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Changing a Sample on ISIS Instruments

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CHANGING A SAMPLE ON ISIS INSTRUMENTS

(instructions for users)

by

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Contents

Abstract

Simple instructions for users of the ISIS instruments are presented to help them to change the sample in the different sample environment apparatus. A safe method of performing the important procedures is described.

HELP

These instructions are prepared to help experienced ISIS users after appropriate training to work on the instruments. These instructions **are not** for first time visitors. If you have any questions or problems during these operations then help is available from:

a) your local contact; this is the first person you should ask for help;

b) technicians associated with the spectrometer;

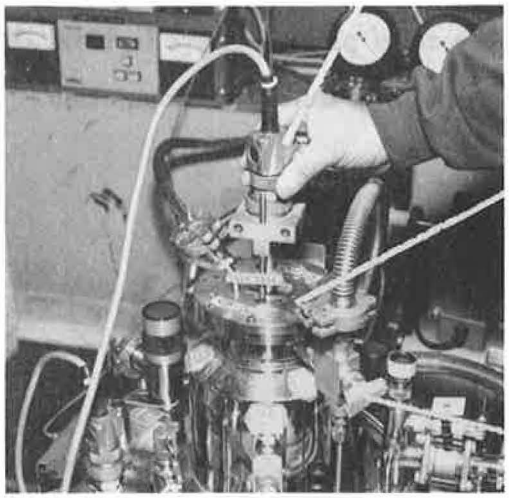
c) the Control Room during silent hours;

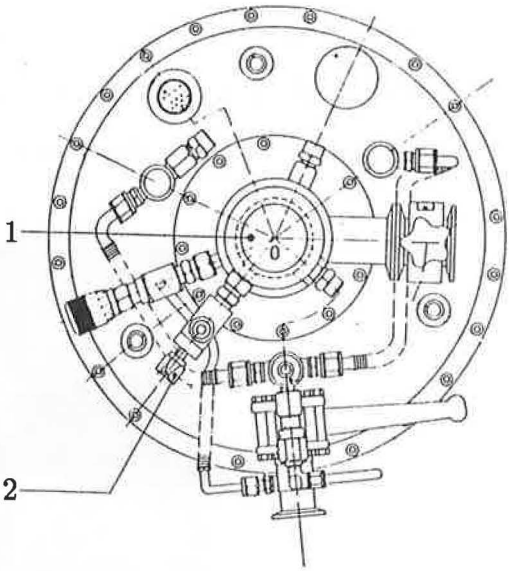
d) the instrument scientist if other help is unavailable.

The list of instrument scientists and technicians is given in the Appendix.

How to change sample in an orange cryostat

Withdrawing the centre stick





The relevant parts of the "orange" cryostat.

1.- The centre stick flange.

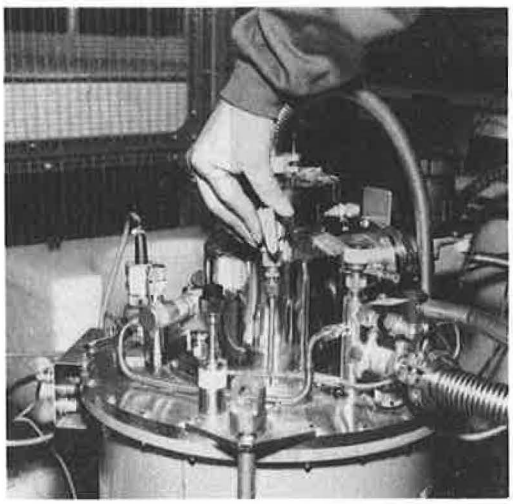
2.- The blue "Hoke" valve.

3.- The centre stick temperature sensor and heater connector.

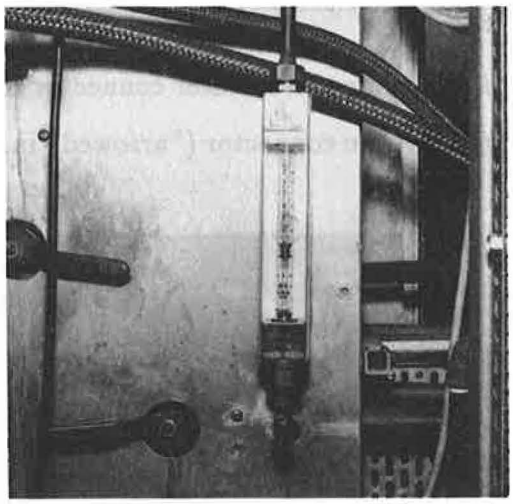
1. Before starting the following operations, ensure that the beam shutter is closed and that you have the necessary interlock key for the access to the sample.

2. Disconnect the temperature sensor and heater connector from the centre stick. It is important that the ring below the connector ("arrowed" in the photograph) remains always in place.

3. You must ensure at all times that air does not enter the cryostat sample volume. Therefore you have to fill the sample volume of the cryostat with helium gas. Turn the blue 'Hoke' valve pointer down. At very low cryostat temperatures (below 4K) it is first necessary to heat the cryostat to ~25K or bleed the helium in particularly slowly. (Failure to do this will cause a relief pressure valve to blow. Wasteful but not serious.) The flow meter will show no flow during this operation.



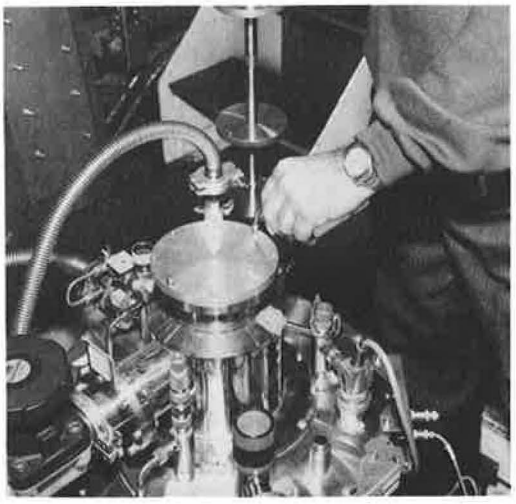
4. Wait until the flow meter again registers flow (about 5 min). The red float is spinning.



5. Remove the screws in the top flange of the sample stick, and have the blanking flange standing by.

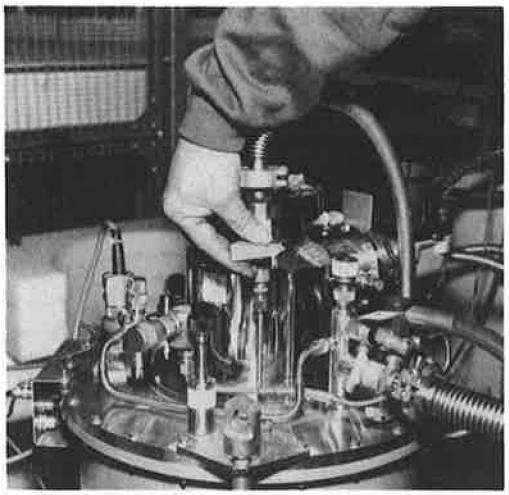
6. Quickly but smoothly remove the centre stick, and cover the sample volume with the blanking flange.

