800 MHz US^2 NMR system

To achieve ultra-high-fields (previously: >700 MHz, now: >850 MHz), the magnet must be cooled to below (ambient pressure) liquid He temperature.

The He cryostat is divided into two sections. The upper section remains at 4.2K. The magnet sits in the lower section, which is cooled to ~2K.

With this design, the He temp in the magnet bath doesn't depend on atmospheric pressure, room temp, etc (as in conventional cryostats).

However, the stability of the system does depend on the He cooling system. If the cooling system goes down, *the magnet will quench in 8-12 hours*.

Because of the cryostat design and the method of subcooling, the He temp in the magnet bath is extraordinarily stable, with variations only in the 0.1 mK range. This type of magnet is known as *ultrastabilized*.

The upper and lower sections of the cryostat are connected by narrow channels so the pressure is always the same in both (slightly higher than ambient).

Cooling is done by a Joule-Thomson cooling unit. Liquid He is allowed to expand through a needle-valve into a heat exchanger.

All that is necessary to keep the whole thing running is a small pump that keeps the pressure in the heat exchanger below 30 mbar.

If the pump completely stops the magnet could quench in 8-12 hours.

However:
There's a backup pump that it can switch to automatically.

The pumps are on emergency power and powered by a UPS that will keep them running for ~9 hours.

We have other spare pumps and equipment to connect everything.

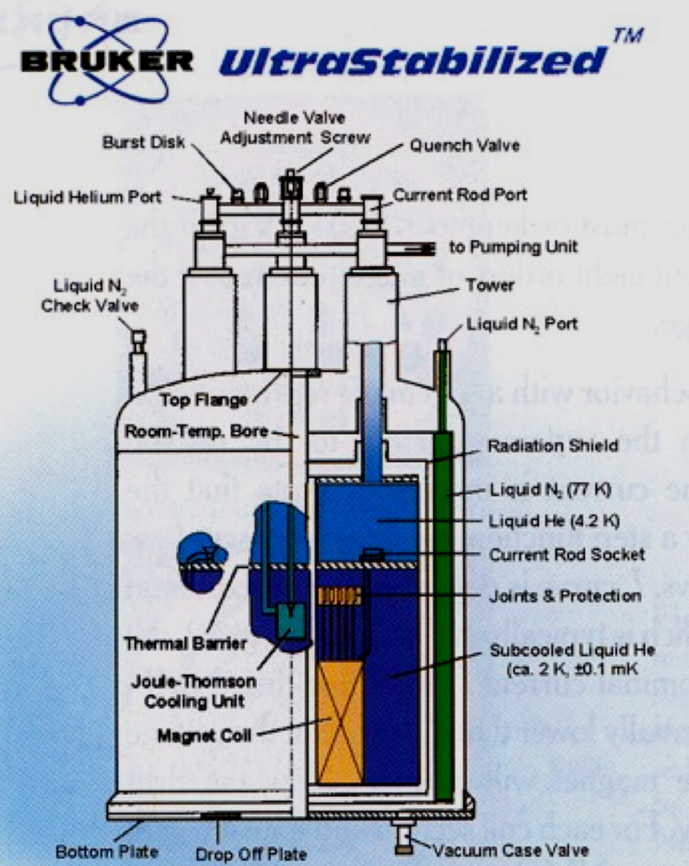
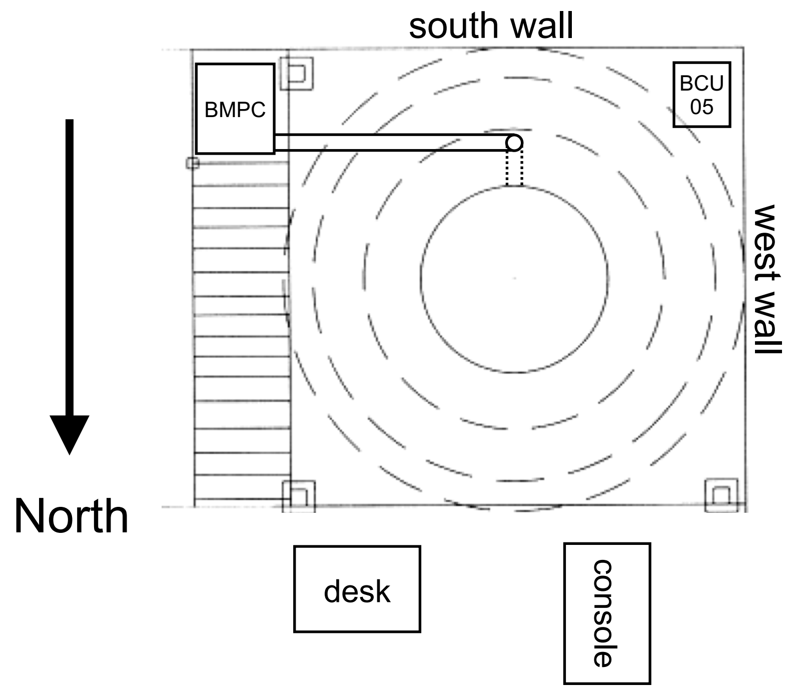
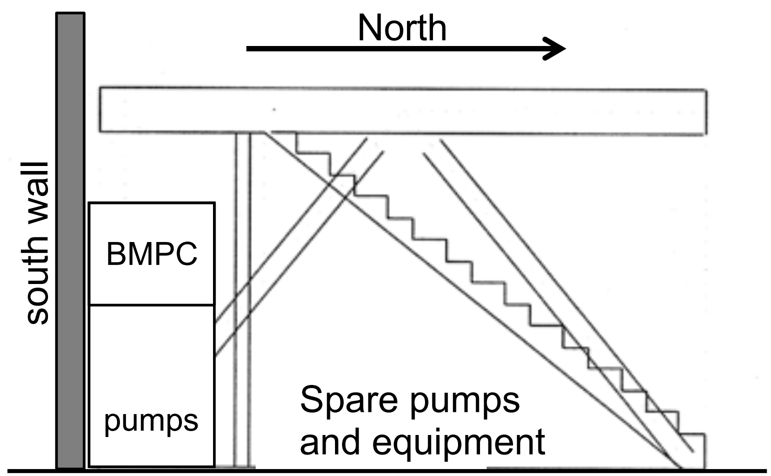


Fig. 9: Schematic of Bruker's UltraStabilized™ cryostat for subcooled ultra-high-field magnet systems.

