Bruker / ColdEdge WaveGuide Cryostat User's Guide

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ColdEdge Technologies

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WaveGuide Cryostat
USER'S GUIDE

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Manual Version 1.0
Part Number ?
July 2018

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This manual is part of the original documentation for the Bruker /ColdEdge WaveGuide Cryostat

Preface 0

Bruker strives to supply you with instructional and accurate documentation. We encourage you to tell us how we are doing. Please send us your suggestions for improvements, corrections, or bug reports. If there is anything you particularly liked, tell us as well. With your input and assistance, Bruker can continually improve its products and documentation.

You can send your messages and correspondence via e-mail, FAX, telephone, or mail. It is important to include the document name, product name, version number, and page number in your response. Here are the addresses and numbers to which you can send your messages.

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Thank you for your help.

Electrical Safety 0.1

Do not remove any of the protective covers or panels of the instrument. They are fitted to protect you and should be opened by qualified service personnel only.

Power off the instrument and disconnect the line cord before starting any cleaning work in the spectrometer. Never operate the instrument with the grounding cord disconnected or bypassed. Facility wiring must include a properly grounded power receptacle.

Chemical Safety 0.2

Individuals working with hazardous chemicals, toxic substances, or enclosed liquid samples must take every precaution possible to avoid exposure to these agents. As a general rule, THINK OF THE CHEMICAL LABORATORY AS A HAZARDOUS ENVIRONMENT IN WHICH YOU MUST CONTINUALLY MAINTAIN A HIGH STANDARD OF VIGILANCE. Do not assume a cavalier attitude -- the substances with which you work present very real, and very serious threats to your health and safety. Adhere to all currently rec-

ommended guidelines for standard laboratory safety as promulgated by governmental codes and contemporary laboratory practice. Inform yourself about the specific risks that are present when you handle actual or potential carcinogens (cancer-causing agents), explosive materials, strong acids, or any liquids that are sealed in glass containers.

Specifically:

- Be extremely careful when you handle sealed glass samples that are rapidly heated or cooled. The rapid cooling of some samples may result in the formation of a solid bolus in the sample tube that may make the tube prone to explosive rupture.
- Educate yourself about the temperature at which chemicals evaporate. When a sample gets close to the temperature at which it evaporates, it may quickly become volatile.
- In general, the safety threat posed by flying glass and violently escaping gases and liquids should not be underestimated.
- Wear safety glasses, face masks, and other protective clothing whenever there is any risk of spillage, breakage, or explosion. Protective shields should also be employed when there is any risk of explosion.
- Be sure that both storage and working areas are properly ventilated. They should be equipped with powerful blowers and fume heads.
- Store chemicals safely. Avoid integrating containers of chemicals that may result in dangerous combinations.
- Practice good housekeeping in work and storage areas. Clean up spills and refuse promptly. Do not leave volatile, combustible, or acidic liquids exposed on counters, benches, or other work areas.
- Make certain all chemical containers are properly labeled and classified, and that especially hazardous materials are appropriately designated with clearly understood decals or warnings.
- Never taste or inhale unmarked chemicals.
- All laboratories should be equipped with fire doors, fire extinguishers, fire smothering materials, and sprinkler systems or showers, as well as a detailed fire safety plan.

Microwave Safety 0.3

As long as the microwaves are contained in metal structures, microwaves can be very safe. Here are some precautions which, if followed, will eliminate the possibility of injury due to the microwaves.

- Do not have an open waveguide when the microwave power is on.
- Switch the bridge to standby when you remove or change EPR cavities.

Never look down an open waveguide when there is microwave power. The eyes are very susceptible to damage from microwaves.

Cryogenic Safety 0.4

Cryogenic liquids are typically liquefied gases condensed to their liquid state at very low temperatures; their boiling points are below -238° F (-150° C) . Different cryogens become liquids under different conditions of temperature and pressure, but all have two common properties: they are extremely cold and small amounts of the liquid can expand into very large volumes of gas.