   If the sample can is damaged and loosing its contents, suspend the operation. Replace the centre stick in the cryostat and contact ISIS Control Room immediately.



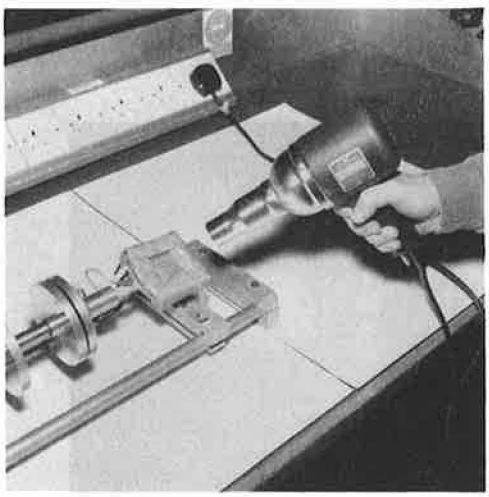
7. Replace the screws and tighten them.

8. Once the blanking flange is in place, the blue 'Hoke' valve should be returned to the horizontal (closed) position.



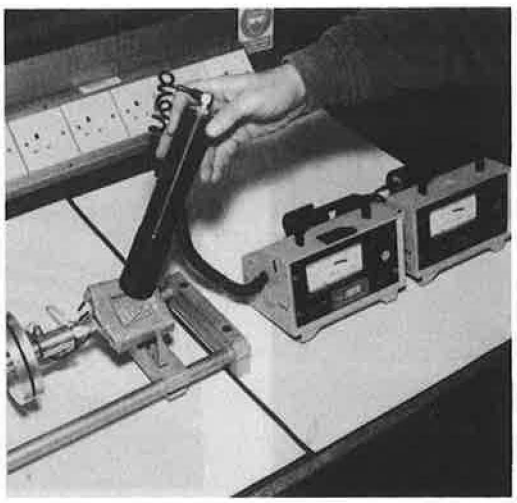
Removing sample

9. Use the hot air blower to bring the sample and centre stick to room temperature. The centre stick must be completely dry before replacing in the cryostat. This prevents freezing and subsequent blocking of the centre stick in the cryostat.



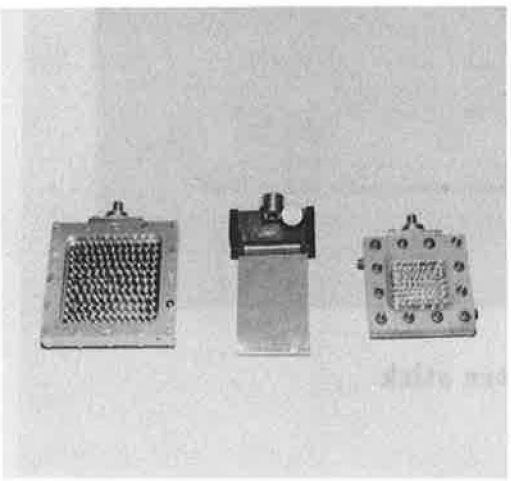
10. On removal, all samples MUST be monitored as soon as possible. Samples should be placed in the active sample cupboard if the radiation levels are below 75 μSv/h (50 $\beta-$counts/sec).

If radiation levels exceed these limits the duty officer MUST be called to monitor the sample and supervise sample handling.



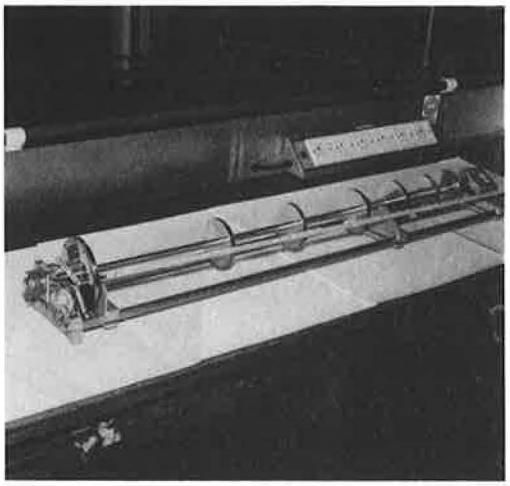
Loading sample

1. Put your sample in a suitable container (aluminium sachet, sample can). Usually sample holders have a standard M8 thread ("male" or "female"). If you want to use your own sample holder, ask your local contact for advice.

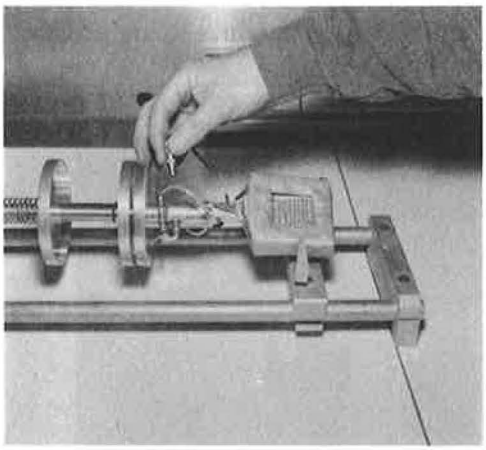


2. Attach the sample onto a centre stick and adjust the sample height. The height depends on the particular cryostat in use, and can be checked. On the side of the cryostat its length is indicated from flange to beam (centre) and to (cryostat) bot-

tom. The alignment frames for the centre stick have an adjustable pointer to help you.



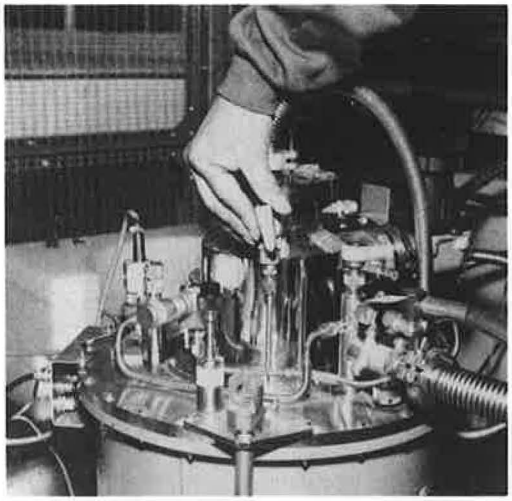
3. Attach temperature sensors (large, 4 pin plug), heaters (small, 2 pin plug) and the other auxillary equipment necessary for your experiment. Make notes of the sensor numbers which will be entered into the computer temperature-control table.



