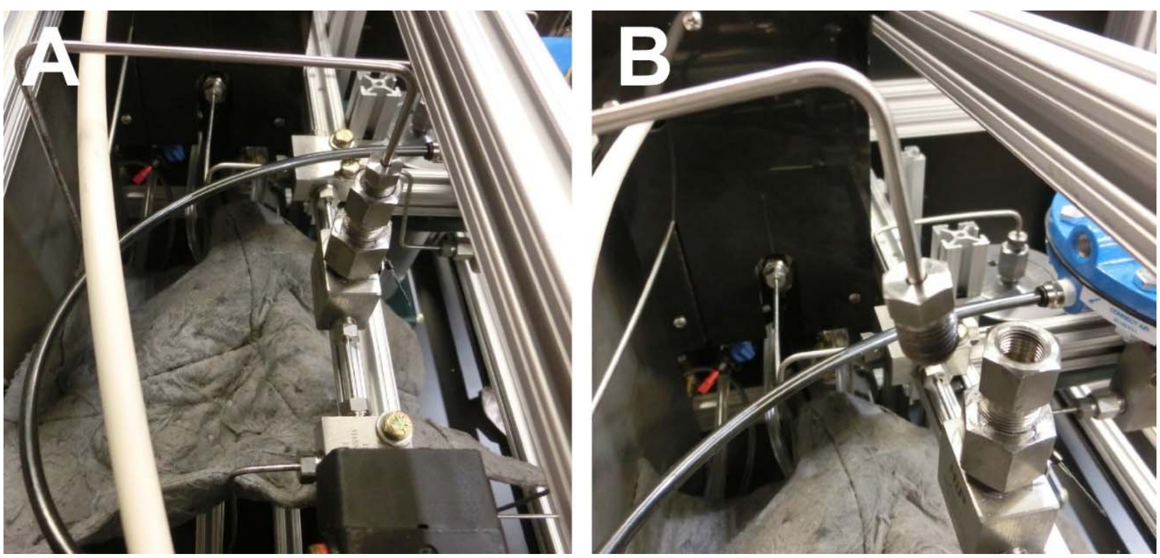

Figure 27: Safety head rupture disc replacement preparation

The first part of this process is to remove the case cover. If the circulator is sitting on the circulator equipment cart it may be necessary to remove the water bath from the cart to get access to all the case screws. This can be done by using a 3/16” hex driver to loosen the two L-shaped framing brackets at the front of the water bath. The 8” framing piece can then be lifted up and clear of the water bath. The outlet and inlet tubing on the back of the water bath should be clamped shut and disconnected from the bath. Unplug the unit and lift the water bath out of the recess.

Remove the seven screws from each side of the case and six screws from the top edges of the case cover. Remove the cover and set it aside.

Shown in Figure 27 is the safety head fixture. Place an absorbent pad beneath the work area to catch excess Galden HT-230 that may be discharged during the disassembly. Disconnect the 1/8” tubing from the top of the safety head. Do this by using a 1/2” wrench on the tube fitting gland with a 3/4” wrench on the adapter fitting below to stabilize and prevent counter rotation of this connection. Since the fluid path has now been diverted through this tubing, there may be a small quantity of fluid that escapes from the tube which should be captured by the absorbent pad.

Continuing with the disassembly, use two 1” wrenches to remove the safety head hold down nut. One wrench is used to secure the safety head body, with the other used to remove the nut. Notice in Figure 28A the placement of the wrenches. The 3/4” hex adapter fitting does not need to be removed independently.

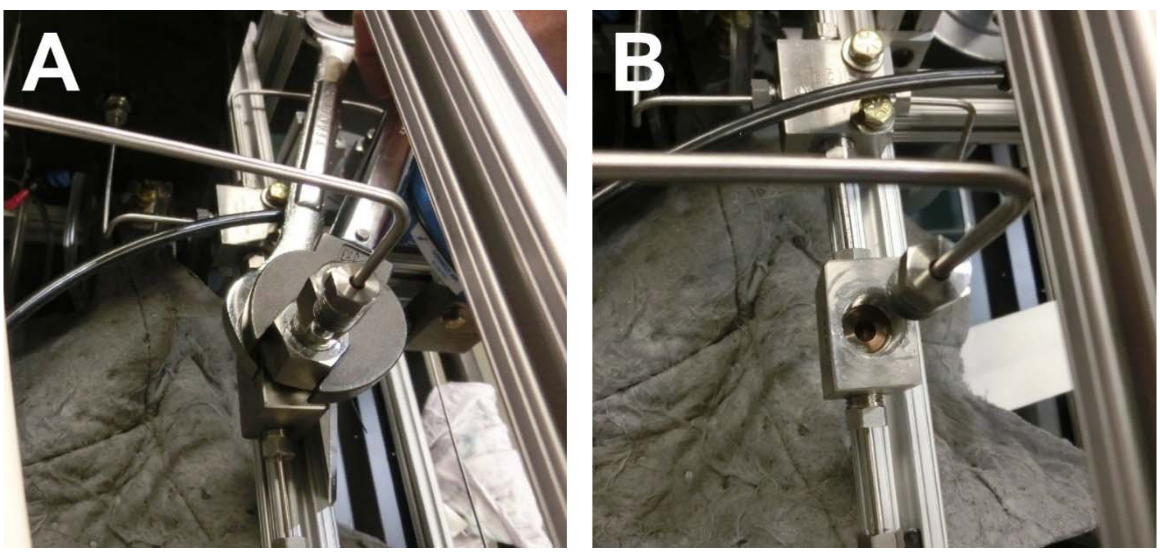

Figure 28: Remove safety head nut and hold down disc washer

Removing the nut exposes the bronze hold down washer (Figure 28B). Use a pair of forceps or needle-nose pliers to remove this piece and set it aside. The burst rupture disc lies below the washer and should also be removed with the forceps. There is likely an amount of Galden HT-230 fluid remaining at the bottom of the safety head. As much as possible this should be extracted with a pipette and the mating surfaces wiped clean.