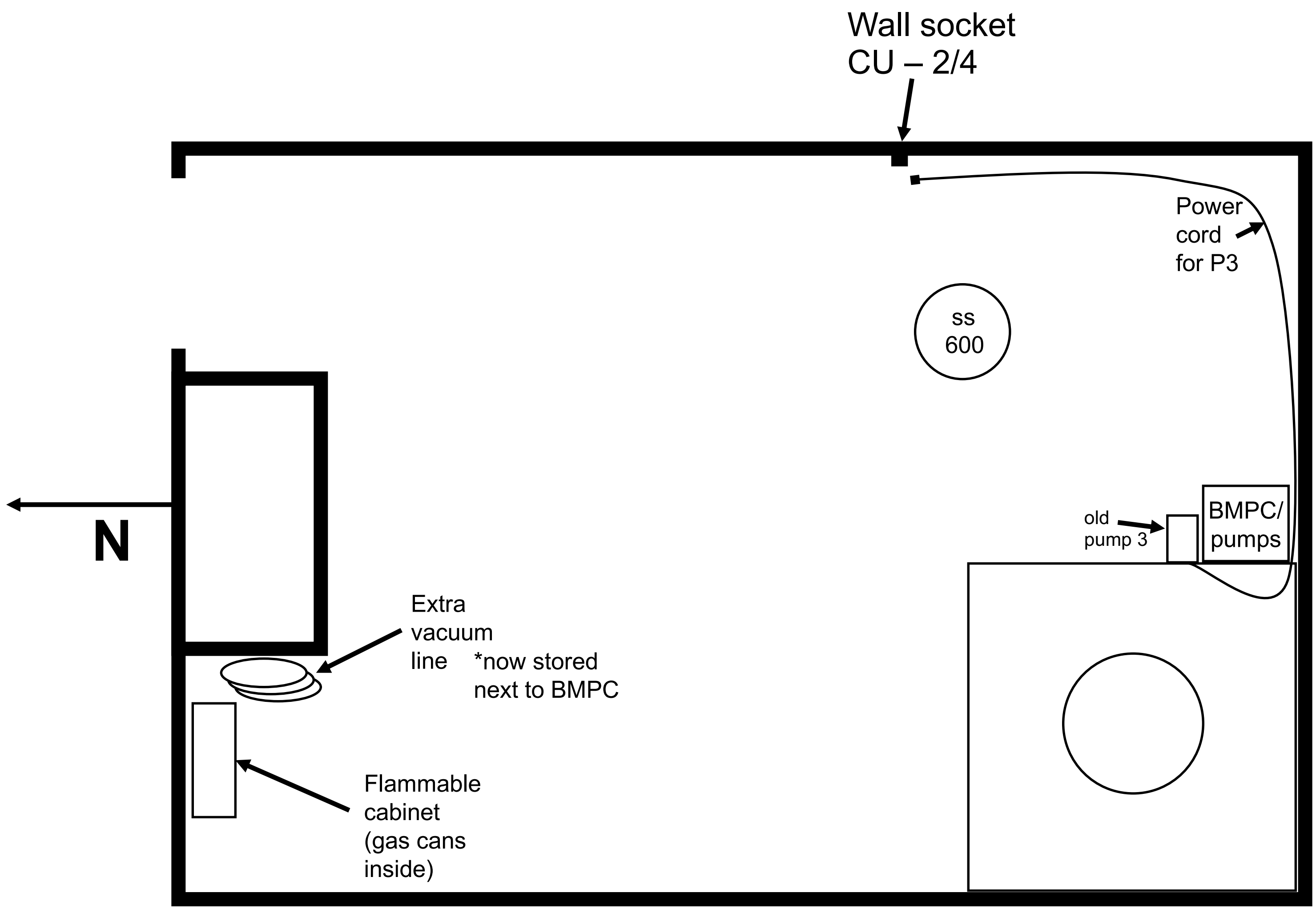
Top view of platform and equipment
Note: the north side of the platform has the same cross-bracing as does the east side (seen in the diagram below).