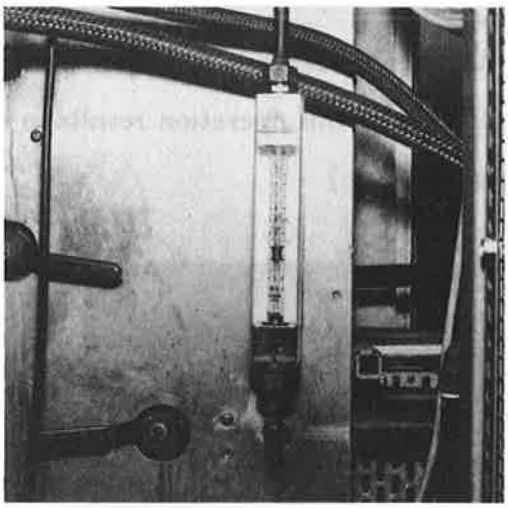
Introducing the centre stick

4. You must ensure as before that air does not enter the cryostat sample volume. Therefore refill the sample volume of the cryostat with helium gas before introduc-

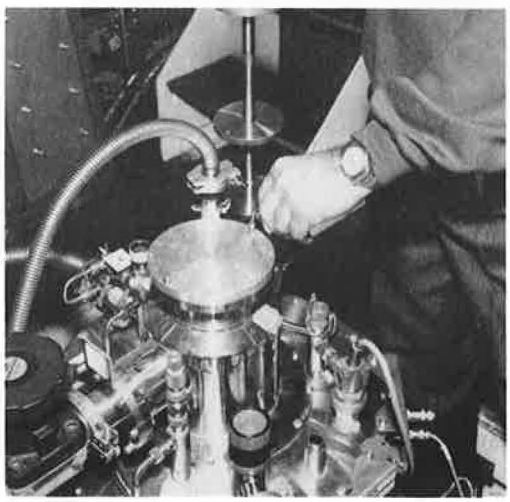
ing the centre stick. Turn the blue 'Hoke' valve to point down.



5. Wait until the flow meter again shows flow (about 5 min).



6. Remove the screws in the blanking flange, and have the centre stick standing by.



7. If you are not quenching your sample then go to no.11 below.

8. Fill the stainless steel dewar with liquid nitrogen.

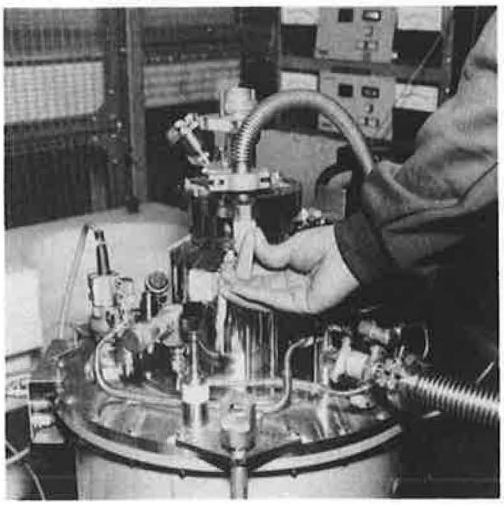
9. Immerse the sample in the liquid nitrogen and wait until the nitrogen stops boiling. (Waiting for a longer time in this operation results in a quicker overall time to achieve your working temperature.)

10. Quickly remove the centre stick from liquid nitrogen, shake off excess liquid <u>well but quickly</u>.

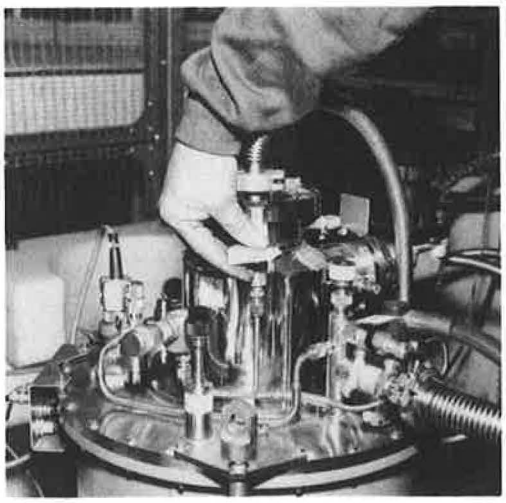
11. Lift the blanking flange and introduce the centre stick into the cryostat as quickly and as smoothly as possible.

12. Replace the screws and tighten them.

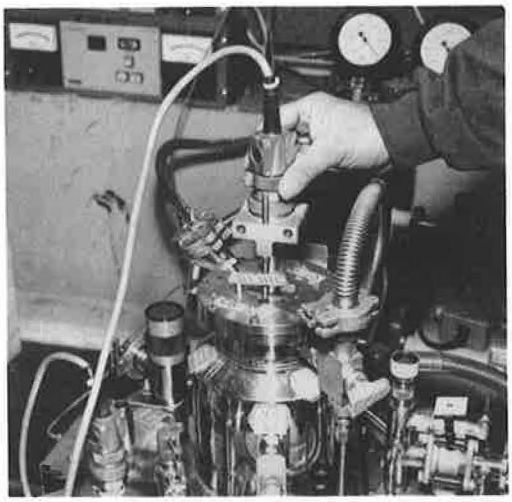
13. Turn the blue 'Hoke' valve to the up position and start the pump to evacuate the sample volume.



14. Pump down to the required exchange gas pressure (the vacuum gauge on the pump should show the residual pressure between 15 and 5 mbar). Return the blue 'Hoke' valve to the horizontal position. (Some users prefer to flush with helium gas via the blue 'Hoke' valve and pump down again to reduce possible contamination with nitrogen.)



15. Connect the temperature sensor and heater connector to the centre stick.



16. Check the sample alignement with respect to the beam, and push the stick all the way down.

17. Ensure that numbers for the temperature sensors in the computer are correct.

18. Open the beam and begin the measurement.

How to change sample in a bottom loading C.C.R.

Removing sample

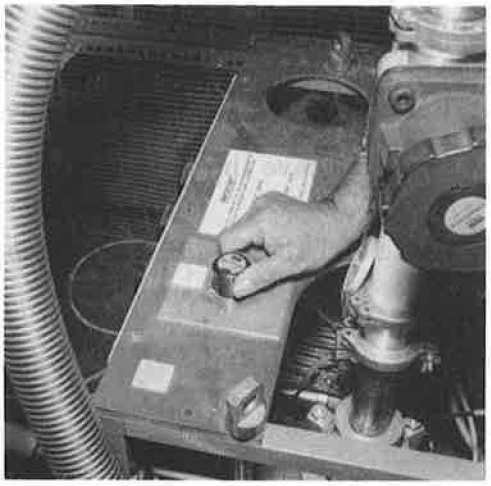
1. Before starting the following operations, ensure that the beam shutter is closed and that you have the necessary interlock key for the access to the sample.