Replace the rupture disc with an equivalent unit. The standard disc is rated to 12,500 psi and can be obtained for High Pressure Equipment Company, P/N RD-12500. The disc should be installed in the orientation shown in Figure 29 that being with the domed center pointing up relative to the safety head orientation. There is a chamfered edge at the bottom of the recess in the safety head where this sits. The bronze hold down washer should be inserted on top of the disc with the curved side down.

Reconnect the safety head hold down nut (1” hex size) using a 1” wrench to secure the safety head with another 1” wrench used to tighten the nut. Tighten this nut to 45 ft-lbs of torque. Finally, reattach the 1/8” tubing by placing a 3/4” wrench on the fitting adapter, and a 1/2” wrench on the tube fitting and tightening to 300 in-lbs of torque.

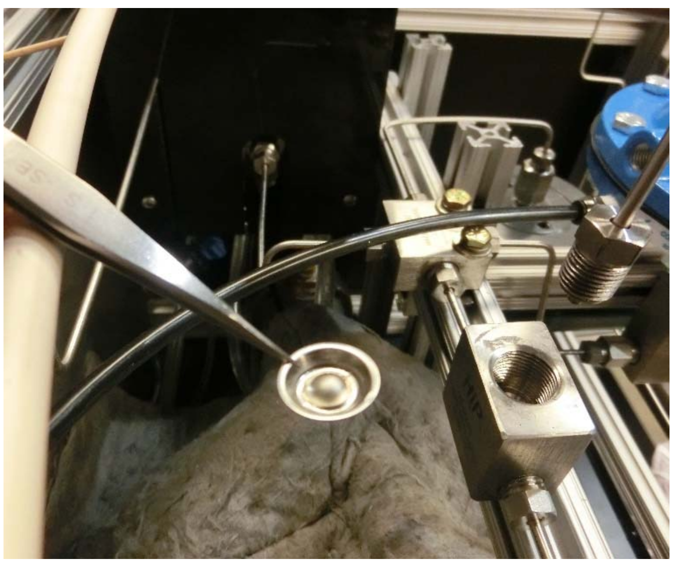

Figure 29: Rupture disc orientation

Before replacing the case cover it is advisable to test for leaks from the safety head by running the circulator. This is best done by using the short bypass tubing to connect the inlet to outlet to isolate the rest of the system from pressure. Obviously the integrity of the vent line from the safety head cannot be tested directly, but checking for the proper assembly of the safety head is worthwhile. Once complete, reattach the case cover and return the water bath to its proper location.

PRIMING THE AIR DRIVE PUMP

Every effort should be made to not run the air-drive pump dry. This is easily achievable by assuring that the fluid level in the reservoir is visible each time the system is run. However, should the system develop a leak or perhaps the system has not been used for an extended period of time the pump may not be primed for operation. With the general operating procedure outlined in the *Operating Instructions* section the pump will likely self-prime and this will not be noticed. The maximum height of the pump inlet above the fluid level is three feet for water, however, the Galden HT-230 is more viscous so that height is decreased in this application.

If the system is not building pressure or no flow is registering, and it is certain that **VENT** valve is closed and the rupture disc has not burst then additional measures may be required to prime the pump. If there is flow through the system then it may be an issue with the pressure regulator. See *Troubleshooting Pressure Regulator* section to address that issue. The following steps require increasing levels of user action to fulfill so follow the steps in order until the problem is resolved.

These procedures are suggested with the goal to minimize disruption to the current setup of the system. Therefore the charge on the accumulator can be left unchanged and the back pressure regulator can be used to check for the build-up of pressure. Depending on the accumulator charge and how much fluid is diverted to filling the accumulator each step might take several minutes to determine success.

Most importantly, connect the bypass tubing between the outlet and inlet of the ports on the back of the circulator to take the overburden cell out of the circuit.

Step 1: Decrease the distance between the pump and the fluid level by adding more fluid to the reservoir. Starting from the stopped condition slowly increase the ***DRIVE AIR*** pressure until the pump just starts to operate. Priming from a dry state requires the pump be run at low flow rates. Let this run for several minutes. When the pump is dry it has a more distinctive metallic clang when operating which disappears when primed. However, this may not be obviously detectable due to the acoustic foam inside the unit.

Increase the setpoint setting on the back pressure regulator to look for pressure build up. If this is not successful more fluid can be added while the system is operating. There should be no functional reason to add fluid above the top of the

Reservoir text on the back of the circulator. More can be safely added, but that is a significant amount of valuable fluid tied up in the circulator.

