

### SUBAMBIENT TEMPERATURE CONTROLLER



# micromeritics®

# **OPERATOR MANUAL**

029-42801-00 Aug 2024 (Rev B)

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- Certain products sold by MICROMERITICS are the products of reputable manufacturers, sold under their
  respective brand names or trade names. We, therefore, make no express or implied warranty as to such
  products. We shall use our best efforts to obtain from the manufacturer, in accordance with his customary practice, the repair or replacement of such of his products that may prove defective in workmanship or materials. Service charges made by such manufacturer are the responsibility of the ultimate purchaser. This states our entire
  liability in respect to such products, except as an authorized person of MICROMERITICS may otherwise agree
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- Expendable items, e.g., sample tubes, detector source lamps, indicator lamps, fuses, valve plugs (rotor) and stems, seals and O-rings, ferrules, etc., are excluded from this warranty except for manufacturing defects. Such items which perform satisfactorily during the first 45 days after the date of shipment are assumed to be free of manufacturing defects.

Purchaser agrees to hold MICROMERITICS harmless from any patent infringement action brought against MICROMERITICS if, at the request of the purchaser, MICROMERITICS modifies a standard product or manufactures a special product to the purchaser's specifications.

MICROMERITICS shall not be liable for consequential or other type damages resulting from the use of any of its products other than the liability stated above. This warranty is in lieu of all other warranties, express or implied, including but not limited to, the implied warranties of merchantability or fitness for use.

### MICROMERITICS CORPORATE PROFILE

Micromeritics Instrument Corporation is the world's leading supplier of high-performance systems to characterize particles, powders and porous materials with a focus on physical properties, chemical activity, and flow properties. Our technology portfolio includes: pycnometry, adsorption, dynamic chemisorption, particle size, intrusion porosimetry, powder rheology, and activity testing of catalysts. The company has R&D and manufacturing sites in the USA, UK, and Spain, and direct sales and service operations throughout the Americas, Europe, and Asia. Micromeritics systems are the instruments-of-choice in more than 10,000 laboratories of the world's most innovative companies and prestigious government and academic institutions. Our world-class scientists and responsive support teams enable customer success by applying Micromeritics technology to the most demanding applications. For more information, please visit www.micromeritics.com.

### PATENTS

For patent information, visit www.Micromeritics.com/patents.

### CONTACT US

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#### Instrument Service or Repair

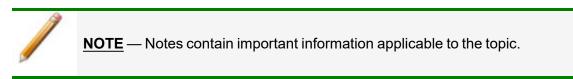
Phone: 1-770-662-3636 International: Contact your local distributor or call 1-770-662-3636 Service.Helpdesk@Micromeritics.com

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### ABOUT THIS MANUAL

The following symbols or icons indicate safety precautions and/or supplemental information and may appear in this manual:





**<u>CAUTION</u>** — Cautions contain information to help prevent actions that may damage the instrument or components.



**WARNING** — Warnings contain information to help prevent actions that may cause personal injury.

### General Safety



Do not modify this instrument without the authorization of Micromeritics Service Personnel.

Any piece of laboratory equipment can become dangerous to personnel when improperly operated or poorly maintained. All employees operating and maintaining Micromeritics instruments should be familiar with its operation and should be thoroughly trained and instructed on safety.

- Read the operator manual for any special operational instructions for the instrument.
- Know how the instrument functions and understand the operating processes.



- Wear the appropriate personal protective equipment when operating this instrument — such as eye protection, lab coat, protective gloves, etc.
- When lifting or relocating the instrument, use proper lifting and transporting devices for heavy instruments. Ensure that sufficient personnel are available to assist in moving the instrument.
- Always pay attention to the safety instructions provided on each label affixed to the instrument and do not alter or remove the labels. When inspecting the instrument, ensure that the safety labels have not become worn or damaged.
- Proper maintenance is critical to personnel safety and smooth instrument operation and performance. Instruments require regular maintenance to help promote safety, provide an optimum end test result, and to prevent costly down time. Failure to practice proper maintenance procedures can lead to unsafe conditions and shorten the life of the instrument.
- Improper handling, disposing of, or transporting potentially hazardous materials can cause serious bodily harm or damage to the instrument. Always refer to the SDS when handling hazardous materials. Safe operation and handling of the instrument, supplies, and accessories are the responsibility of the operator.