Side view of stairway

Room Diagram - showing relevant pieces of equipment

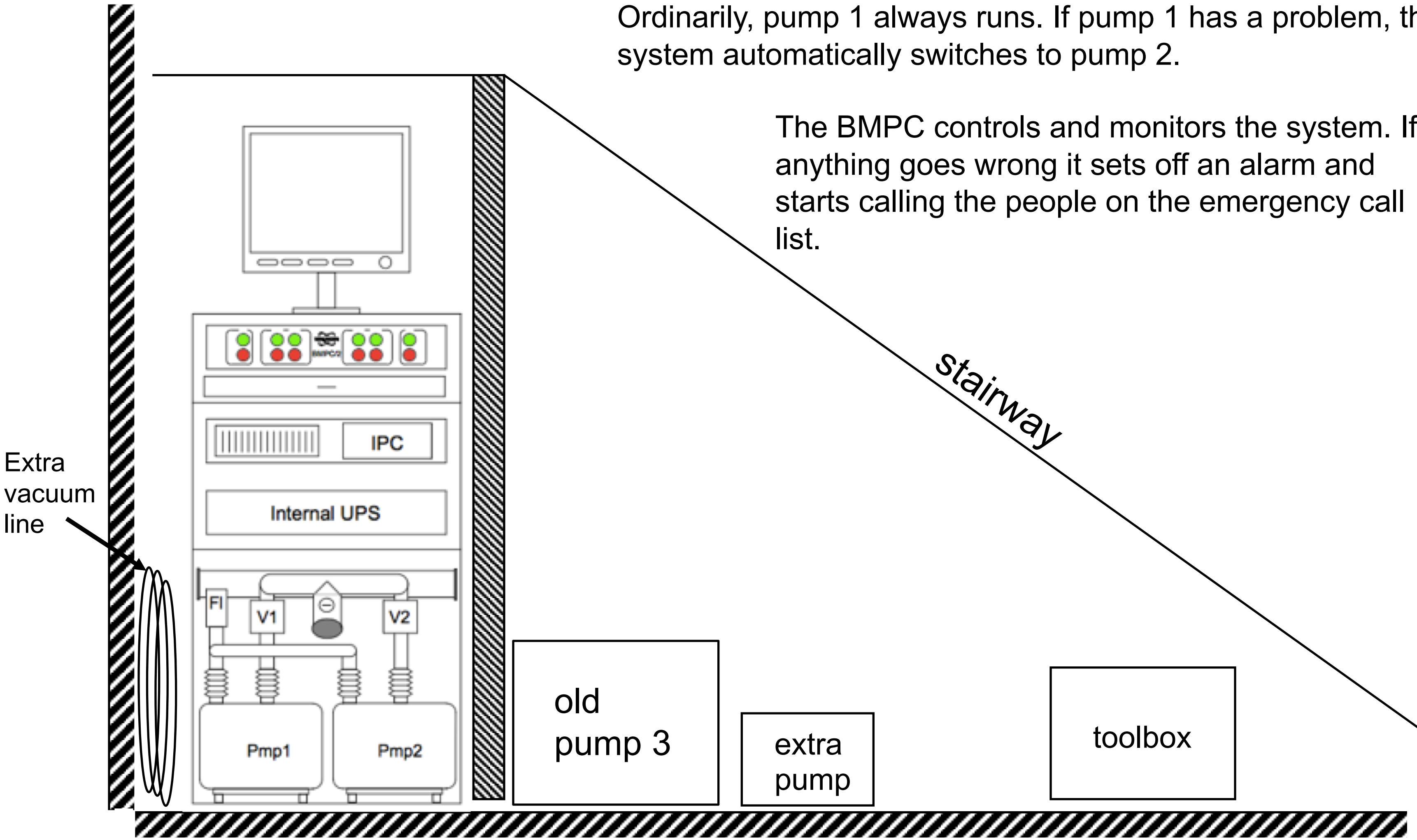


BMPC and pumps

One pump must always run otherwise the 800 will quench in 8-12 hours.

Ordinarily, pump 1 always runs. If pump 1 has a problem, the system automatically switches to pump 2.

The BMPC controls and monitors the system. If anything goes wrong it sets off an alarm and starts calling the people on the emergency call list.



If pump 1 and pump 2 both fail, the extra pump can be attached, or the old pump 3 can be attached and turned on manually by plugging it into the 220 volt wall socket (CU-2/4).

The new BMPC:

BMPCII

*BMPC:
Bruker Magnet Pump Control

The vacuum pumps are inside the BMPC2 cabinet:

Extra pumps and spare parts are under the stairway:

old pump 3

new spare pump

The software has several available displays.

Here is the “jumbo display”.

You can see typical values for the main parameters.

The temperatures are the most important (they are measured in Ohms because resistance is what’s actually measured). The higher the number, the lower the temperature.

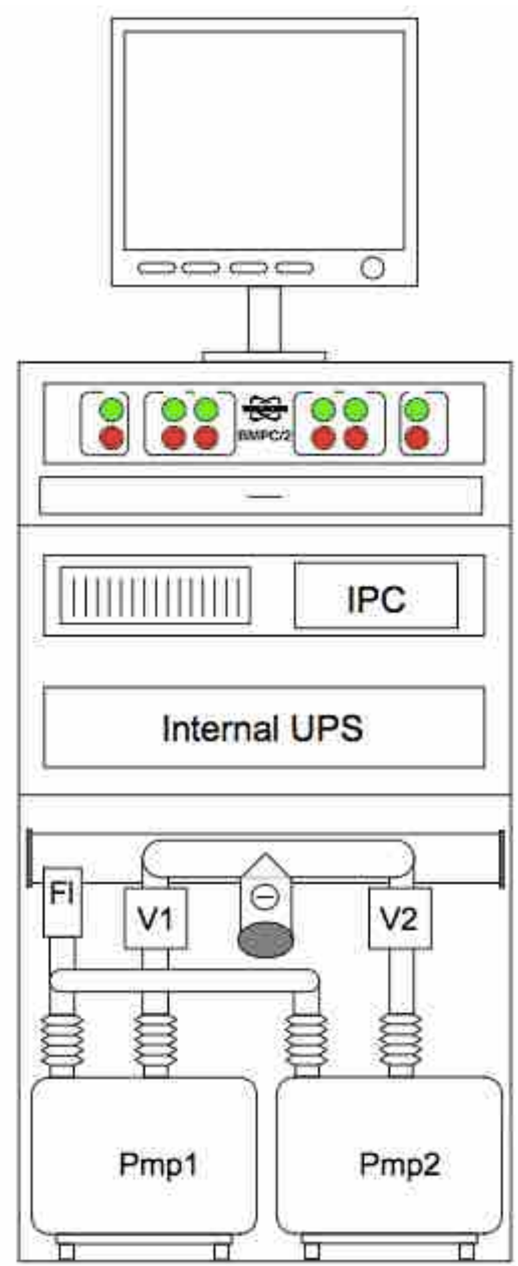
If T1 gets below 18143, and/or T2 gets below 13650, the magnet is in danger of quenching.

The BMPC monitors the system and controls the pumps.

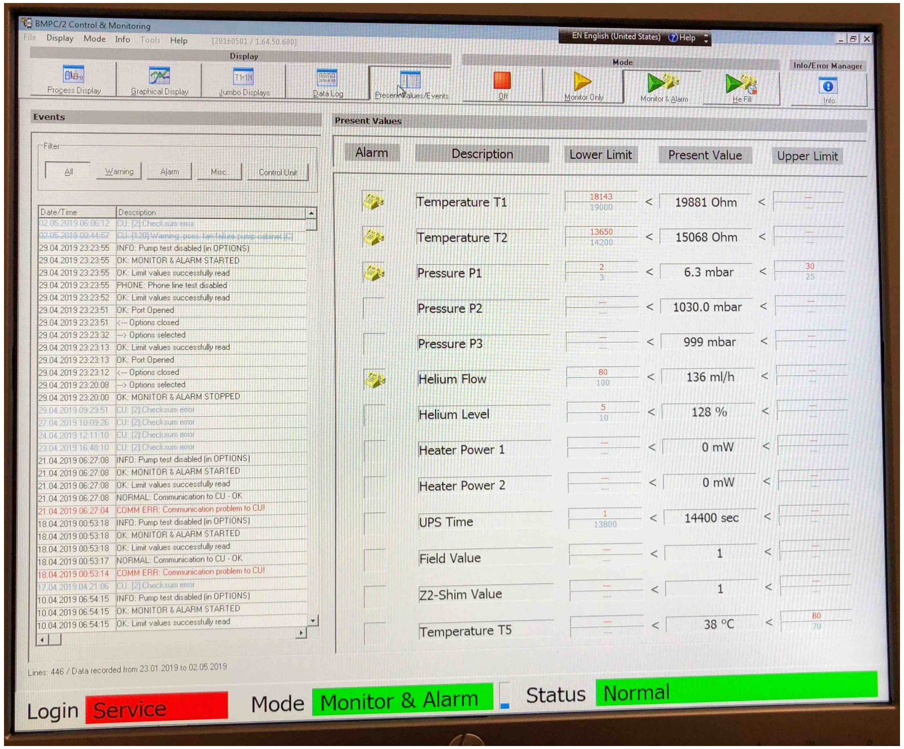
Normally, pump 1 is always on (and valve 1 is open). If there's a problem with pump 1, the BMPC will automatically switch to pump 2.