- Contact with skin can cause frostbite or burns. Wear loose fitting cryogenic gloves when handling liquids. Closed-tow shoes or boots and long pants should be worn.
- Provide adequate ventilation as evaporating liquids can displace oxygen and create a risk of asphyxiation
- Soft and flexible objects become hard and brittle and may crack with sharp edges that can cut or otherwise pose risk of injury

Cryogenic liquids should not be stored in sealed containers as evaporating gas can potentially build to explosive pressures

Oxford Mercury ITC Manual 1
Lakeshore 336 Temperature Controller Manual 2
GAST Pump Manual 2

Introduction 1

This user's guide for the Bruker / ColdEdge WaveGuide Cryostat is meant to be a general guide for using the WaveGuide Cryostat. Users are encouraged to explore Chapter 6 for additional information about the operation of the coldhead, compressors and gas lines.

Using the WaveGuide Cryostat User's Guide 1.1

How to Find Things 1.1.1

Preface First, you should read the safety guide in the preface of the manual. Microwaves can be dangerous, particularly to your eyes. Cryogenic liquids can cause frostbite or burns. With normal precautions, the risk for injury can be minimized.

Chapter 2 Users are given a brief introduction to the basic principles of operation of the WaveGuide Cryostat

Chapter 3 Installation of the WaveGuide Cryostat and interfacing the WaveGuide Cryostat.

Chapter 4 Operation of the WaveGuide Cryostat

Chapter 5 Maintenance and Troubleshooting

Fonts 1.1.2

Special fonts are used in the text to differentiate between normal manual text and the text displayed in the program.

Times This is the font used for the normal text in the manual.

Helvetica This is the font used for text that is displayed by the program or must be entered into the program by you.

Special notes 1.1.3

Some special notation is employed in this manual to simplify the descriptions.

< ... > The content between the brackets needs to be substituted with proper entries by the user.

> The right bracket indicates sequential selection of the menu entries. For example, Processing > Filtering > Smoothing means clicking the Processing button in the menu bar, followed by clicking Filtering in the sub-menu, and then clicking Smoothing.

Warning box.

You will see a warning box sometimes in the lefthand margin. These are meant to point out critical information. In particular, it warns you about any procedures or operations that may be dangerous to the spectrometer or you. Always read and follow this advice.

Hint box.

In addition, there are also hint boxes in the lefthand margin These are meant to be helpful hints and point out important information.

Take note box.

In between the special notes previously mentioned are important issues of which you should make note. These are presented in take note boxes.

Introducing the WaveGuide Cryostat 2

The WaveGuide Cryostat is a completely closed cycle cryogenic cooling engine for the laboratory environment. This new cryogenic cooling technology is designed for direct replacement of older liquid transfer cooling systems.

Depending on the cryocooler employed, the WaveGuide Cryostat can be of 4K or 10K temperature versions. It utilizes a standard quartz dewar insert and standard 4 mm sample tubes. The WaveGuide Cryostat is a completely closed cycle system. It employs a cryocooler running on a closed cycle helium compressor to produce a minimum desired temperature. An auxiliary recirculation circuit is used to provide the transfer gas and is also fully closed cycle. It employs a separate closed cycle helium compressor which only requires high purity helium gas to replenish the small amount of gas lost during sample changes.

The WaveGuide Cryostat is a perfect fit for the EPR laboratory environment that uses cryogenic cooling. It replaces the traditional large dewar of liquid helium and transfer line; it does not block space in front of the spectrometer.

Principles of Operation 2.1

The WaveGuide Cryostat uses Gifford McMahon (GM) cooling technology to cool an auxiliary helium gas circuit to liquid cryogen temperatures. This cold gas is then passed around the base of the sample tube positioned in a special quartz dewar located in the microwave resonator. The WaveGuide Cryostat is based upon a closed cycle GM cooler. It employs a cryocooler, a compressor and flexible gas lines between the compressor and the cryocooler. This cryocooler generates the minimum temperature at the cold station. The cold station in turn cools the gas from the auxiliary helium circuit. Helium is circulated through the WaveGuide Cryostat and cooled using innovatively designed internal heat exchangers and Joule-Thomson (JT) technology. Components in the auxiliary helium circuit include a recirculator compressor, a gas manifold to regulate helium flow, connecting flexible gas lines between the recirculator compressor and gas manifold and between the gas manifold and the WaveGuide Cryostat cryocooler. Typical cooling time depends on the type of cold head attachment, temperature to be achieved and heat loads. The WaveGuide Cryostat cools down in about 60-75 minutes to minimum temperature with normal loads.