### INTENDED USE



The instrument is intended to be operated by trained personnel familiar with the proper operation of the equipment recommended by the manufacturer and as well as relevant hazards involved and prevention methods. Other than what is described in this manual, all use is seen as unintended use and can cause a safety hazard.

The instrument is intended to be used as per applicable local and national regulations.

### TRAINING

It is the customer's responsibility to ensure that all personnel operating or maintaining the equipment participate in training and instruction sessions. All personnel operating, inspecting, servicing, or cleaning this instrument must be properly trained in operation and machine safety before operating this instrument.

### ENVIRONMENTALLY FRIENDLY USE PERIOD

#### **Hazardous Substances Table**

	Hazardous Substances					
Part Name	Lead (Pb)	Mercury (Hg)	Cadmium (Cd)	Hexavalent Chromium (Cr (VI))	Polybrominated biphenyls (PBB)	Polybrominated diphenyl ethers (PBDE)
Metal Parts	о	о	о	о	о	о
Electronic Components	о	о	о	ο	o	ο
Hose and Hose Fittings	о	ο	o	ο	0	0

o Hazardous substance is below the specified limits as described in SJ/T11363-2006.

x Hazardous substance is above the specified limits as described in SJ/T11363-2006.

The Environmentally Friendly Use Period (EFUP) for all enclosed products and their parts are per the symbol shown here unless otherwise marked. Certain parts may have a different EFUP (for example, battery modules) and are marked to reflect such. The Environmentally Friendly Use Period is valid only when the product is operated under the conditions defined in the product manual.



### SYMBOLS THAT MAY APPEAR ON THE INSTRUMENT

The following symbols or icons indicate safety precautions and/or supplemental information and may appear on your instrument:



Use extreme caution when working on the instrument where one of these symbols may be displayed. These symbols indicate the part may be hot and cause serious burns.



Use the cotton gloves provided in the accessory kit when handling heated surfaces. These cotton gloves are not intended to protect hands when heated surfaces are above 60  $^\circ$ C.



When working on an instrument where this symbol is displayed, refer to the corresponding Operator Manual for additional information.



When this symbol is displayed, toxic or flammable gases require proper venting of exhaust.

This symbol can also indicate the instrument uses mercury which is an extremely toxic substance. Read the Safety Data Sheet (SDS) and be aware of the hazards of mercury and know what to do in the event of a spill or an exposure incident.

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## **1 ABOUT THE CRYOCOOLER II AND CRYOCOOLER III**

CRYOCOOLER II



The CryoCooler II is an optional unit for operations with the AutoChem II, AutoChem III, and AutoChem HP at subambient temperatures. With it, a sample can be cooled and held for an extended time at any temperature between +20 and -100 °C. See <u>Specifications for the</u> <u>CryoCooler II and III on page 1 - 6</u> for guaranteed temperature ranges. This holding temperature can be shifted at any time either up or down within the overall range. After being at a fixed temperature, the sample temperature can be ramped down or up to ambient at rates anywhere between 1 and 50 °C/min but, with optimum control at rates being between 10 and 25 °C/min. At ambient temperature, the furnace controller takes over and proceeds with ramped heating without CryoCooler II involvement.

The CryoCooler II unit consists of a mechanical pump, a storage Dewar for liquid nitrogen (LN<sub>2</sub>) and a transfer line that attaches to the AutoChem II, AutoChem III, or AutoChem HP furnace. Stable subambient temperatures are achieved by causing small, frequently repeated injections of LN<sub>2</sub> to fill the furnace cavity within which is located the sample. (Actually, the stream that reaches the sample compartment is not liquid, but cold vapor.) Upward ramped temperatures are obtained by simultaneously diminishing the frequency of LN<sub>2</sub> inputs and increasing the energy input to the furnace.



Moisture from the atmosphere condenses in the transfer line when the unit is withdrawn from the cold storage Dewar. If there is water in the transfer line when the unit is inserted into the storage Dewar, it will freeze and prevent operation. See <u>Maintenance and Troubleshooting on page 5</u> - 1 for information on how this is prevented and what to do when it occurs.