Step 2: The configuration of the siphon tube to the pump and drain is shown in Figure 30. On the left side of this image is the drain fitting. This particular fitting contains a sizeable dead volume that can reduce the effectiveness of the pump suction by drawing on expansion of the air bubble instead of the fluid. Using a suitable container to catch the fluid, open the drain to allow the air bubble to pass out of the fluid circuit. Close the drain valve.

Figure 30: Fluid siphon and drain lines

Repeat the process in Step 1 of priming the pump at low flow rates and checking for pressure build up in the system.

Step 3: This process requires access to the back of the circulator. Refer to Figure 10 to review the **PUMP PRIMING** section. Remove the plug from the port within this section and connect a short length of 1/4” OD tubing. To this tubing connect a syringe that can be used to draw fluid. With the circulator running at a low flow condition draw fluid into the syringe followed by slow discharge back into the line. Iteratively do this until the flow returns to the system. Close the valve and replace the plug in the port.

WATER BATH OPERATION AND MAINTENANCE

The water bath supplied with the system for delivering cooling fluid to the internal cool-side heat exchanger is the Cole-Parmer Polystat with an 8.6L heating/cooling bath and circulation capacity. For detailed operating instructions covering startup procedures and changing the temperature set point please refer to the manual for that instrument.

This unit has the capacity to deliver 17 liters/min to the circulator to cool the returning confining fluid from the overburden cell. This cooling requirement is necessary because some of the components of the system cannot tolerate the high temperatures of the circulating media.

The equipment cart is equipped with a filtration device, shown in Figure 31 to help minimize particulates building up in the system. This device does not remove bacteria so it is highly recommended that some antibacterial/fungal solution be added to the water in the bath. Due to the wide variety and potential toxicity of certain treatments this was not provided with the bath.

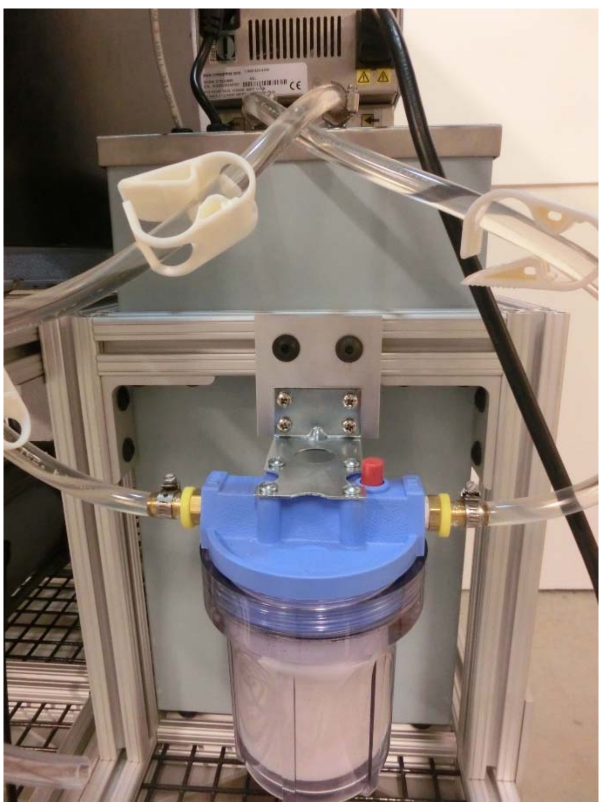

Figure 31: Water filtration device

The filtration device will remove particulates down to 5 microns, so it is not sufficient to remove bacteria. The system was flushed during the testing process, but it is possible additional particulates remain. Literature for the filtration device has been included with the general documents or with the additional filter cartridge shipped with the unit. Replacement cartridges can be readily obtained from McMaster-Carr p/n 5165K39. Pinch clamps have been placed on the water circulation tubing to be used when cartridge replacement is necessary.

TORQUE SPECIFICATIONS

Torque requirements for the preparation of fittings from High Pressure Equipment Company (www.highpressure.com).

AF1	55 in-lbs.
AF2	120 in-lbs.; initial compression 300 in-lbs.; final seating
HF4	300 in-lbs.
Safety head hold down nut	45 ft-lbs

For AF1 and AF2 fittings there should be bottoming-out or dead-stop when the connection is properly assembled.

TUBING SPECIFICATIONS

The unit is shipped with a variety of tubing. The chart below documents the suggested tubing should replacement or additional tubing be required. The supply house and part numbers can be found in the parts list supplied with the circulation system.

Main compressed air line	3/8” OD x 1/4” ID; Nylon; Shore D48
Nitrogen low pressure line; compressed air line to gas booster	1/4” OD x 1/8” ID; Polyethylene tubing; Shore D44
Galden HT-230 lines; internal and drain lines	1/4” OD x 1/8” ID; Clear PVC tubing; Shore A85
Water bath fluid lines	1/2” OD x 3/8” ID; Clear Tygon tubing
Water drain lines	3/8” OD x 1/4” ID; Clear Tygon tubing
Stainless steel tubing	1/8” OD x 0.06” ID – or- 1/16” OD x 0.03” ID

FURTHER INFORMATION

This document may be updated periodically to reflect questions from users. Please check back at www.daedalusinnovations.com in the support section for more recent versions of this document.