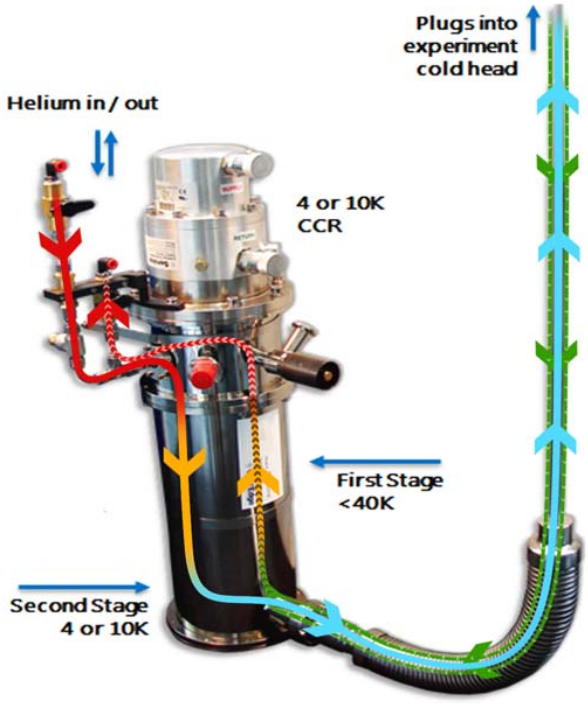

Figure 2-1 Helium recirculated transfer gas pathway. - new diagram***

A gas control manifold is provided to control the recirculated helium while operating. It allows regulation of the supply pressure to optimize the temperature requirements of the experiments. The manifold will also serve in purging and cleaning any contamination introduced into the system during cryostat or sample changes.

A magnet specific bracket is used to position the WaveGuide Cryostat system and attached resonator to the proper location in the electromagnet.

Additional material from various OEM (Original Equipment Manufacturer) products is included in Chapter 6:

- The SRDK408D2 and CH210N Coldheads and a description of the functional operation of the Gifford McMahon process.
- The F70L/H Compressor documentation.
- The HC-4E1 Compressor documentation.
- The flexible gas line documentation.
- The Mercury Temperature Controller documentation.
- The Lakeshore Temperature Controller documentation.

Component Description 2.2

WaveGuide Cryostat Cooling Engine 2.2.1

This is a completely closed cycle cryogenic cooling system. It consists of a GM cooler, a compressor to operate the GM cooler, a Recirculator compressor, and industrial flexible hose connections.

- **GM Cooler –** 4K or 10K coldhead used to cool the gas flowing through the heat exchangers and JT technology.
- **Compressor -** Supplies helium gas to operate GM cooler.
- **Recirculator compressor -** Recirculates gas from the gas control manifold to the sample position in the WaveGuide Cryostat and recovers the gas for complete closed loop operation.

Manifold 2.2.2

A gas manifold is provided to regulate the circulating helium gas as it travels from the WaveGuide Cryostat to the sample space. It includes a series of valves and ports that isolate the Recirculator compressor during sample changes and cryostat exchange, allow gas to be added during the purge operation, and allows the cryostat and quarz dewar to be evacuated for the removal of moisture

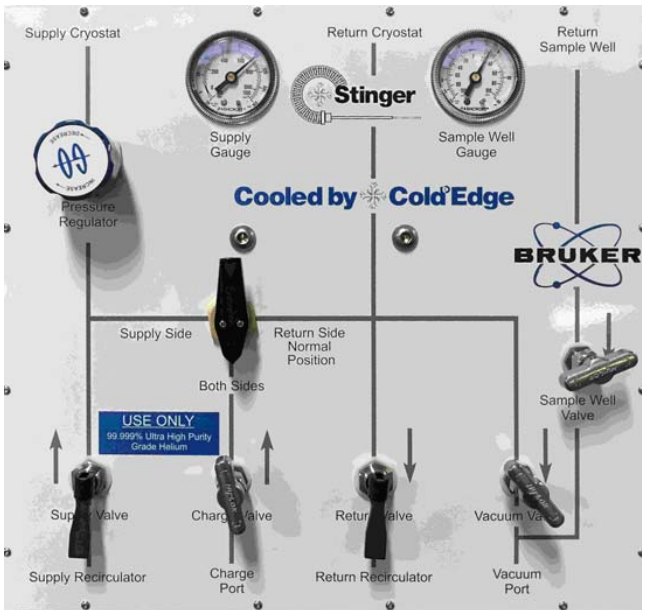

Figure 2-2 Gas Handling Manifold

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