Ensure the mechanism is clean and dry before inserting it into the  $LN_2$ . If the transfer system has accumulated water or water vapor inside, it will freeze when inserted and prevent operation.

### CRYOCOOLER III



The CryoCooler is an optional unit for operations with the AutoChem II and AutoChem III only at subambient temperatures. With it, a sample can be cooled and held for an extended time at any temperature between +20 and -100 °C. See <u>Specifications for the CryoCooler II and III on</u> <u>page 1 - 6</u> for guaranteed temperature ranges. This holding temperature can be shifted at any time either up or down within the overall range. After being at a fixed temperature, the sample temperature can be ramped down or up to ambient at rates anywhere between 1 and 50 °C/min but, with optimum control at rates being between 5 and 25 °C/min. At ambient temperature, the furnace controller takes over and proceeds with ramped heating without CryoCooler involvement.

The CryoCooler III unit consists of an electrical heater, a storage Dewar for liquid nitrogen  $(LN_2)$  and a transfer line that attaches to the AutoChem III furnace. Stable subambient temperatures are achieved by causing small, frequently repeated injections of  $LN_2$  to fill the furnace cavity within which is located the sample. (Actually, the stream that reaches the sample compartment is not liquid but cold vapor.) Upward ramped temperatures are obtained by simultaneously diminishing the frequency of  $LN_2$  inputs and increasing the energy input to the furnace.

Moisture from the atmosphere condenses in the transfer line when the unit is withdrawn from the cold storage Dewar. If there is water in the transfer line when the unit is inserted into the storage Dewar, it will freeze and prevent operation. See <u>Maintenance and Troubleshooting on page 5</u> <u>- 1</u> for information on how this is prevented and what to do when it occurs.

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Ensure the mechanism is clean and dry before inserting it into the  $LN_2$ . If the transfer system has accumulated water or water vapor inside, it will freeze when inserted and prevent operation.

### DEWAR PRECAUTIONS



Always handle glass Dewars with care. Any product incorporating a vacuum is a potential safety hazard and should be treated with caution. If in doubt, contact your safety officer.



Improper handling, disposing of, or transporting potentially hazardous materials can cause serious bodily harm or damage to the instrument. Always refer to the SDS when handling hazardous materials. Safe operation and handling of the instrument, supplies, and accessories are the responsibility of the operator.



Do not pour liquid nitrogen directly into a sink. Doing so may cause drain pipes to burst.



The CryoCooler unit is intended for use with liquid nitrogen only; under no circumstances is it to be used with liquid oxygen.



Always wear a safety face shield or goggles when handling liquid nitrogen to prevent the possibility of splashing into the eyes.

When handling Dewars containing liquefied gases or cryogenic liquids:

- Wear protective equipment:
  - goggles or face shield
  - an insulated or rubber apron
  - insulated gloves
- When pouring liquefied gases from one container to another:
  - cool the receiving container gradually to minimize thermal shock
  - ° pour the liquified gas slowly to prevent splashing
  - vent the receiving container to the atmosphere

### SPECIFICATIONS FOR THE CRYOCOOLER II AND III

### CRYOCOOLER II

	Temperature Hold Stability
from -80 to 20 °C	± 5° C
from -100 to -80 °C	± 10 °C

	Rate Range (best fit)	Max Degrees Error (from best fit)
10 °C/min	from 6 to 12 °C/min	±5°C
20 °C/min	from 15 to 30 °C/min	± 10 °C

### CRYOCOOLER III

	2 Minute Stabilization	15 Minute Stabilization
from -60 to 20 °C (ideal)	±2°C	±0.5 °C
from -100 to -60 °C (low)	±5°C	±2°C

	Rate Range (best fit)	Max Degrees Error (from best fit)
5 °C/min	from 3 to 6 °C/min	3 °C
10 °C/min	from 6 to 12 °C/min	5 °C
25 °C/min	from 15 to 30 °C/min	10 °C

Due to physical limitations, the CryoCooler III installed on an AutoChem II is tuned to match the CryoCooler II specification.

