

CRYOCOOLER

SUBAMBIENT TEMPERATURE CONTROLLER



OPERATOR MANUAL

029-42801-00
May 2017
(Rev A)

TRADEMARKS

Micromeritics is a registered trademark of Micromeritics Instrument Corporation.

Teflon is a registered trademark of E. I. DuPont de Nemours Company.

Copyright

The software described in this manual is furnished under a license agreement and may be used or copied only in accordance with the terms of the agreement.

CONTACT US

Micromeritics Instrument Corporation

4356 Communications Drive
Norcross, GA / USA / 30093-2901
Phone: 1-770-662-3636
Fax: 1-770-662-3696

www.Micromeritics.com

U.S. Inquiries

U.S. Sales Phone: 1-770-662-3636

USSales@Micromeritics.com

Quotes, Orders, and Customer Satisfaction

Customer Service Phone: 1-770-662-3636

Orders@Micromeritics.com

U.S. Instrument Service Department

U.S. Service Department Phone: 1-770-662-3666

Service.Helpdesk@Micromeritics.com

Micromeritics Scientific Services

[Customer Support Portal](#)

<http://techsupport.micromeritics.com/portal>

ABOUT THIS MANUAL

Parts and accessories can be found online at www.Micromeritics.com.

The following icons may be found in this manual:



NOTE - Notes contain important information applicable to the topic.



CAUTION - Cautions contain information to help prevent actions that may damage the analyzer or components.



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

Table of Contents

| | |
|-------------------------------|---------------------|
| Contact Us | <i>i</i> |
| About this Manual | <i>ii</i> |
| 1 About the CryoCooler | <i>1 - 1</i> |
| 2 Installation | <i>2 - 1</i> |
| 3 Maintenance | <i>3 - 1</i> |

Blank Page

1 ABOUT THE CRYOCOOLER

The CryoCooler is an optional unit for operations with the AutoChem at subambient temperatures. With it, a sample can be cooled and held for an extended time at any temperature between +20 and -100 °C. Temperatures are guaranteed to be held within ± 5 °C between +20 and -80 °C, and within ± 10 °C between -80 and -100 °C. 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 involvement.

The CryoCooler unit consists of a cryogenic pump mechanism, a storage dewar for liquid nitrogen (LN₂) and a transfer line that attaches to the AutoChem furnace. Stable subambient temperatures are achieved by causing small, frequently repeated injections of LN₂ 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 LN₂ inputs and increasing the energy input to the furnace.

Water within the CryoCooler mechanism inevitably causes it to malfunction. Its accumulation must be avoided. Water collects in the transfer section when it is withdrawn while cold from the LN₂ dewar and allowed to warm to ambient temperature. Moisture from the atmosphere condenses within the mechanism. When the transfer section is subsequently re-immersed in LN₂, this water freezes and prevents the motor from powering on, or blocks the transfer line. See [Maintenance on page 3 - 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 LN₂. If the transfer system has accumulated water or water vapor inside, it will freeze when inserted and prevent operation.

Blank Page

2 INSTALLATION

1. Attach one end of the provided cable to the connector labeled *SubAmbient Cooler* on the front panel of the analyzer. Attach the other end of the cable to the CryoCooler.
2. To verify the CryoCooler is operating properly before inserting it into LN₂, enable manual control. To enable manual control, in the analyzer application, go to **Unit > Enable Manual Control**. On the analyzer schematic, right-click the CryoCooler icon and set the speed to 100%. The green light on the housing should illuminate and the rotor at the end opposite the housing should be visibly turning. Power off the CryoCooler using manual control, but leave the cable connected.
3. Insert the transfer mechanism into its dewar filled approximately two-thirds full of LN₂. Boiling will result as the mechanism contacts the LN₂ so insert it 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.



Insert the transfer mechanism into the LN₂ slowly. Inserting the mechanism too quickly will cause boil-off of the LN₂ and possible splashing onto the hands.

4. Place the dewar on the floor near the front of the analyzer or beside the analyzer on the workbench.

INSTALL THE HOSE SUPPORT BRACKET KIT

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 hose



1. If already attached, disconnect the CryoCooler from the AutoChem furnace.
2. Open the furnace. 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 hose support bracket.

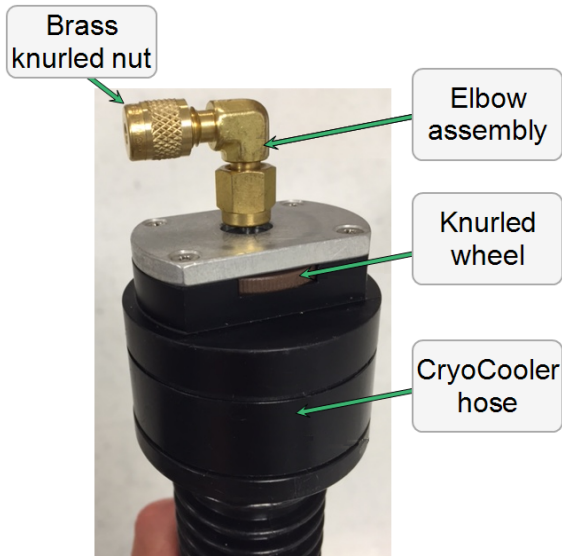
3. Install the new bracket to the underside of the furnace using the original screws removed in the previous step.