If any of the main parameters go outside their safe range, the BMPC will go into an alarm condition, notify Bruker, and start calling the people on the call list.

Most problems have easy solutions. But some are serious and may require exchanging a pump or taking other action.



The BMPC software is currently set so that it will activate the alarm in the following circumstances:



- Temperatures 1 or 2 are too high.
  (T1/T2 values are too low)
- Pressure 1 is too high or too low.
- Helium flow is too low

There are only a few causes for all these errors. The Bruker manual titled "BMPC II", located on top of the BMPC, next to the monitor, contains comprehensive descriptions of all possible errors, troubleshooting, and instructions on what to do.

A short description of this follows.

Main BMPC alarms:

<u>T1 or T2 too high.</u> These are temperatures measured at the bottom and top of the magnet coil, respectively.

Possible reasons: pump failure, closed valves at the pumping unit, leak in the pumping line, closed or blocked needle valve.

<u>P1 too high.</u> The pressure in the pumping line is too high.

Possible reasons: pump failure, closed valves at the pumping unit, leak in the pumping line.

<u>P1 too low.</u> The pressure in the pumping line is too low.

Possible reasons: butterfly valve on top of the cryostat is closed, closed or blocked needle valve.

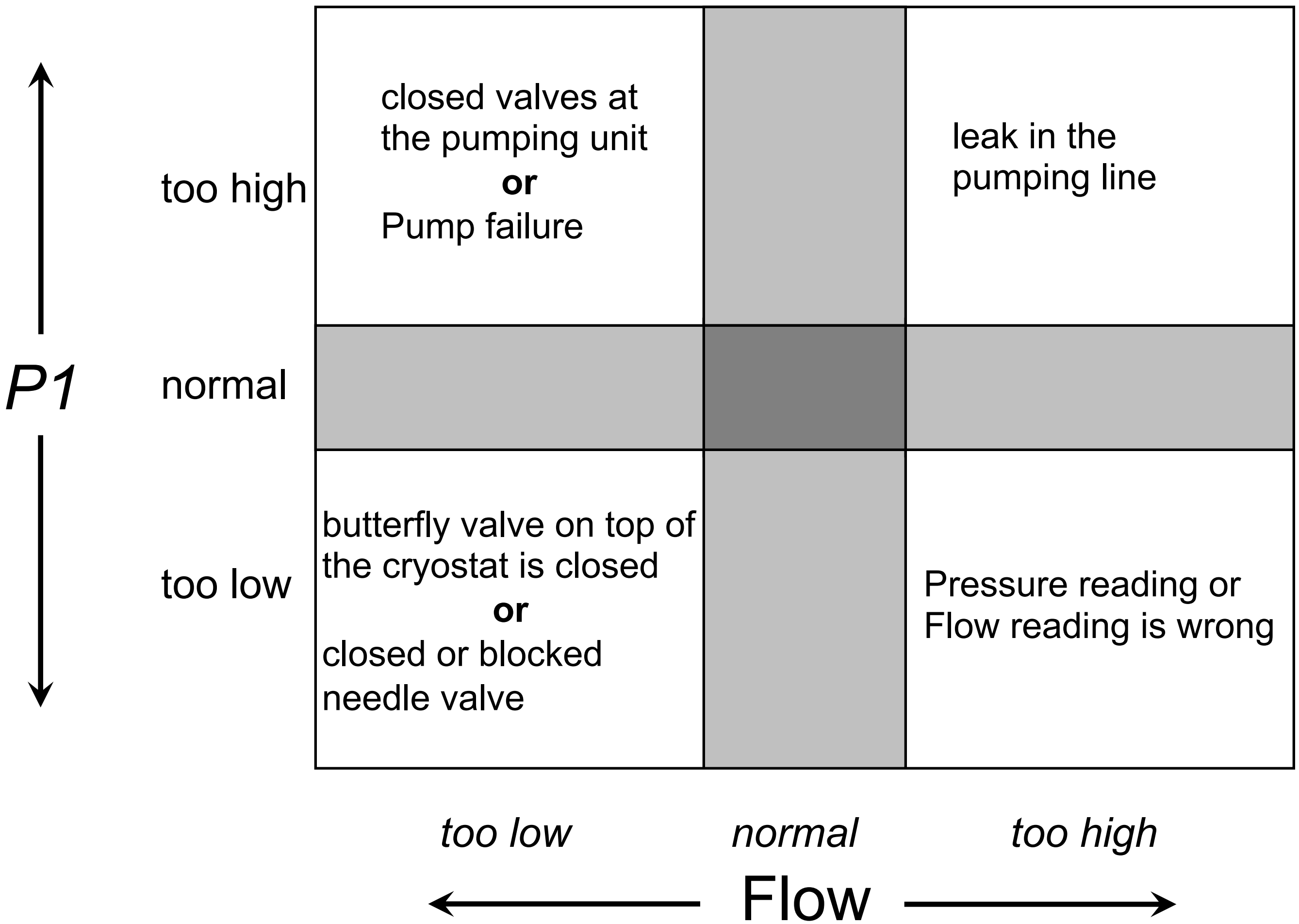
<u>He flow too low.</u> The He gas flow through the pumping line is too low.