### **2** INSTALL THE TRANSFER TUBE SUPPORT BRACKET

This section is applicable to the AutoChem II, AutoChem HP, and AutoChem III.

Install the transfer tube bracket kit onto the analyzer first. Then follow the instructions to install the CryoCooler.

CryoCooler II Installation on page 3 - 1 CryoCooler III Installation on page 4 - 1

This system is designed to remove the strain on the furnace cooling connection. A Phillips head screwdriver is required to perform this installation.

#### KIT CONTENTS [PART NUMBER 029-33030-00]



Bracket assembly, with a hinged door secured by a thumbscrew



Elbow assembly, to be attached to the CryoCooler transfer tube

- 1. If already attached, disconnect the CryoCooler from the AutoChem furnace.
- 2. Open, then remove the furnace from the AutoChem and lay it on the instrument deck so that the two screws on the underside of the furnace are accessible. Locate two screws on the underside of the furnace, below the CryoCooler connection. Remove the two screws and save them for the next step. (The screws may be very tight.)



Remove these two screws. These screws will be used to attach the transfer tube support bracket.

3. Install the new bracket to the underside of the furnace using the original screws removed in the previous step. Replace the furnace back onto the AutoChem.



Secure bracket with two screws previously removed.

4. On the CryoCooler transfer tube, at the discharge assembly, loosen the knurled wheel which was previously used to secure the transfer tube to the furnace.

5. Push the 1/8 in. tube of the elbow assembly into the transfer tube fitting. Tighten the knurled wheel.



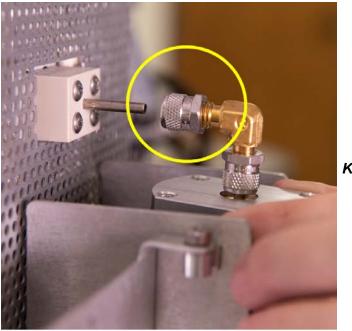
6. Open the gate of the bracket assembly.

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7. Slightly loosen the brass knurled nut on the elbow assembly.

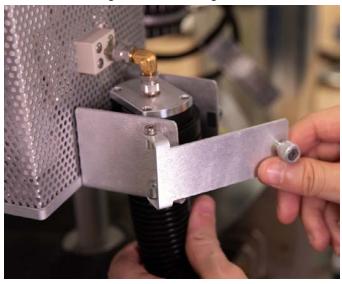


8. Guide the new elbow assembly and the CryoCooler transfer tube onto the bracket and push the elbow gently onto the furnace cooling tube.



Knurled nut

- 9. Tighten the knurled nut counter-clockwise until secure.
- 10. Close the gate and hand tighten the knurled nut on the gate clockwise .



11. Allow five minutes for thermal expansion and contraction effects to dissipate, then start the AutoChem analysis.

### **3 CRYOCOOLER II INSTALLATION**



See Install the Transfer Tube Support Bracket on page 2 - 1 prior to installing the CryoCooler.





The CryoCooler must be free of internal moisture before insertion into  $LN_2$ . If present, moisture will freeze and prevent operation. The system was free of moisture when it left the factory; however, dry air, nitrogen, or other inert gas should be passed through its transfer tube to ensure it has remained dry.

- 1. Place the Dewar on the floor near the front of the analyzer or beside the analyzer on the workbench.
- 2. Select one of the following instructions :

#### For AutoChem II and AutoChem HP installations

Attach one end of the provided cable to the connector labeled *SubAmbient Cooler* on the front panel of the AutoChem analyzer. Attach the other end of the cable to the CryoCooler II.

- a. Restart the AutoChem application. The application will automatically sense the presence of the CryoCooler.
- b. To verify the CryoCooler II is operating properly before inserting it into LN<sub>2</sub>, enable manual control. To enable manual control, in the AutoChem application, go to Unit [n] > Enable Manual Control. On the schematic, right-click the CryoCooler box on the right side of the window and set the rate to 100%. The green light on the CryoCooler housing should illuminate and the rotor at the end opposite the housing should be visibly turning.
- c. Power off the CryoCooler II using manual control, but leave the cable connected.