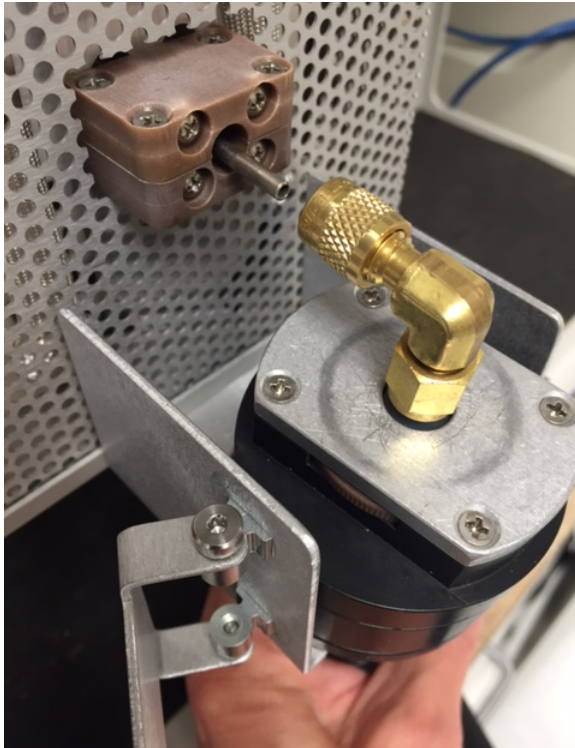
Secure bracket with two screws previously removed.

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

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



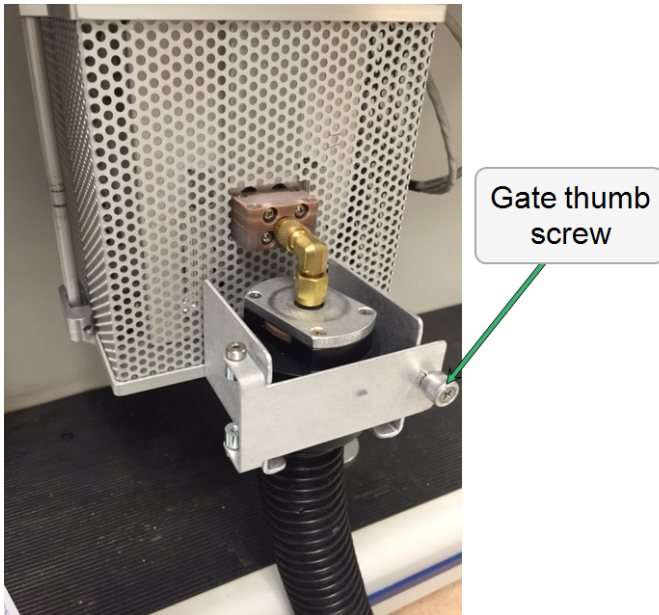
6. Open the gate of the bracket assembly.
7. Slightly loosen the brass knurled nut on the elbow assembly.
8. Guide the new elbow assembly and the CryoCooler hose onto the bracket and push the elbow gently onto the furnace cooling tube.



9. Tighten the knurled nut



10. Close the gate and secure it using the thumb screw. The system is now ready to use.



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

If the CryoCooler 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 [Maintenance on page 3 - 1](#) for information on ice formation.

3 MAINTENANCE

The buildup of ice and frost particles is unavoidable in any device which uses LN₂ in a laboratory environment. The CryoCooler 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.

REMOVE ICE BUILDUP

If ice buildup should occur, a thermal overload switch activates and cuts power to the pump motor. The green light will also be OFF in this condition.

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 line 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. It is advisable to heat the pump mechanism with a laboratory heat gun until it is too warm to hold. When doing so, be careful not to heat the plastic transfer line directly; it will melt if overheated.
4. Slowly reinsert the mechanism into the dewar and proceed with the sample analysis.



The CryoCooler reservoir retains LN₂ for several 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 counterdiffusion of moisture laden ambient air and consequent deposition of ice in the transfer line.

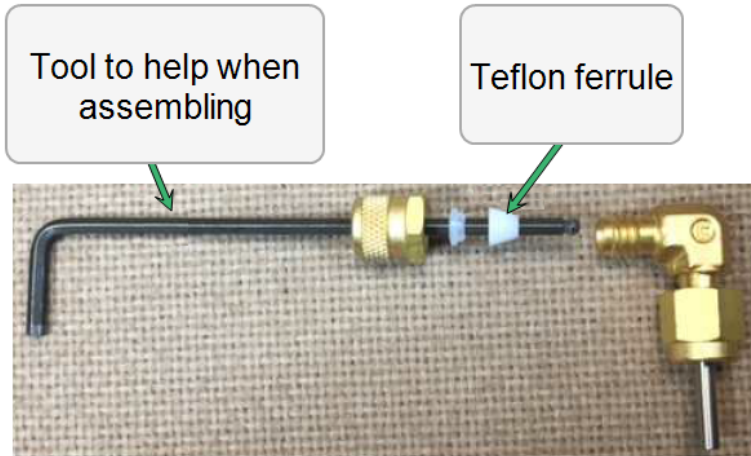
REPLENISH THE DEWAR

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 neck of the dewar, allowing it to rest on the rim of the neck. The nozzle of the Model 021 LN₂ 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, allowing introduction of insignificant ice.

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.



1. Disconnect the CryoCooler from the AutoChem furnace.
2. Remove the knurled brass nut.
3. Remove the Teflon ferrule.
4. Place the ferrule as shown above.