Technical support can also be obtained by emailing questions to support@daedalusinnovations.com, or contacting Daedalus directly at 610-358-4728.

Other correspondence can be directed to:

Daedalus Innovations, LLC
200 Racoosin Drive, Suite 106
Aston, PA 19014

APPENDIX A: VIBRATION ISOLATION

The accumulator within the circulator is intended to dampen pulsing of the air driven pump. However, additional vibrations can be transmitted through the stainless steel tubes which provide the flow path for the heated confining fluid. This vibration can sometimes affect diffusion measurements in the NMR instrument. Presented here is a strategy for minimizing these vibrations. Further aspects of mounting the NMR instrument are also addressed.

NMR Instrument Table

The NMR instrument should be mounted on a sturdy table. There should be enough room for access beneath the table. When inserting the Daedalus cell in the NMR instrument there are several high pressure connections that need to be made. Making these connections requires some space for maneuverability of hand tools. Any table designed should take this into account. Additionally there are connections to be made above the magnet.

The table will also need to be able to have mechanisms for securely mounting the high pressure tubing. In addition, a bulkhead panel (further described below) will need to be securely mounted on the table. These suggestions greatly help to minimize transmitted vibrations.

Isolation Solution

The vibration from the circulator is transmitted primarily through the stainless steel tubing containing the confining fluid. Provided the magnet is mounted on a sturdy table separate from the circulator cart, there is no need to isolate the vibrations through the floor.

Successful installations of similar systems have had isolation springs installed between the circulator and the NMR instrument. The isolations springs (see **Error! Reference source not found.**) are constructed out of 1 or 2 loops of approximately 5” in diameter, of 1/16” high pressure inch tubing. Each end of the tubing should be fitted with the gland 15-2AM1 and the sleeve 15-2A1.

The circulator cart from Daedalus has a bulkhead panel which will have the couplers 15-21AF2-B with the adapter 15-21AF1AM2. This adapter will accept the 1/16” tubing connector part 15-2AM1 and 15-2A1.

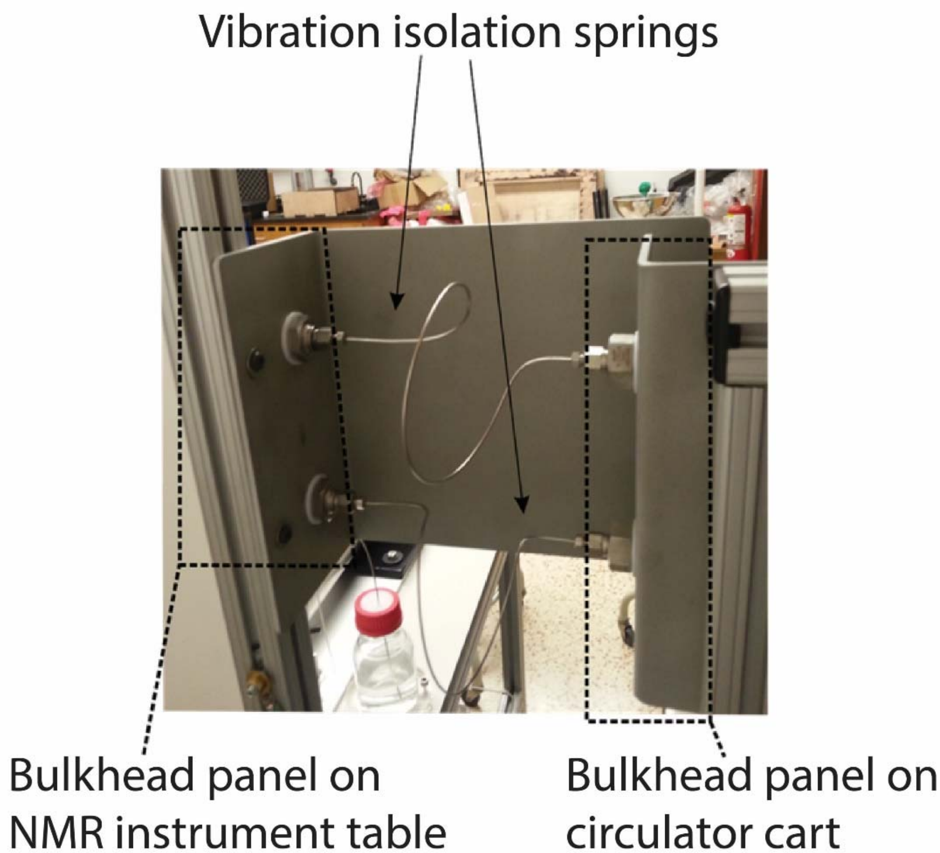

Figure 32: Vibration isolation springs

A second bulkhead panel, or something similar will need to be constructed and mounted on the NMR instrument table. The bulkhead panel should be mounted in such way that it doesn't introduce significant resonances. On the panel will need to be mounted two bulkhead connectors, part number 15-21AF2-B each fitted with the adapter 15-21AF1AM2. It is recommended the bulkhead panel be constructed out of aluminum plate and to mount the bulkhead connectors in PTFE sleeved washers. An example sleeve could be obtained from McMaster-Carr P/N #2706T38. The sleeve would need to be trimmed to fit, but otherwise works quite well.