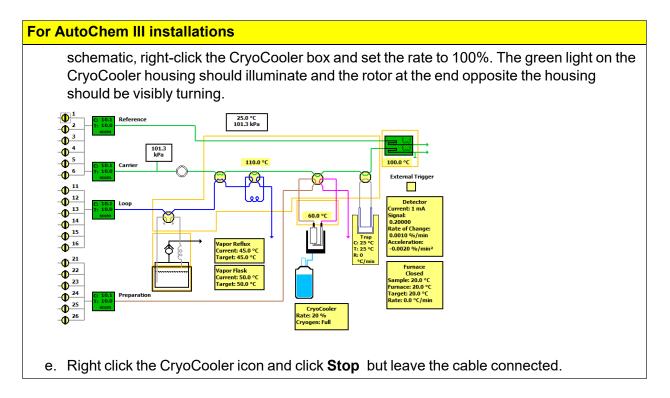
#### For AutoChem III installations

Attach one end of the provided cable to the connector labeled *CryoCooler* on the front panel of the AutoChem III analyzer. Attach the other end of the cable to the CryoCooler II.

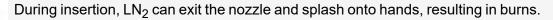
- a. Start the AutoChem III application, then go to Unit [n] > Unit Configuration.
- b. In the CryoCooler Type group box, select the CryoCooler II option.

Configuration IP address: Serial #: demo Change IP Board ID	CryoCooler Type O CryoCooler O CryoCooler	III 99.9999 mL
Software Versions	Mass Flow Contr	oller Range
MIC BIOS: Demo Boot Block	Carrier:	
Controller: Demo Application	Reference:	100.00 cm³ STP/min
Application: AutoChem III Version 1.00	Loop:	100.00 cm³ STP/min
	Preparation:	100.00 cm³ STP/min
Options	Interface Board	Version
Heat zones: Yes Sorption trap: Yes	Rev slot 02:	2
Preparation gas: Yes KwikCool: Yes	Date slot 02:	1/1/1970
Vapor: Yes	Rev slot 03:	2
Compressed air source: Compressor	Date slot 03:	1/1/1970
ОК	Cancel	

- c. Click **OK** to close and save the change.
- d. To verify the CryoCooler II is operating properly before inserting it into LN<sub>2</sub>, enable manual control. To enable manual control, in the analyzer application, go to *Unit [n] > Enable Manual Control*. The blue CryoCooler icon should display on the schematic. On the



3. Slowly insert the transfer mechanism into the Dewar filled approximately two-thirds full of  $LN_2$ .



Boiling will result as the mechanism contacts the  $LN_2$  so insert the mechanism slowly over a period of several minutes. This permits most of the resulting vapor to escape before the mechanism is finally centered on the Dewar opening.

If the CryoCooler II fails to respond, it may be due to components having not yet thermally adjusted to the new temperature surroundings or to ice formation from previously condensed moisture. Wait a few minutes and try again. Failure this time is almost certainly due to ice formation. See <u>Maintenance and Troubleshooting on page 5 - 1</u>.

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### **4 CRYOCOOLER III INSTALLATION**



See Install the Transfer Tube Support Bracket on page 2 - 1 prior to installing the CryoCooler.





The CryoCooler III is compatible with the AutoChem 2920, AutoChem II and AutoChem III only. It is strongly advised that the CryoCooler III not be installed or used on other AutoChem models.

#### CryoCooler III Pump Components

Component	Description
Red cap	<b>Relief valve.</b> Remove this cap if the CryoCooler III is idle for an extended period of time. This cap must be in place for the CryoCooler III to operate.
Green button	Not used.
Transfer tube connection	Conducts LN <sub>2</sub> to the instrument.
Cable connection	Connects the instrument to the CryoCooler III.

- 1. In the AutoChem III software, go to Unit [n] > Unit Configuration.
- 2. In the CryoCooler Type group box, select the CryoCooler III option.