Possible reasons: pump failure, closed valves at the pumping unit, closed butterfly valve on top of the cryostat, closed or blocked needle valve, **faulty He flow meter.*

There are only a few possible causes of pressure or flow errors:

A. Pump failure
B. Closed valves at the pumping unit
C. Leak in the pumping line
D. Butterfly valve on top of the cryostat is closed
E. Closed or blocked needle valve

Here is a chart to help you determine the cause of an alarm:

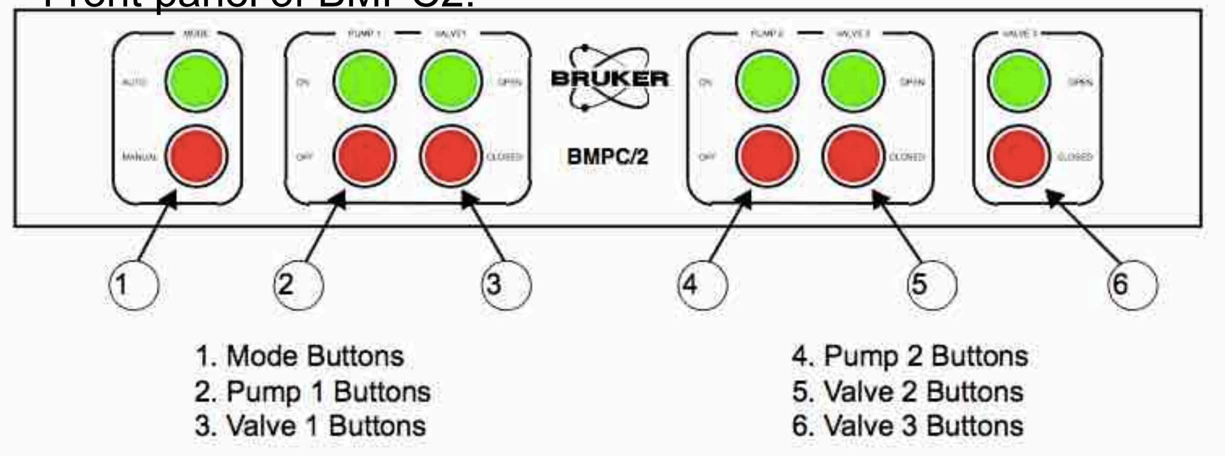


Once the cause of the alarm has been determined, you must remedy the problem. Here are the main possible causes and solutions:

Pump failure:

There are several possible causes for this. You must determine what the cause is, and remedy it.

Front panel of BMPC2:



One likely cause is that pump 1 failed and the system was in manual mode (or automatic mode failed). In that case you can just switch on pump 2 and valve 2. *If BMPC is in automatic mode, you must first switch to manual mode.

The procedure for turning on pumps/valves is: make sure pump1/valve1 are off. Then: **First** turn the pump on, wait ~30 seconds, **then** turn the valve on. No valve should ever be opened until the associated pump is on.

Pump failure:

There should always be two working pumps, so it should always be possible to fix this problem just by pushing the buttons.

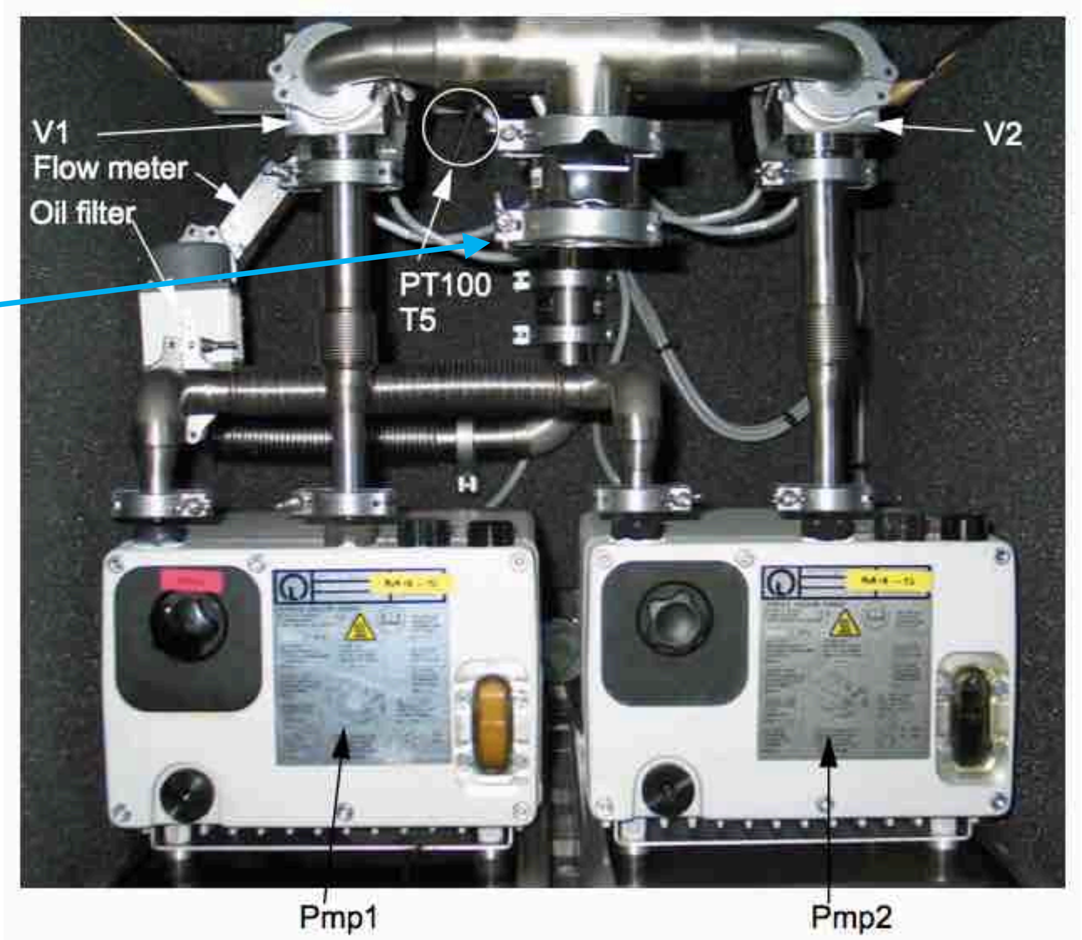
However, if you get really unlucky, and neither pump works, then you'll have to replace a pump:

There are detailed instructions on how to replace a pump in the manual.

You can also attach the spare pump (or the old pump 3) to the auxiliary attachment point.