The PTFE provides an insulating layer between the bulkhead connector and the panel. The stainless steel tubing will be housing fluids at temperatures of up to 200°C. Care should be taken so that the mounting is such that a user is significantly protected from, and warned about, the high temperatures. The bulkhead panel shown labeled in Figure 33.

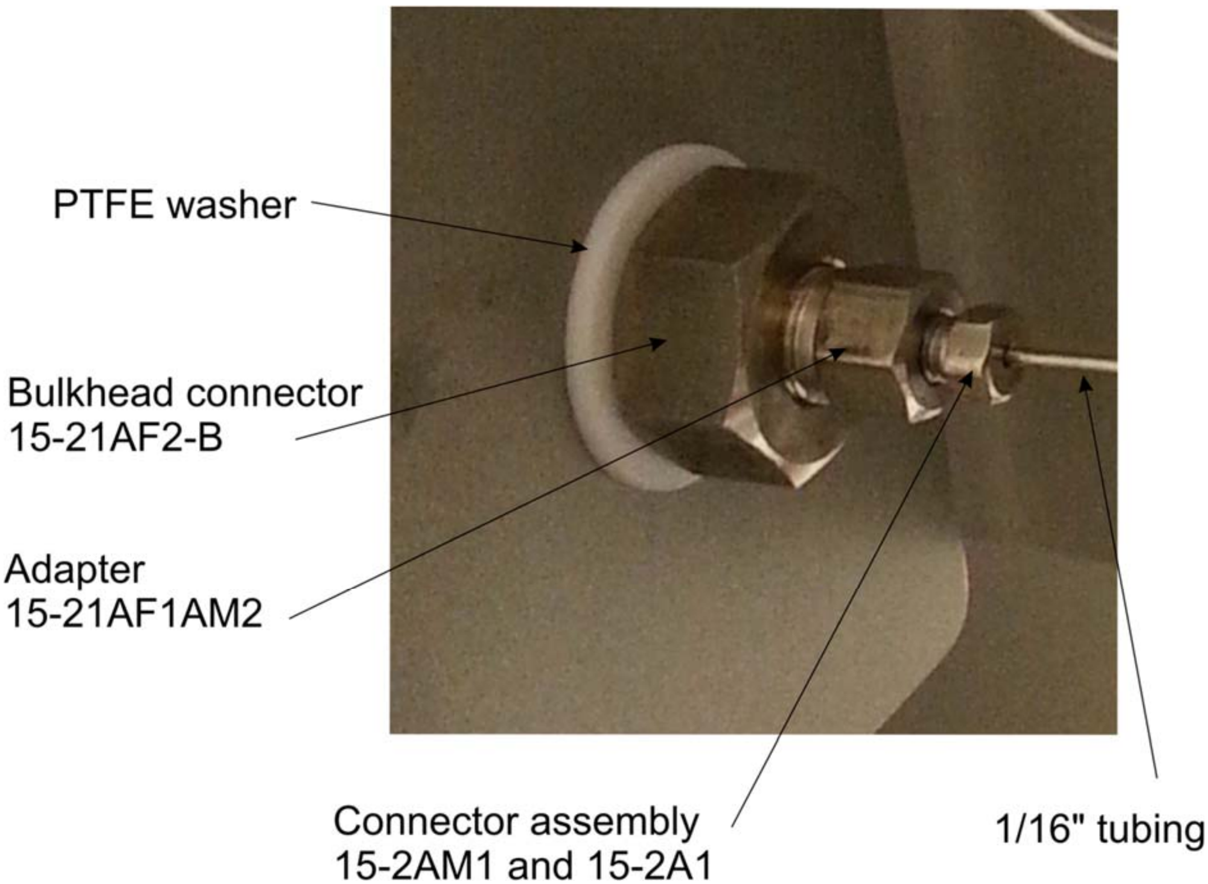

Figure 33: Bulkhead panel assembly with part labels

APPENDIX B: USING WRENCHES ON HIGH PRESSURE COMPONENTS

Initial mating of the connections should be able to be achieved by hand. Occasionally the connections may not be lined up and the strain on the connection may make hand tightening difficult. This is especially true for connections made at the top and the bottom of the cell. Instead of forcing the connection with wrenches, small movements of the connections should be made to help engage the threads. Stainless to stainless connections are particularly prone to seizing under high torque so wherever permissible anti-seize compound for stainless steel should be used on the threads. For connections common to the overburden cell the AM2 style fittings are most prone to this. If the compound has worn off and applying more does not help lubricate the threads, the connections may need to be re-tapped.

Using Wrenches

Always ensure that the flats of the wrench are fully engaged on the connector. Failure to do so could result in slippage and damage the flats of the connector. This is shown in Figure 34.