Configuration IP address: Serial #: demo Change IP Board ID	CryoCooler Type © CryoCooler III O CryoCooler II
Software Versions	Mass Flow Controller Range
MIC BIOS: Demo Boot Block	Carrier: 100.00 cmÂ <sup>3</sup> STP/min
Controller: Demo Application	Reference: 100.00 cmÂ <sup>3</sup> STP/min
Application: AutoChem III Version 1.00	Loop: 100.00 cmÂ <sup>3</sup> STP/min
	Preparation: 100.00 cm³ STP/min
Options	Interface Board Version
Heat zones: Yes Sorption trap: Yes	Rev slot 02: 2
Preparation gas: Yes KwikCool: Yes	Date slot 02: 1/1/1970
Vapor: Yes	Rev slot 03: 2
	Date slot 03: 1/1/1970

- 3. Click **OK** to close and save the change.
- 4. Power down the Autochem III and unplug the Autochem III from its power source.

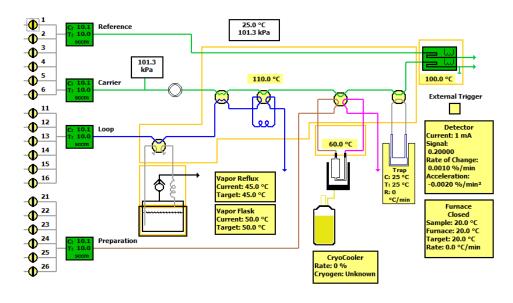
5. Attach the 8 pin DIN connector of the provided cable to the CryoCooler pump.



6. Attach the 9-pin male connector of the provided cable to the connector labeled *CryoCooler* on the lower front panel of the AutoChem III analyzer. Ensure that both screws are used to secure the cable.



- 7. Plug the AutoChem III into the power source. The CyroCooler III pump will beep 9 times and a yellow light on the pump will blink.
- To verify the CryoCooler III is operating properly before inserting it into LN<sub>2</sub>, enable manual control. To enable manual control, in the analyzer application, go to *Unit [n] > Enable Manual Control*. The yellow CryoCooler icon should display on the schematic.





A blue Dewar icon indicates that either CryoCooler II is selected in the configuration window or the pump is currently running while being installed.

- 9. Fill the Dewar approximately two-thirds full of LN<sub>2</sub>.
- 10. Wear protective gloves and face shield and slowly insert the transfer mechanism into the Dewar opening.



During insertion,  $LN_2$  can exit the nozzle and splash onto hands, resulting in burns.

Boiling will result as the mechanism contacts the  $LN_2$  so insert the mechanism slowly over a period of several minutes. This permits most of the resulting vapor to escape before the mechanism is finally centered on the Dewar opening.

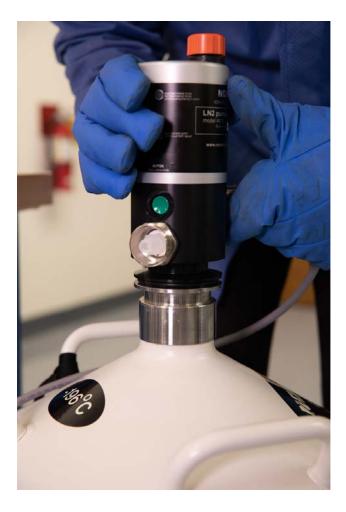


If the pump has recently been removed from the Dewar, the transfer mechanism must be free of ice prior to inserting into the Dewar. Allow the mechanism to completely thaw then wipe it with a clean, dry cloth prior to insertion into the Dewar. Inserting the icy mechanism into the Dewar will cause ice buildup and may clog the transfer tube. See <u>Maintenance and Troubleshooting on page 5 - 1</u>.





11. Lower the transfer mechanism into the Dewar until the flange of the CryoCooler III and the flange of the Dewar opening meet.





12. Wrap the hinged clamp around the CryoCooler III flange and the Dewar flange. Ensure that the clamp is securely seated around both flanges.



13. Seat the metal clamp closure into the opposite end of the clamp and hand tighten the knurled nut to secure the clamp.





When installing the clamp onto the flanges, the seal must be airtight. Ensure that the clamp catches both the flange of the Dewar and the flange of the CryoCooler.

14. On the CryoCooler III pump, connect the transfer tube to the CryoCooler III pump by inserting the plastic plug on the transfer tube onto the plastic connector on the CryoCooler pump. Do not remove the plastic insert from either the transfer tube or the connector on the pump. The pump will not function properly if either of the plastic plugs is removed.