2. Switch off the C.C.R. compressor unit.

3. Disconnect the temperature control and heater connectors from the C.C.R.

4. Switch Turbo Pump off and close the pumping unit valve. Vent Turbo by opening

vent valve on the pump for about 1 min. Close vent valve when Turbo stops.



5. Fill in the sample environment volume with air (or nitrogen where applicable).

6. Unscrew the bolts from the C.C.R. flange and lift the C.C.R. using the local crane and standard lifting equipment. (You must have been trained and have a RAL licence to use the crane. If not, seek HELP).

7. Warm the outer heat shield with a hot air blower and remove it from the C.C.R. Warm the C.C.R. with air blower till it is dry.
   If the sample can is damaged and loosing its contents, suspend the operation.

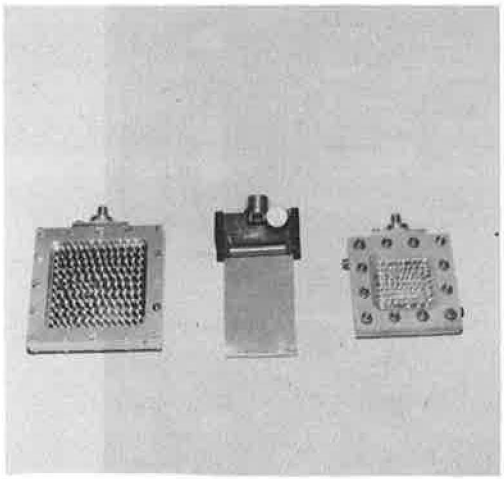
Replace the heat shield and contact ISIS Control Room immediately.

8. On removal, all samples MUST be monitored as soon as possible. Samples should be placed in the active sample cupboard if the radiation levels are below 75 μSv/h (50 $\beta-$counts/sec). If radiation levels exceed these limits the duty officer MUST be called to monitor the sample and supervise sample handling.

Loading sample

1. Put your sample in a suitable sample can. Usually sample holders have a standard M8 thread ("male" or "female"). If you want to use your own sample holder, ask

your local contact for advice.



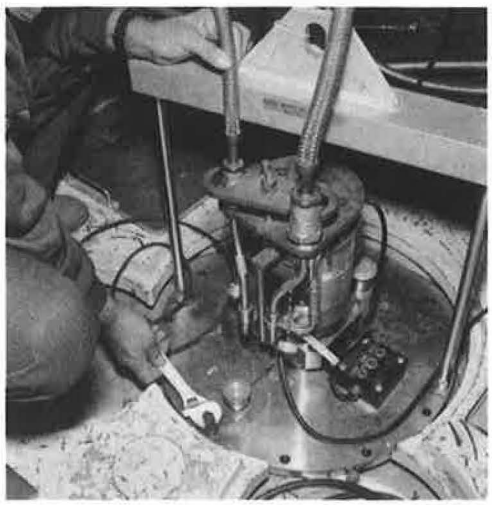
2. Attach the sample onto the C.C.R. saddle, ensure the orientation is correct.

3. Attach temperature sensors and other auxillary equipment directly onto the sample can. Make notes of the sensor numbers which will be entered into the computer temperature-control table.

4. Attach the heat shield, and ensure the correct orientation of the "windows" in the shield.

5. Load the C.C.R. into the beam using the local crane. (You must have been trained and have a RAL licence to use the crane. If not, seek HELP). Check the overall orientation with respect to the beam is correct.

6. Replace the bolts and tighten the C.C.R. flange.



7. Pump the sample volume by restarting the Turbo Pump and opening pumping unit valve. Wait until the vacuum gauge shows better than 10^{-3} bar.

8. Connect the temperature sensors (the 4 pin plug) and heater (the 3 pin plug) to the C.C.R.

9. Ensure that the temperature sensor numbers are correct in the computer.

10. Start the C.C.R. compressor.

11. Wait until the sample is at a required temperature.

12. Open the beam and begin the measurement.

Trouble shooting on a C.C.R. compressor.

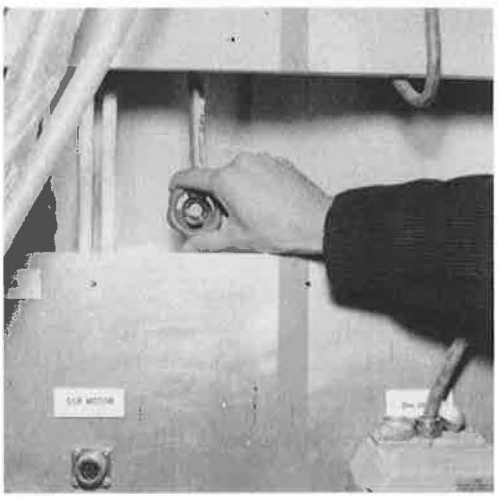
Some general troubles with the C.C.R. compressor which the experienced user can manage are presented below. All these events must be then reported to your local contact, instrument technician or instrument scientist. If the described recipes do not work seek HELP.

Compressor overheating

It may happen sometimes that the C.C.R. compressor stops because of overheating. The most obvious reason for this is the lack of cooling water. To restart the compressor do the following.

1. Switch off the C.C.R. compressor.

2. Check the water supply line and ensure that the water taps are fully open.



3. Reset the two buttons under the front panel of the compressor. (One button is slightly above and the other slightly below the centre of the hole.)

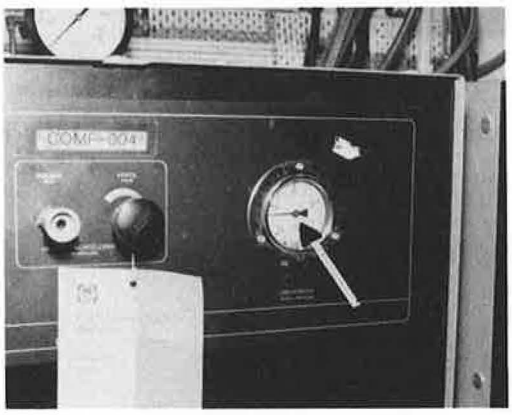
4. Switch on the compressor.

Lack of gas

This is the other common reason why compressors stop. To restart

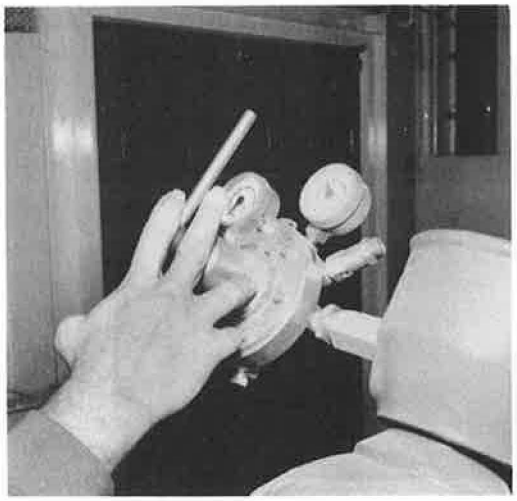
1. Switch off the compressor, and wait a few minutes.