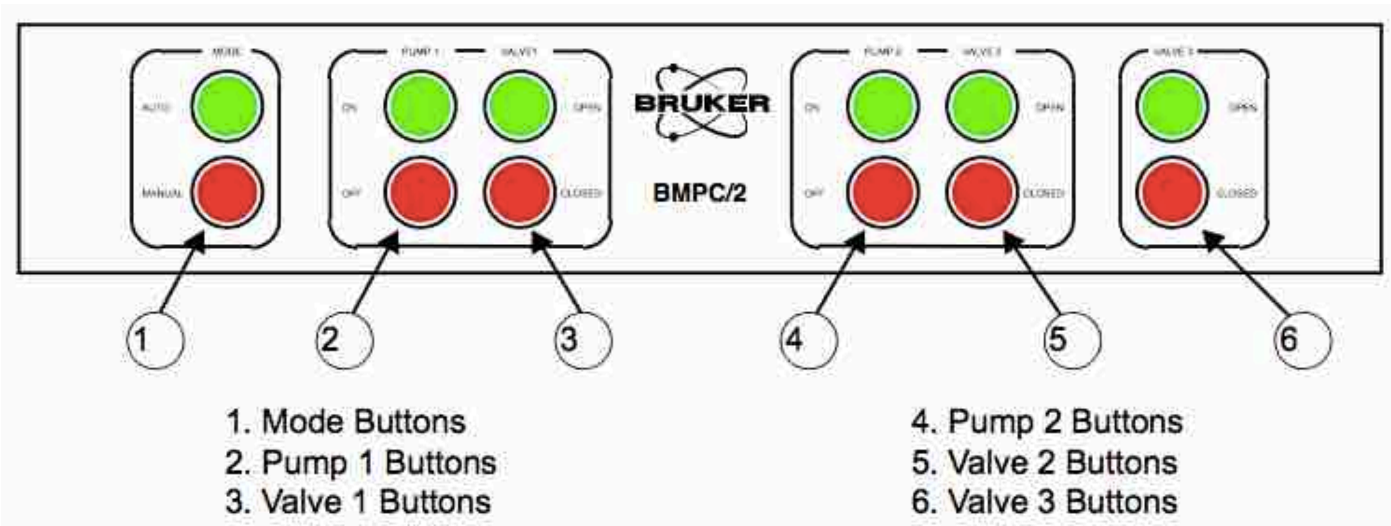
The old pump 3 may be the best option here. It's 4X more powerful, and it has a 50 foot power cord that can be plugged into outlet CU-2/4.

All manner of vacuum attachments are in the spare parts under the stairway.



Closed valves at the pumping unit

Switch the BMPC to manual mode (if it's in automatic mode). Follow the same procedures as on the previous pages.

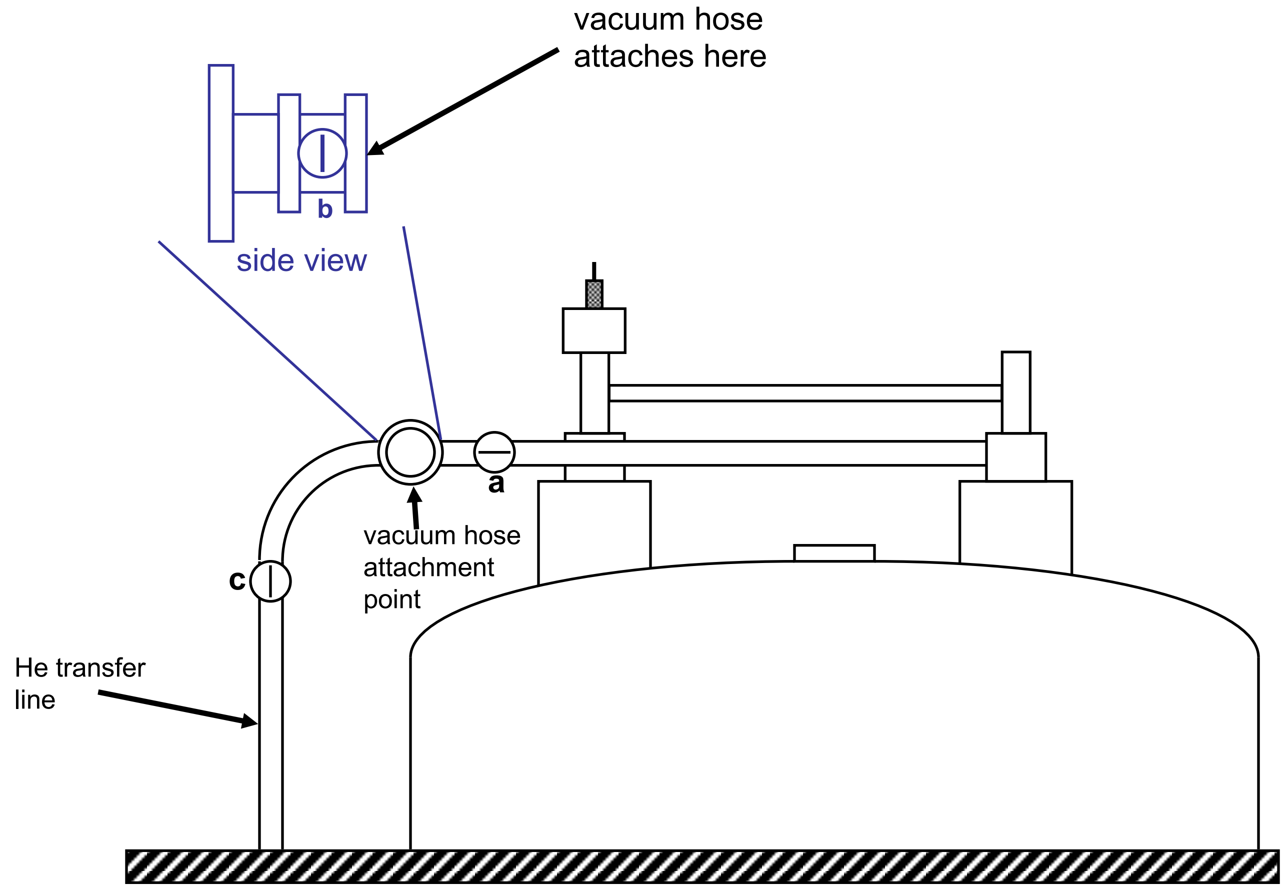


Determine which valve is closed. Try to open it. If it doesn't work, turn off the associated pump and try to start the other pump (turn it on, wait 30 sec, turn on its valve).

If this won't work, then again you'll have to attach one of the spare pumps to the auxiliary port.

Leak in the pumping line

If this is the problem, immediately close valve C (90 deg turn) as seen in the diagram below. This disconnects the cryostat from the pumping line, and will prevent ice blockage of the needle valve. Then you will have to install the temporary pumping line using the spare pump and the vacuum hose (next page).