- 15. Hand tighten the plastic transfer tube connector to the pump.
- 16. Slide the metal nut on the transfer tube onto the to metal connector on the pump. Hand tighten the nut until secure.

### INSTALL THE CRYOCOOLER III ON AN AUTOCHEM II

The installation of the CryoCooler III on an AutoChem II is similar to the installation on an AutoChem III. Exceptions are:

- Configuration setup
- Transfer pipe connection
- Thermocouple placement
- Power management

#### **CONFIGURATION SETUP**

The AutoChem II configuration is set to the CryoCooler II by default. To use CryoCooler III on the AutoChem II, the CryoCooler option must be selected manually:

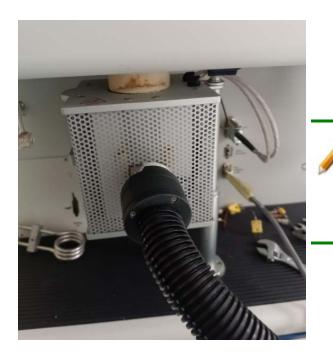
- 1. Start the AutoChem II application, then go to Unit [n] > Unit Configuration.
- 2. In the CryoCooler Type group box, select the CryoCooler III option.

Unit Configuration (Unit 4 - C(b), down)	
Unit Configuration (Unit 1 - S/N: demo)	×
Configuration	
IP address:	
Serial #: demo	
Change IP Board ID	
Software Versions	
MIC BIOS: Demo Boot Block	
Controller: Demo Application	
Application: MicroActive for AutoChem II	
Options	
Temperature M	
CryoCooler: Yes	
CryoCooler type:	
◯ CryoCooler II	
KwikCool: No	
Vapor generator: Yes	
Loop volume: 0.00000 cm <sup>3</sup>	
OK Cancel	

3. Click **OK** to close and save the change.

### TRANSFER PIPE CONNECTION

Due to the furnace and sample tube structure on AutoChem II, it is recommended to connect the transfer pipe directly to the nozzle instead of using the elbow connector. This approach prevents bending of the transfer tubes after extensive use.



If the application indicates that the Dewar is empty despite being filled with  $LN_2$ , the issue is likely a blocked transfer pipe or nozzle. Connecting the transfer pipe directly as above will resolve this issue.

#### SAMPLE THERMOCOUPLE PLACEMENT

The sample thermocouple should be placed as low as possible to ensure its tip is below the nozzle. Failure to do so will compromise temperature control capabilities.

#### **POWER MANAGEMENT**

To protect the power board, it is recommended to power off the instrument before connecting the CryoCooler III and then power the instrument back on. While hot-swapping is generally supported, it may occasionally fail to recognize the cooler.

### **5 MAINTENANCE AND TROUBLESHOOTING**

The CryoCooler reservoir retains  $LN_2$  for many days when not used. If use is temporarily suspended, leave it plugged into the analyzer but disconnected from the furnace inlet. An internal heater produces a slight outflow of gaseous nitrogen sufficient to prevent the counter diffusion of moisture laden ambient air and consequent deposition of ice in the transfer tube.



When the transfer tube is cold, it is hard and brittle making it extremely fragile. Flexing a cold tube may cause it to break.

The buildup of ice and frost particles is unavoidable in any device which uses  $LN_2$  in a laboratory environment. The CryoCooler III has been designed to minimize problems from ice. Ice problems can be avoided almost entirely if, on a weekly basis, the Dewar is emptied of liquid nitrogen and its interior carefully and thoroughly dried. Repeated filling and evaporation can lead to an accumulation of ice crystals within the Dewar.

### ICE BUILDUP

**CryoCooler II** —A thermal overload switch activates and cuts power to the pump motor. The green light will also be OFF in this condition.

CryoCooler III — A 10 or 11 beep alarm will sound and the pump will cease to operate.