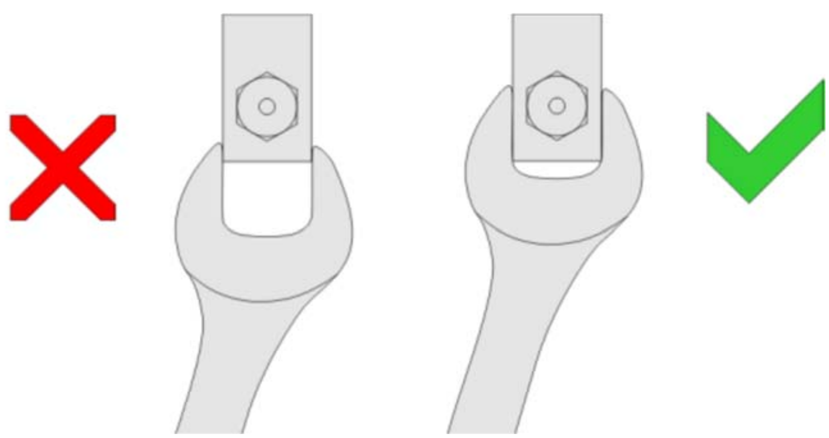

Figure 34: Proper engagement of wrenches

Using Torque Wrenches

A torque wrench is designed to yield slightly when the desired torque is applied. The desired torque is chosen by rotating the handle until the torque value (in-lbs.), is just visible above the handle indicator. The torque must always be applied in the direction of the arrow shown on one side of the torque wrench. Once the desired torque is reached the head will yield slightly and an audible click will be heard. When this happens no further torque should be applied. The torque wrench is supplied with an instruction manual for proper setting and use of the wrench.

Tightening Fittings

When applying a large torque with two wrenches it is essential that the torque is always applied in a squeezing motion. When using a fixed wrench and a torque wrench to tighten the fittings it is significantly easier to control the motion and apply the desired torque if the angle between the two tools is kept at small as possible. The fixed wrenches supplied with the overburden cell have the opening at an angle of 15° from the main lever arm. Turning a wrench upside down will change the angle of the main lever arm by 30°, when the wrench is engaged on the same flats. When tightening the high pressure fittings the orientation of the fixed wrench should be chosen in conjunction with the flats on the connector to ensure that the angle is kept to a minimum. Figure 35 shows three options for arranging a torque wrench and a fixed wrench for a connector, and how the positioning can be altered to bring minimize the angle between the lever arms of the wrenches.

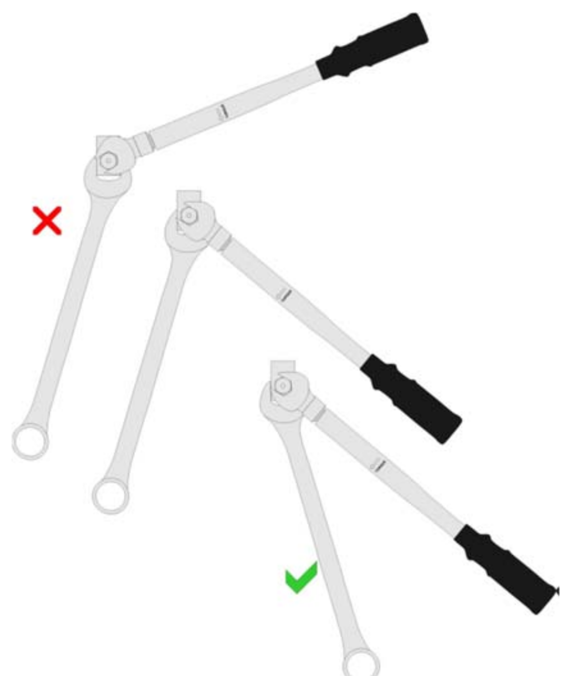

Figure 35: Positioning the fixed and torque wrenches for tightening

When using pairs of wrenches it is important to, as much as possible, have them in the same plane (Figure 36). This makes the torque much easier to control.

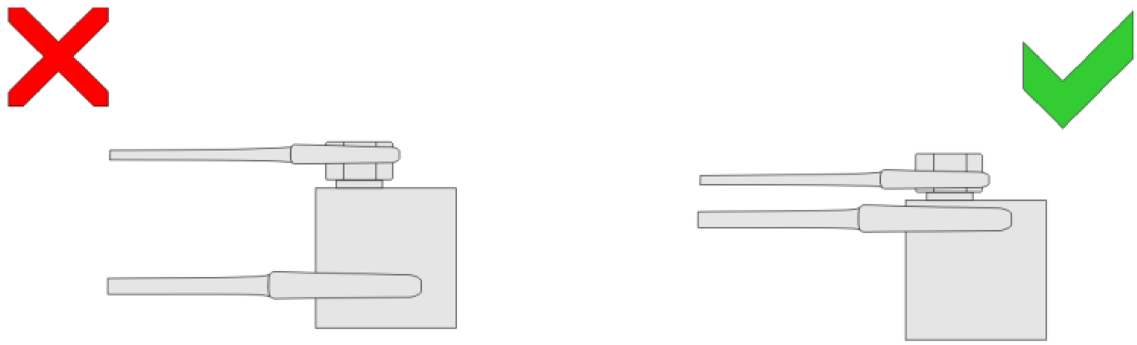

Figure 36: Keep wrenches close when tightening

Loosening Fittings

When loosening the fittings the fixed wrenches should be used. The same principles as tightening fittings apply, always use a squeezing motion, always engage the flats fully, try to keep the wrenches in the same plane, and try to minimize the angle between the two wrenches. Since the fittings will give significantly as soon as the initial seal is broken it is very important to have excellent control over the torque. The best way to do this is to try and arrange the two wrenches so that the lever arms can be spanned with both hands and squeezed to loosen. This gives the best way of sensing the torque and being able to release it quickly.