2. Check the helium gas pressure in the compressor by reading the front panel pressure gauge. It should be between 14 and 16 bar when the compressor is stopped.

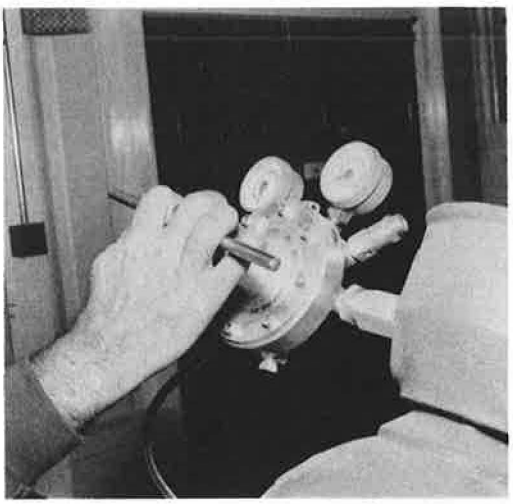


3. If the pressure is too low the compressor needs filling.

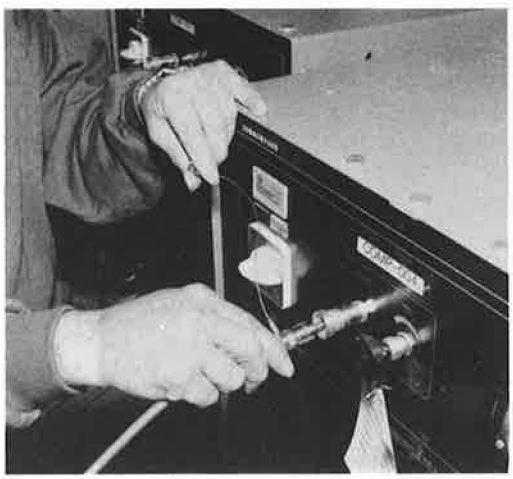
4. Set (completely unscrew) the pressure regulator to zero on the gas cylinder.



5. Open the gas cylinder valve and set the pressure regulator to 5 psi.

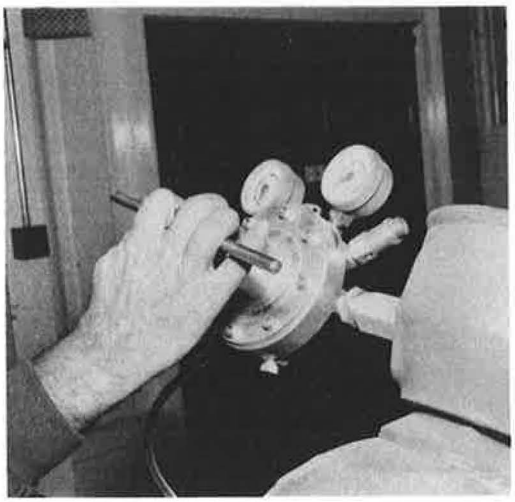


6. Purge the quick-release-coupling on the compressor with the gas from the charging line. To do this insert the coupling but do not push home, you will hear the gas escaping.

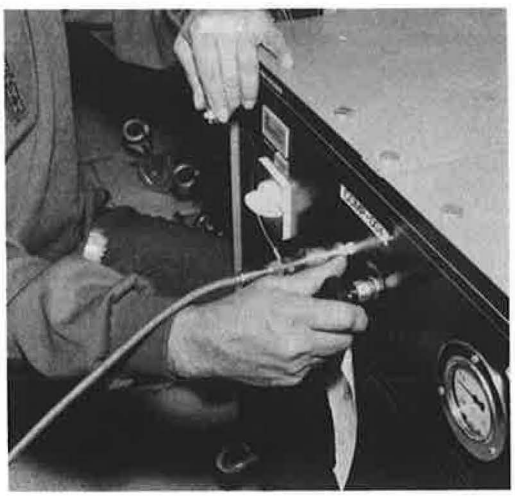


7. Fully insert the quick-release-coupling into the compressor.

8. Set the pressure regulator to 16 bar by slowly screwing in the torque bar.

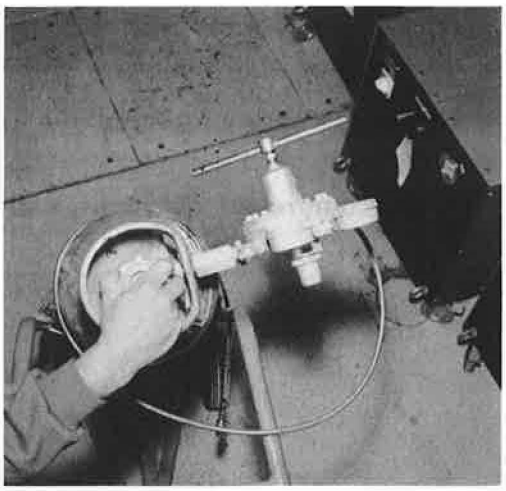


9. Open the black ("ventil") gas valve on the compressor front panel.

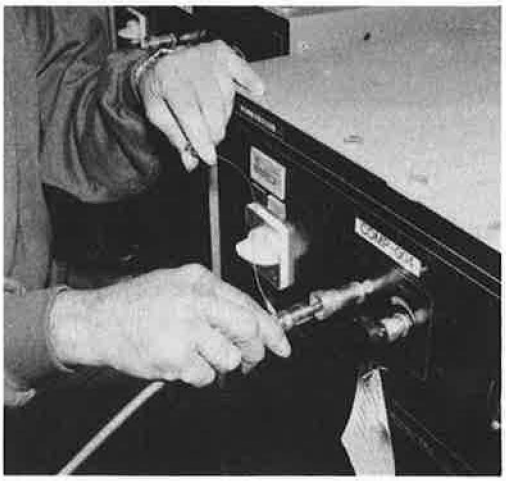


10. When the C.C.R. pressure gauge shows 14-16 bar close the black gas valve.

11. Close the gas cylinder valve and set pressure regulator to zero.



12. Disconnect charging line from the quick release coupling. For this, hold the charging pipe with one hand and with the other hand pull the quick-release-ring, away from the compressor.



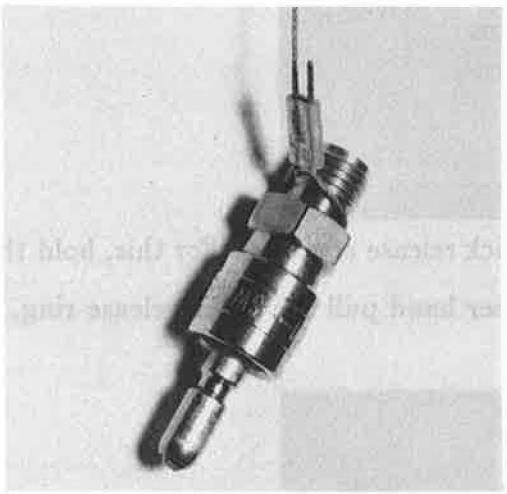
13. Reset the two buttons under the front panel of the compressor. (One button is slightly above and the other slightly below the centre of the hole.)