To remove ice buildup with either CryoCooler:

- 1. Remove the transfer mechanism from the Dewar.
- 2. Unplug the transfer mechanism from the analyzer to allow a few minutes for the thermal protector to reset .
- 3. Blow dry air or nitrogen into the transfer tube and out of the pump, holding the mechanism nearly vertical. Also blow into the pump inlet and into one of the holes of the member connecting the housing and the pump just above the connection point to the pump. When doing so, be careful not to heat the plastic transfer tube directly; it will melt if overheated.



For the CryoCooler II only, it is advisable to heat the pump mechanism with a laboratory heat gun until it is too warm to hold.

4. Slowly reinsert the mechanism into the Dewar and proceed with the sample analysis.

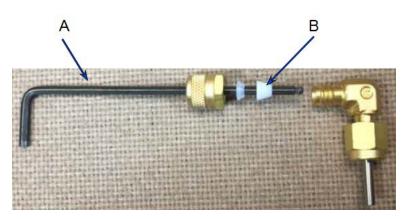
### REPLENISH THE DEWAR

Replenishing the Dewar without removing the pump and then having to dry it can be accomplished in either of two ways:

- Lift the housing a few inches above the Dewar allowing it to rest on the flange. The nozzle of the Model 021 LN<sub>2</sub> transfer system can then be inserted and the Dewar refilled.
- Withdraw the transfer mechanism from the nearly empty Dewar and immediately insert it into another full Dewar.

### TEFLON FERRULE REPLACEMENT

The Teflon ferrule in the elbow may become damaged and require replacement. It may be helpful to use a piece of 1/8" tube, or a small tool, to align the parts when reassembling.



- A. Tool to help when assembling
- B. Teflon ferrule
- 1. Disconnect the CryoCooler II from the AutoChem furnace.
- 2. Remove the knurled brass nut.
- 3. Remove the Teflon ferrule.
- 4. Place the ferrule as shown above.

### TROUBLESHOOTING THE CRYOCOOLER III



When the pump is inserted into the Dewar, the power to the pump should be ON to prevent internal freezing of the pump.

#### Warning LED is ON and a double beep every 30 seconds

- Cause: There is less than 4 liters of  $LN_2$  left in the Dewar.
- Action: Fill Dewar with  $LN_2$ .

#### Warning LED flashes and 1 beep is audible

- *Cause:* Pump is not cold or the storage Dewar is empty.
- Action: Check the Dewar level and refill.

#### Warning LED flashes and 2 beeps are audible

- Cause: Dewar level sensor is not faulty.
- Action: Contact Customer Service.

#### Warning LED flashes and 3 beeps are audible

- Cause: Pump flow sensor is not faulty.
- Action: Contact Customer Service.

#### Warning LED flashes and 4 beeps are audible

- Cause: No pressure is building.
- Action: Check for a leak in the system.

#### Warning LED flashes and 10 beeps are audible

- Cause: Exhaust may be blocked or frozen.
- Action: Check for blockage or freezing and clear the obstruction.

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#### Warning LED flashes and 11 beeps are audible

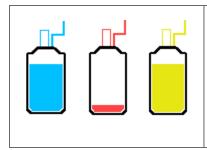
*Cause:* The pressure measuring tube is frozen.

Action: Check for frozen tube and clean blockage.

#### Instrument breaker has tripped.

- Cause: The CryoCooler III was plugged in to the instrument while it was on.
- Action: Unplug the AutoChem III from the power source for about 20 seconds. Connect the CryoCooler cable. Plug the AutoChem III back in.

#### CryoCooler III Dewar Icons



Blue. Indicates the Dewar is full

Red Indicates the Dewar is empty. When the pure

**Red.** Indicates the Dewar is empty. When the pump speed is set to 100%, the Dewar displays as red and empty, a warning light illuminates, and the pump beeps continuously.

**Yellow.** Indicates Dewar level is unknown and pump rate is set to zero (for CryoCooler III installations only).

### SAFE SERVICING



Do not service or modify this instrument without the authorization of Micromeritics Service Personnel.



Always wear a safety face shield or goggles when handling liquid nitrogen to prevent the possibility of splashing into the eyes.

To ensure safe servicing and continued safety of the instrument after servicing, service personnel should be aware of the following risks:

### PARTS AND ACCESSORIES

Parts and accessories are located on the Micromeritics web page.

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