APPENDIX C: SAMPLE TEMPERATURE MONITORING

The temperature of the core sample can be measured at the faces using thermocouples. The 1/4” piston tubing used for the core injection fluid has an inner diameter of 0.083” which provides sufficient space to insert small diameter thermocouples, but maintain sufficient volume for injection fluid. Long length thermocouples with 1/16” outer diameters can be inserted inside these tube sections and secured with a high pressure fitting.

The part list for making this connection is as follows:

- HiP P/N 15-21AF1HM4-T (adapter for 1/16” thermocouples)
- HiP P/N 60-23HF4 (high pressure Tee for HM4 fittings
- HiP P/N 15-21AF1HM4 (standard reducer from1/4” to 1/16” tubing)
- Omega P/N TJ36-CPIN-116-U-18-XX (18” T-type thermocouple) or TJ36-ICIN-116-U-18-XX (18” J-type thermocouple)
- Omega P/N TC-08 (8-channel USB thermocouple data acquisition module)

The assembly is shown below in Figure 37.

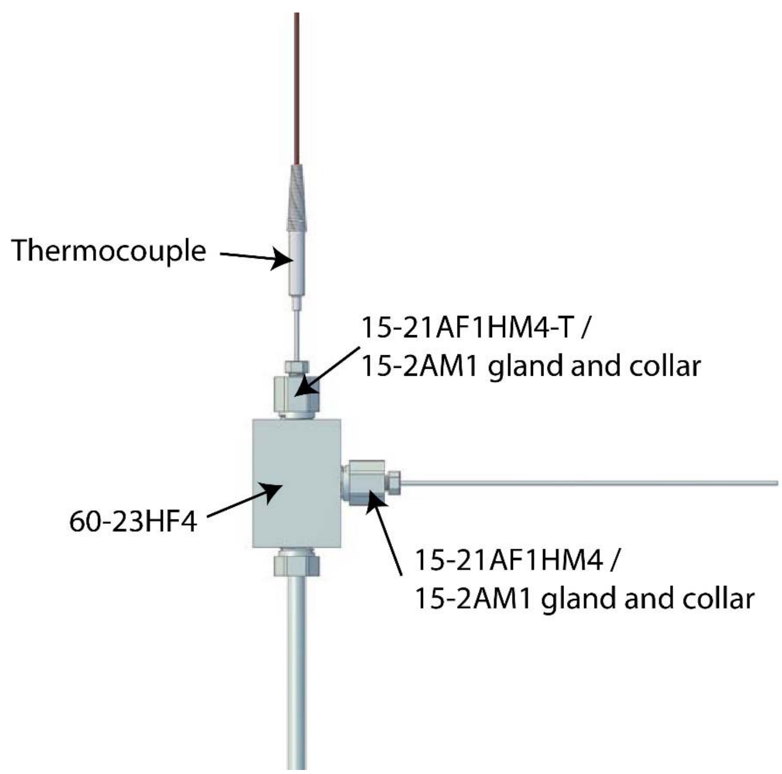

Figure 37: Thermocouple mounting diagram

This assembly connects directly to the piston tubes passing through the overburden cell end caps. The fluid passing to the core would enter through the side port of the thermocouple assembly. This assembly can be duplicated for the opposite side of the cell for monitoring the top and bottom of the sample.

The thermocouple is secured in the 15-21AF1HM4-T adapter using a standard 15-2AM1 gland and collar. The AM1 fitting is a compression fitting which will bite into the thermocouple to from the pressure seal. This means that once the gland is fitted to the thermocouple it cannot be removed. Thus, the positioning is specific to a certain core mount and piston tube length. Generally, for the standard components supplied with the overburden cell this should not cause problems when switching between samples. However, if the piston tube or core mount size is altered it will require specific thermocouple/gland setups to match the combined length.

First Time Positioning of the Thermocouple

To properly position the thermocouple the piston tube should be connected to the core mount and tightened to specification. The 60-23HF4 Tee should be connected to the piston tube and the 60-21AF1HM4-T adapter tightened to the Tee as shown in Figure 37. Then with a new 15-2AM1 gland and collar placed on the thermocouple, it should be passed through the 60-21AF1HM4-T adapter until the probe tip is near the end of the core mount. The tip should be slightly recessed from the face (see Figure 38). The AM1 gland can then be tightened to the 60-21AF1HM4-T adapter. Be sure to monitor the probe tip placement as the gland is tightened as it can move slightly during the initial phases of tightening. The gland/collar can still be moved during this phase if alignment problems occur. Once placement is confirmed tighten the AM1 gland to the same specification used for 1/16" tubing connections.