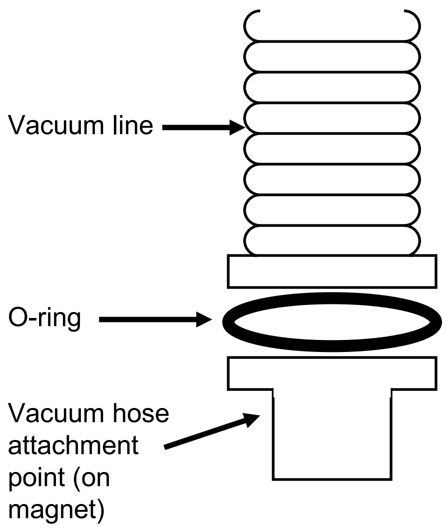


Top of magnet (as seen from the stairway)

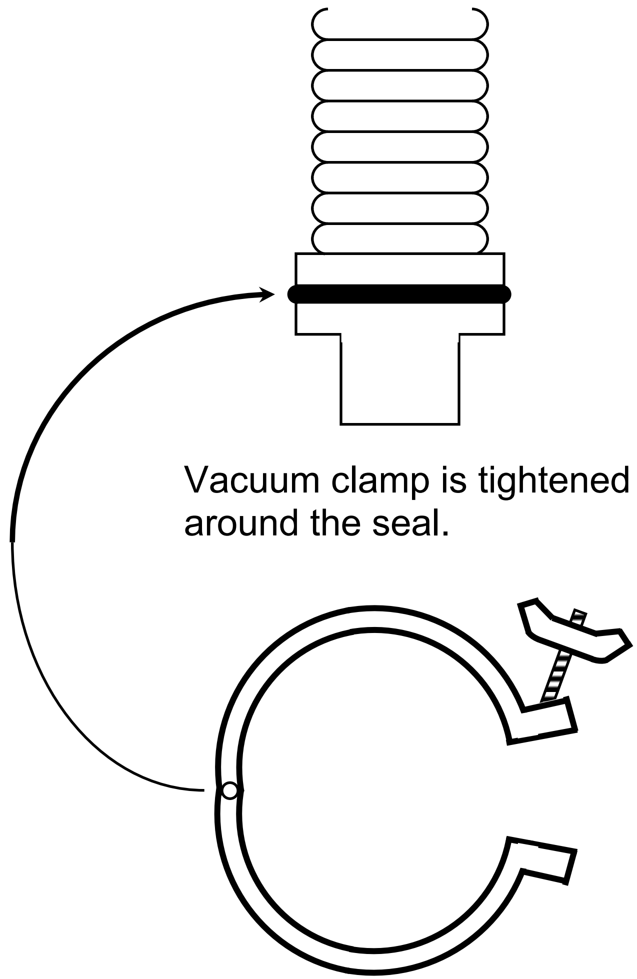
Connecting vacuum lines using vacuum clamps

All the vacuum line connections described above are made by connecting the two lines with an O-ring between, and clamping them together with a vacuum clamp.

Example: vacuum line attached to Vacuum hose attachment point.



The vacuum seals and O-rings should fit together snugly.