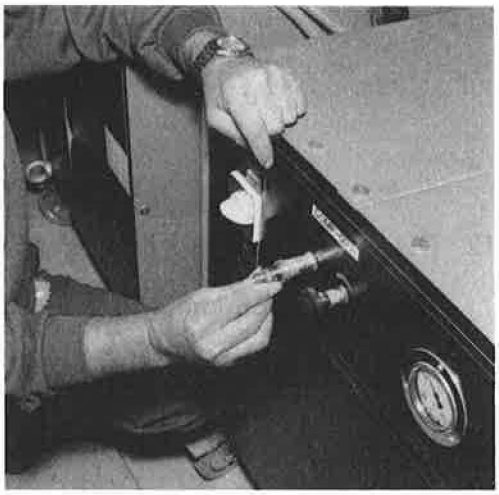
14. Switch on the compressor.

Excess gas pressure in the compressor

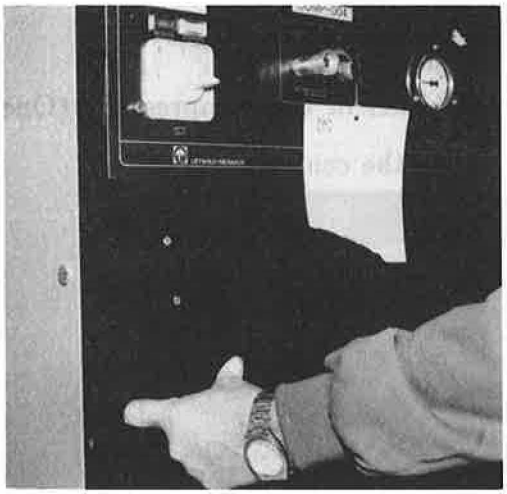
Compressors can also stop because of excess pressure. This happens if the pressure goes above 23 bar when working. To release the excess pressure

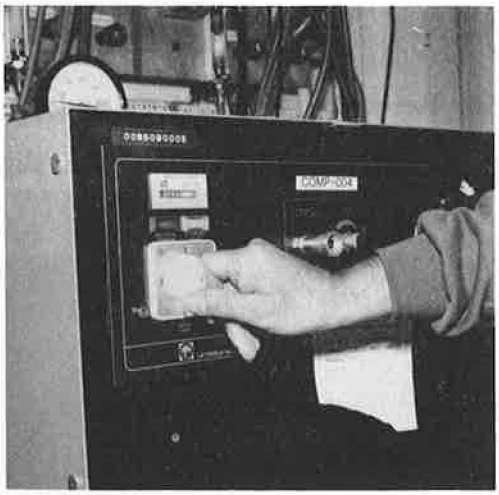
1. Insert the special fitting into the compressor gas input, and push home.



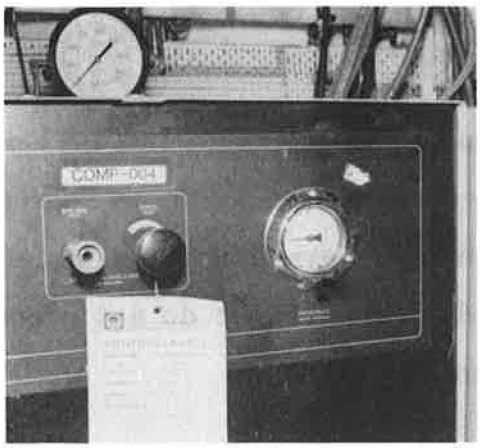


2. Reset the two buttons under the front panel of the compressor. (One button is slightly above and the other slightly below the centre of the hole.) and start the compressor.



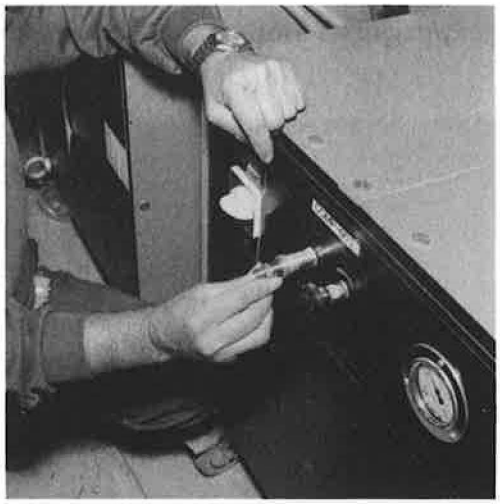


3. The pressure is read from the pressure gauge on the front panel.



4. Open the black gas valve on the compressor front panel and lower the pressure to ca. 21 bar. If the compressor cuts-out again: lower the pressure by 0.5 bar, close the black gas valve and start again from item 2.

5. Disconnect the special fitting from the compressor. For this, hold the fitting with one hand and with the other hand pull the quick-release-ring, away from the compressor.



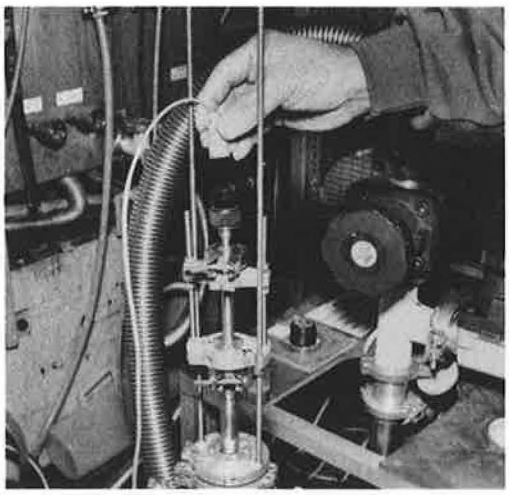
How to change a sample in a furnace

Removing sample

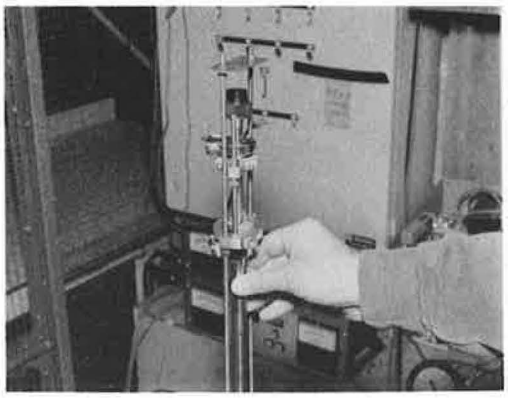
1. Before starting the following operations, ensure that the beam shutter is closed and that you have the necessary interlock key for the access to the sample.
   If you may have reason to believe that the sample can is ruptured and the sample is dispersed in the furnace do not proceed futher. Contact ISIS Control Room immediately.