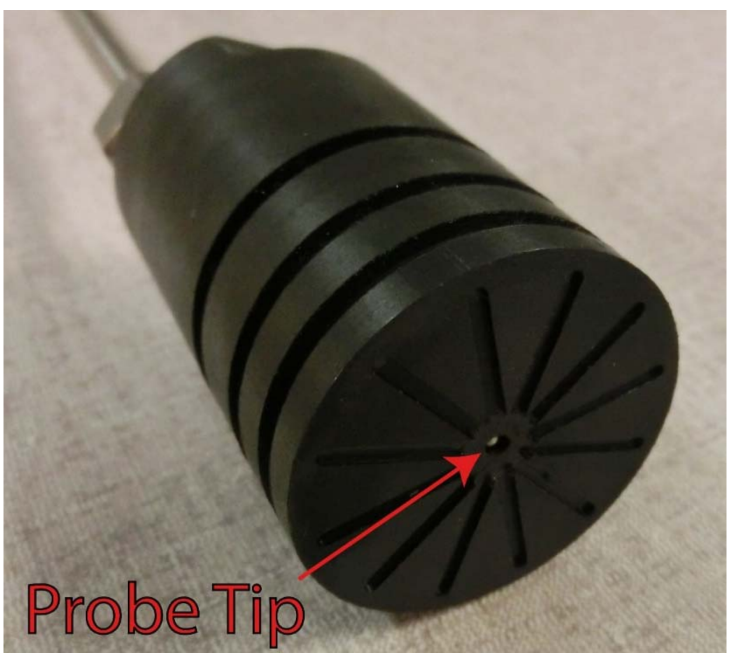

Figure 38: Thermocouple probe placement

The thermocouples should be connected to the Daedalus circulation system to provide feedback for maintenance of the sample temperature. The thermocouples can also be connected to the Omega P/N TC-08 which provides USB connection to a PC for output monitoring. It is important to note that the thermocouples specified are immersion thermocouples and may not read accurately if the core injection fluid is not present. Therefore, it is important when monitoring both faces of the core that fluid be delivered to both ends even when flow of fluid through the core is not intended.

The thermocouple assembly shown in Figure 37 can be treated as a unit. When disassembling the overburden cell simply loosen the HM4 fitting on the bottom of the 60-23HM4 Tee to free it from the piston tube. The thermocouple can then be carefully retracted from the piston tube and the assembly set aside for the next use.

APPENDIX D: CONFINING PORT ADAPTERS FOR 1" CORE HOLDERS

The smaller end-plug dimensions for the 1" diameter core holders do not permit large bore tubing to be connected directly to the confining port plug. As configured, the fitting port is AF1-style (HiP) for 1/16" tubing. The largest bore tubing is only 0.03" inner diameter, but the through hole in the end-plug is 0.0625" diameter. This constriction can create a sizeable, but still manageable pressure drop across the top and bottom of the overburden cell. Depending on the circulation system used it may also inhibit stable temperature regulation of the core at low circulation flow rates.

The overcome this limitation an experimental adapter has been developed (Figure 39). This direct connect adapter is made from 1/4" tubing with a 0.083" inner diameter. One end is machined to accept any of the HiP fittings for 1/4" tubing; that being left-hand threaded 1/4"-28 threads with 60° chamfer. The other end is machined to match the dimensions of an assembled AM1 gland and collar. This permits the tubing itself to be threaded directly into the confining port plug without additional glands or collars.

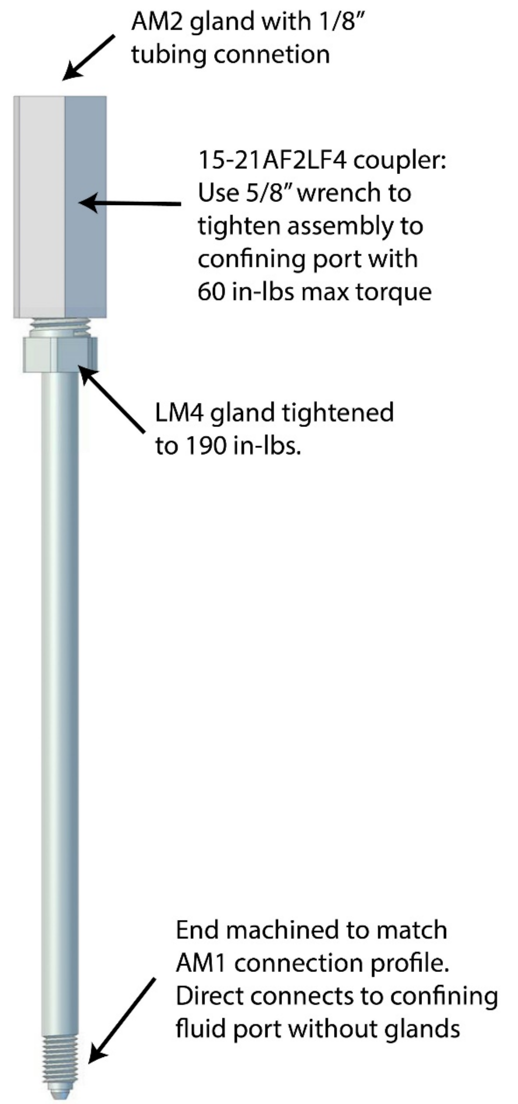

Figure 39: Confining port direct connect adapter

The opposing standard end can be terminated as needed. The assembled adapters as shipped are terminated with a coupler that accepts LM4 fittings on one end and AM2 fittings on the other. This permits the standard 1/8" tubing, with 0.06" inner diameter to be connected to the confining port. Other couplers could also be connected is required.

Tighten the LM4 gland to the coupler with 190 in-lbs torque. Connect the assembled adapter to the confining port by using a 5/8" wrench on the coupler. **Tighten to 60 in-lbs torque.** Excess torque could damage the end-plug. **Due to the low torque of application it is important the connecting 1/8" is not causing a counter rotation to be applied to the adapter. This will cause it to disengage and leak.**