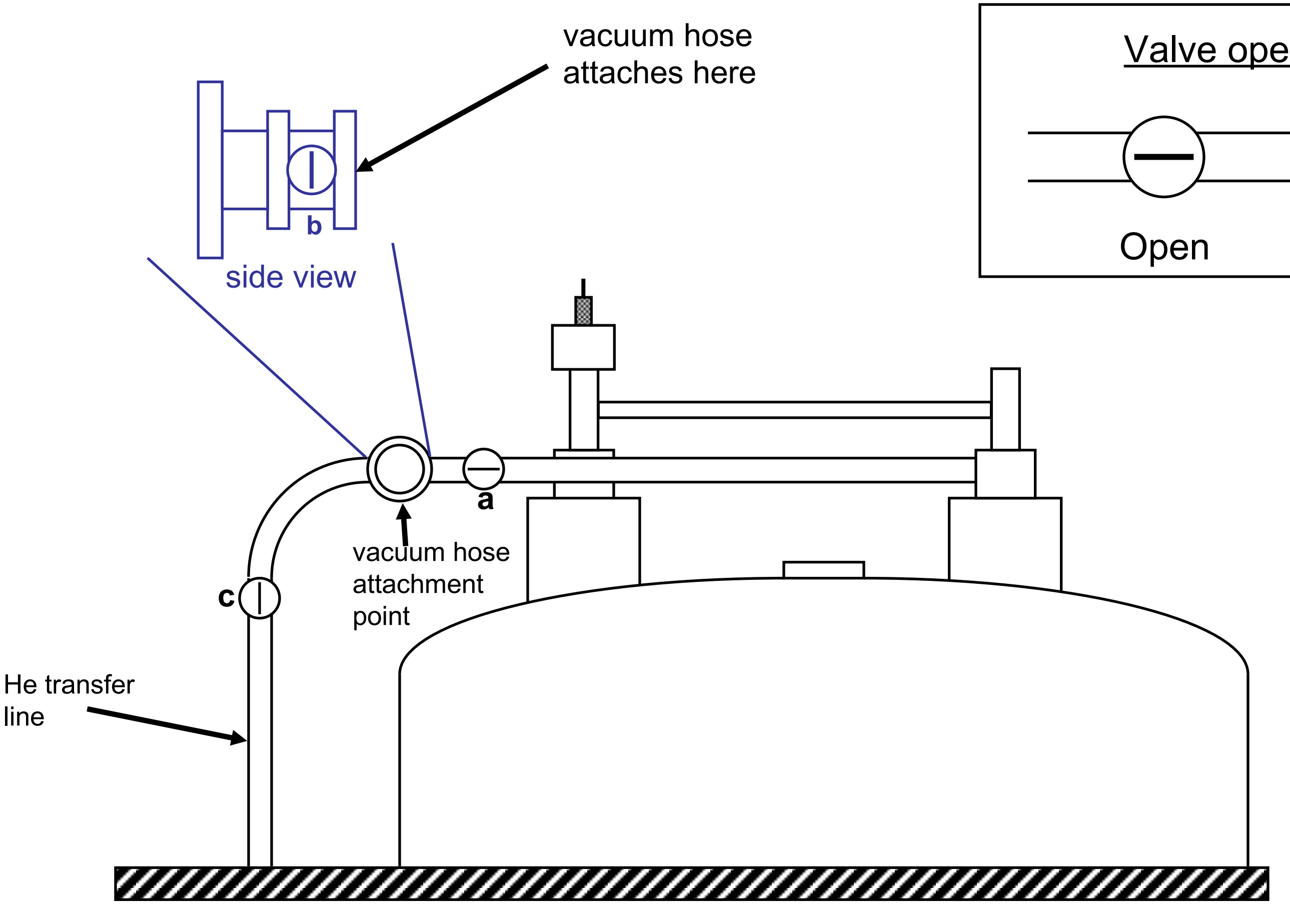


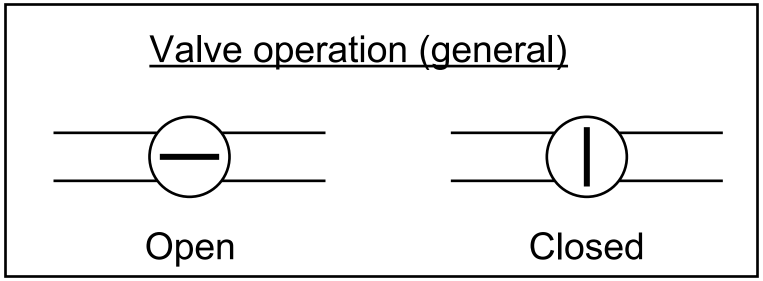
Vacuum clamp is tightened around the seal.

All necessary O-rings, vacuum clamps and adaptors can be found among the spare parts under the stairway.

Closed butterfly valve on top of cryostat

If this is the case, one of the butterfly valves (C or A in the diagram below) was manually closed. Reopen the closed valve(s).





Top of magnet (as seen from the stairway)

Closed or blocked needle valve

The needle valve controls the He flow through the Joule-Thompson cooling unit. Our system is set so that the He flow is around 135 ml/hour (there is a He flow meter in the BMPC). If the needle valve is closed, then the He will warm up and the magnet will quench, even if the pumps are running.

First, you want to have extra pumping capacity. Switch the BMPC to manual mode. Turn on pump 2, wait ~30 seconds, then turn on valve 2.

Open the needle valve completely (clockwise from the top is open), and see if the He flow increases (watch the flow gauge for five minutes or so). If the flow doesn't increase, close the needle valve all the way, then open it all the way again and watch the flow. Keep doing this until the flow increases.

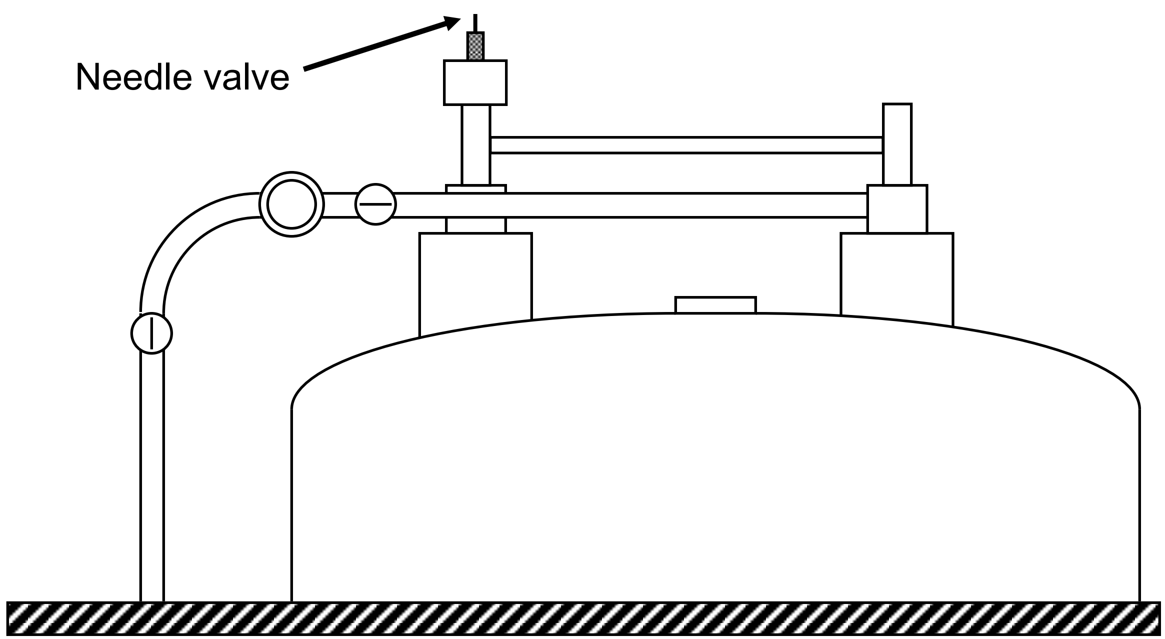
Once a high flow is reached (i.e., higher than 135), wait until the coil temperatures T1 and T2 reach values **lower than** standard operating conditions*.

*T1 and T2 are not displayed as temperatures, but as resistances: the higher the resistance, the lower the temperature. Under normal operating conditions, the values are:
VT1 ~19882
VT2 ~15070
→ So you should wait until VT1 and VT2 are **higher than** these values.

If the flow remains **above** the standard operating value, turn off valve 2, then pump 2. Watch the He flow for some time.

If the flow remains high, begin to slowly close the needle valve. Try to reach a point at which the flow is ~20% higher than the normal value, and leave it at this higher value.

**Whenever you turn the needle valve, watch the He flow gauge on the BMPC for about five minutes to assess the effect. It takes some time for the flow to respond to the change.



Top of magnet (as seen from the stairway)

If any of these errors occurs, you should first stabilize the magnet then call someone at Bruker for further instructions. The BMPC is supposed to fax Bruker Germany if any alarm goes off. So someone there should be trying to call the lab (unless it's the middle of the night).

Here are the contact numbers in the order that you should call them:

Bill Bartsch:	(978)987-6073 (cell) (978)667-9580 extension 5317
Bruker Germany	011 49 721 5161 0
Doug Lyons	(978)376-6375 (cell)
Bruker USA (Billerica)	(978)667-9580. #1 for NMR. Then #2 for service.
Bruker West Coast	(510)683-4300

UCLA Facilities Management, 24 hour emergency number: 825-9236. (in case the problem is a power outage)

<u>If the BMPC calls you</u>

First press the star button to acknowledge the call. It will then give you a status message with a detailed description. You must then enter the code: 2, 3, 4, 5. Enter the numbers slowly (one second in between each). You are now responsible for the system.