2. Set maximum power for the furnace equal to zero using the computer command "CSET MAX_POWER1 0". This will ensure that the furnace will not be overheated during the sample change.

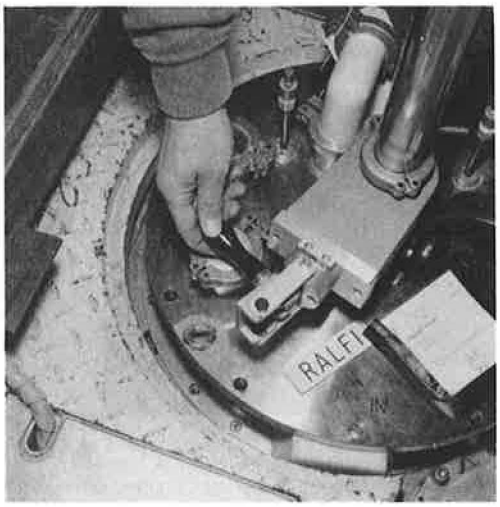
3. Disconnect the thermocouple plug from the sample centre stick. (Pull, don't unscrew.)



4. Lift the sample stick to the upper limit stop so that the sample is inside the detachable section of tube.



5. Close the vacuum gate valve on the top of the furnace. (If this doesn't close your sample has not been fully withdrawn.)



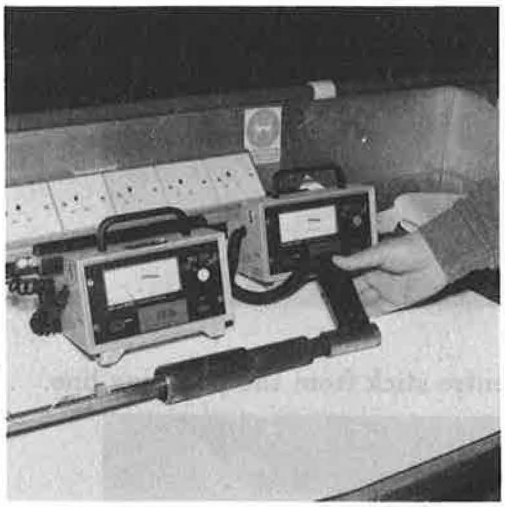
6. Close the vacuum valve which connects the pumping unit to the sample centre stick. Allow sample to cool for about 1/2 hour.

7. Disconnect the sample centre stick from the pumping line.

8. Disconnect the centre stick from the body of the furnace.

9. Remove the sample. (Note! The sample could be very HOT).

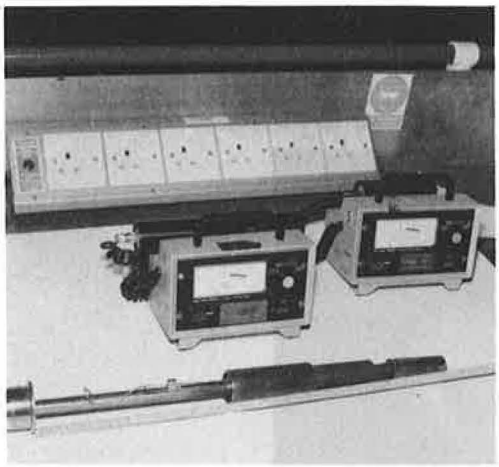
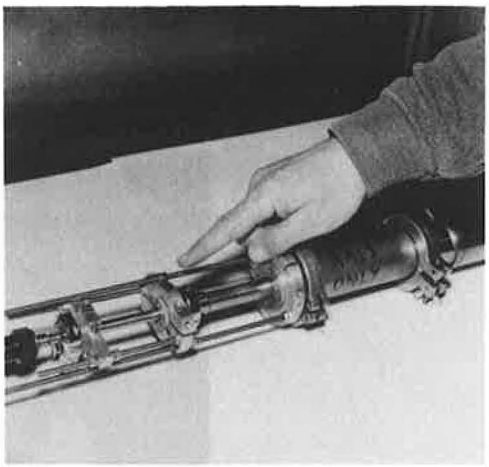
10. On removal, all samples MUST be monitored as soon as possible. Samples should be placed in the active sample cupboard if the radiation levels are below 75 μSv/h (50 $\beta-$counts/sec). If radiation levels exceed these limits the duty officer MUST be called to monitor the sample and supervise sample handling.



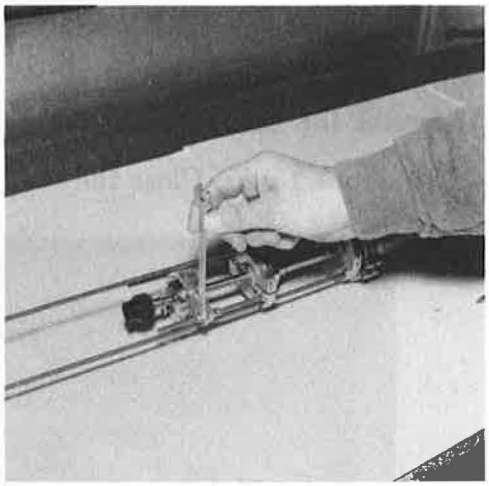
Loading sample

1. Put your sample in a suitable sample can. Usually sample holders have a standard M8 thread ("male" or "female"). If you want to use your own sample holder, ask local contact for advice.

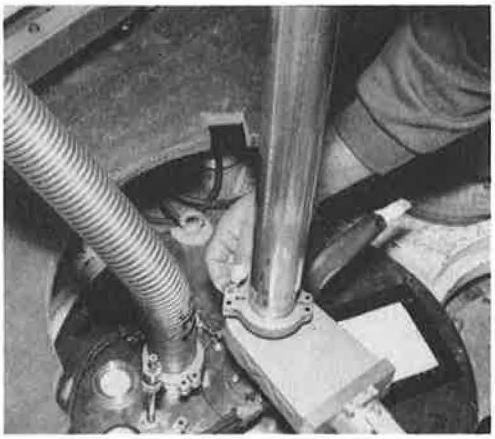
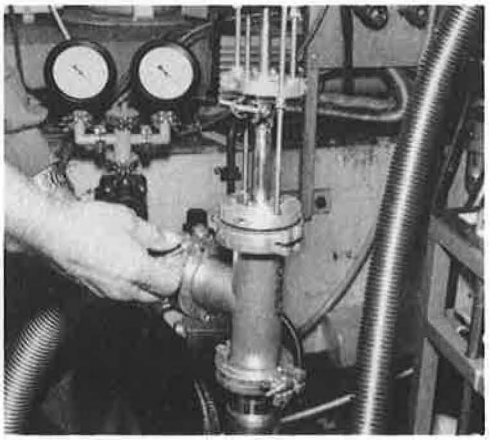
2. Attach the sample onto the furnace centre stick and adjust its height. Fix the desired height using the nuts on the leading threads.

3. Adjust the position of K-type thermocouple by first slackening off the nut(s) holding it to the centre stick.

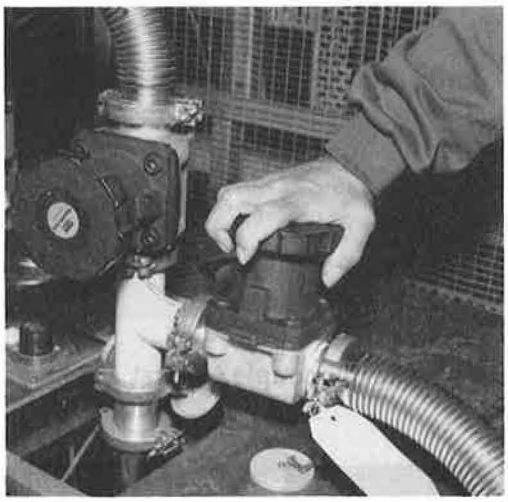



4. Connect the centre stick to the body of the furnace and connect the vacuum pipe to the centre stick.

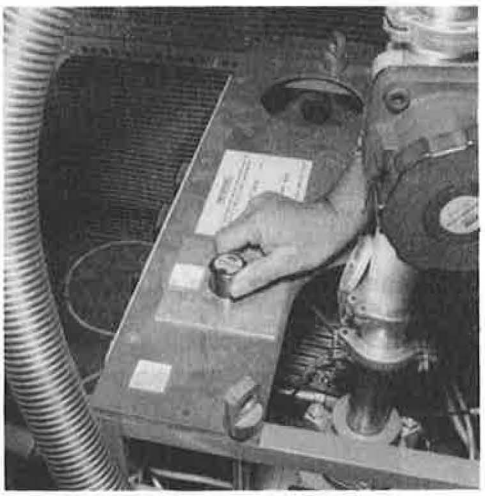



5. You must ensure that air does not enter the body of the furnace. This will irreversibly damage the heating elements. Therefore you MUST follow exactly the procedure described below.

6. Close the valve connecting the pumping unit to the body of the furnace.



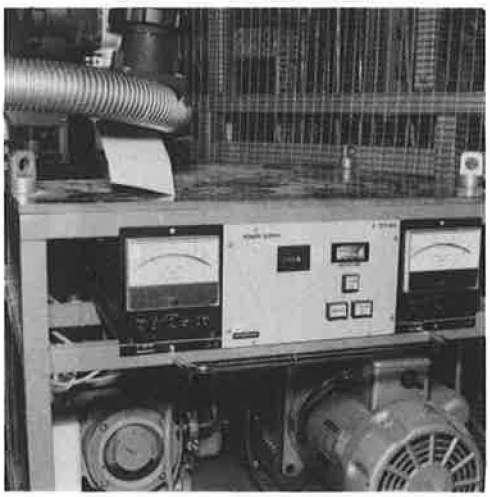
7. Switch the Turbo Pump off. Vent the Turbo by opening the vent valve on the pump for about 1 min. Close the vent valve.



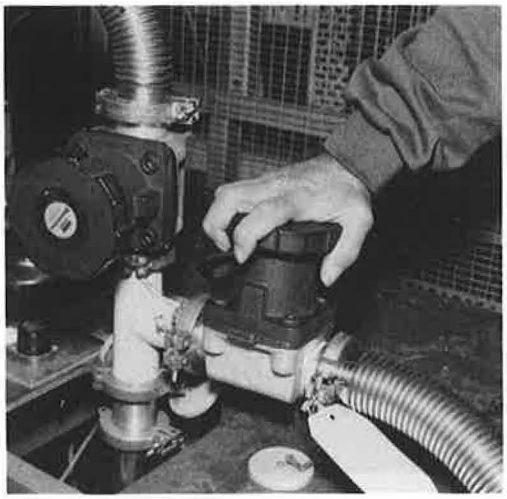
8. Open the valve connecting the pumping unit with the centre stick.

9. Start Turbo Pump.

10. Evacuate the centre stick until the vacuum is <u>better than 10^{-3}</u> bar.

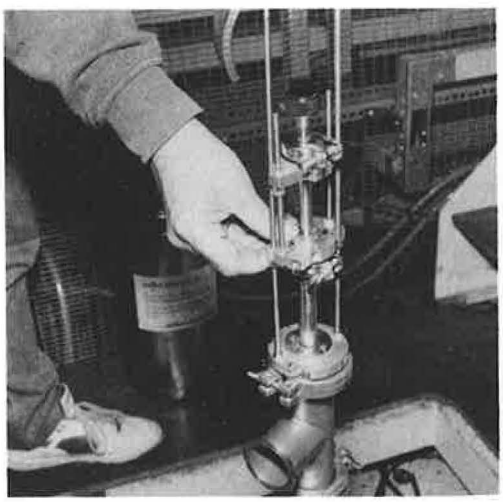


11. Open the valve connecting the pumping unit and the body of the furnace.



12. Open the furnace gate valve.

13. Lower the sample into the body of the furnace.



14. Connect the temperature sensors to the centre stick.

15. Enter the sensor number into the computer temperature-control table. (For the "K"-type thermocouple the correct number is "−2".)

16. Set the heating power to a desired value using a "CSET MAX_ POWER1 value" command.

17. Open the beam and begin measurements.

APPENDIX

Instrument	Instrument Scientist	Ext.	Bleep
CRISP	J Penfold	5681	187
	J Webster	5193	201
	D Bucknall	5871	
eVS	J Mayers	5882	185
HET	T.G.Perring	5352	
	R Eccleston	6437	161
	Z A Bowden	5683	165
HRPD	R M Ibberson	5871	206
	K S Knight	5220	
IRIS	C J Carlile	5684	180
	M A Adams	6157	210
LAD	W S Howells	5680	184
	A C Hannon	5358	
LOQ	R K Heenan	6744	183
	S M King	6437	242
MARI	S M Bennington	5193	241
	R Eccleston	6437	161
POLARIS	S Hull	6628	
	R I Smith	5683	
PRISMA	U Steigenberger	5145	207
ROTAX	H Tietze	5685	
	W Schmidt	5352	
SANDALS	A K Soper	5543	
	A C Hannon	5358	
SXD	D A Keen	6556	
	C C Wilson	5137	203
TEST beam	K Knight	5220	
TFXA	J Tomkinson	6686	
	M A Adams	6157	210
MuSR	C A Scott	5135	214
ISIS Main Control Room		6789	

Instrument technicians	Ext.	Bleep
J Chauhan	6182	189
J Dreyer	6502	
	5721	
M L Yates	6502	
	5721	234