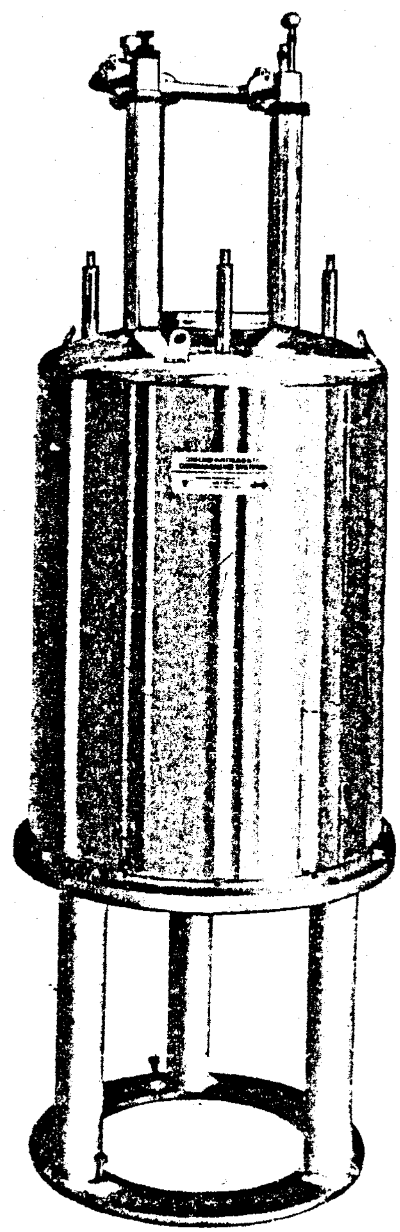
OXFORD
INSTRUMENTS

24

# Superconducting Magnets for NMR



OPERATING INSTRUCTIONS

45

SUPERCONDUCTIVE MAGNET
SYSTEM

Project No. W23462

IMPORTANT

Please read the manual before commissioning the system. It is possible to damage the cryostat and magnet if the correct procedures are not followed.

Oxford Instruments cannot accept responsibility for damage to the system caused by failure to observe the correct procedures laid down in the instruction manual.

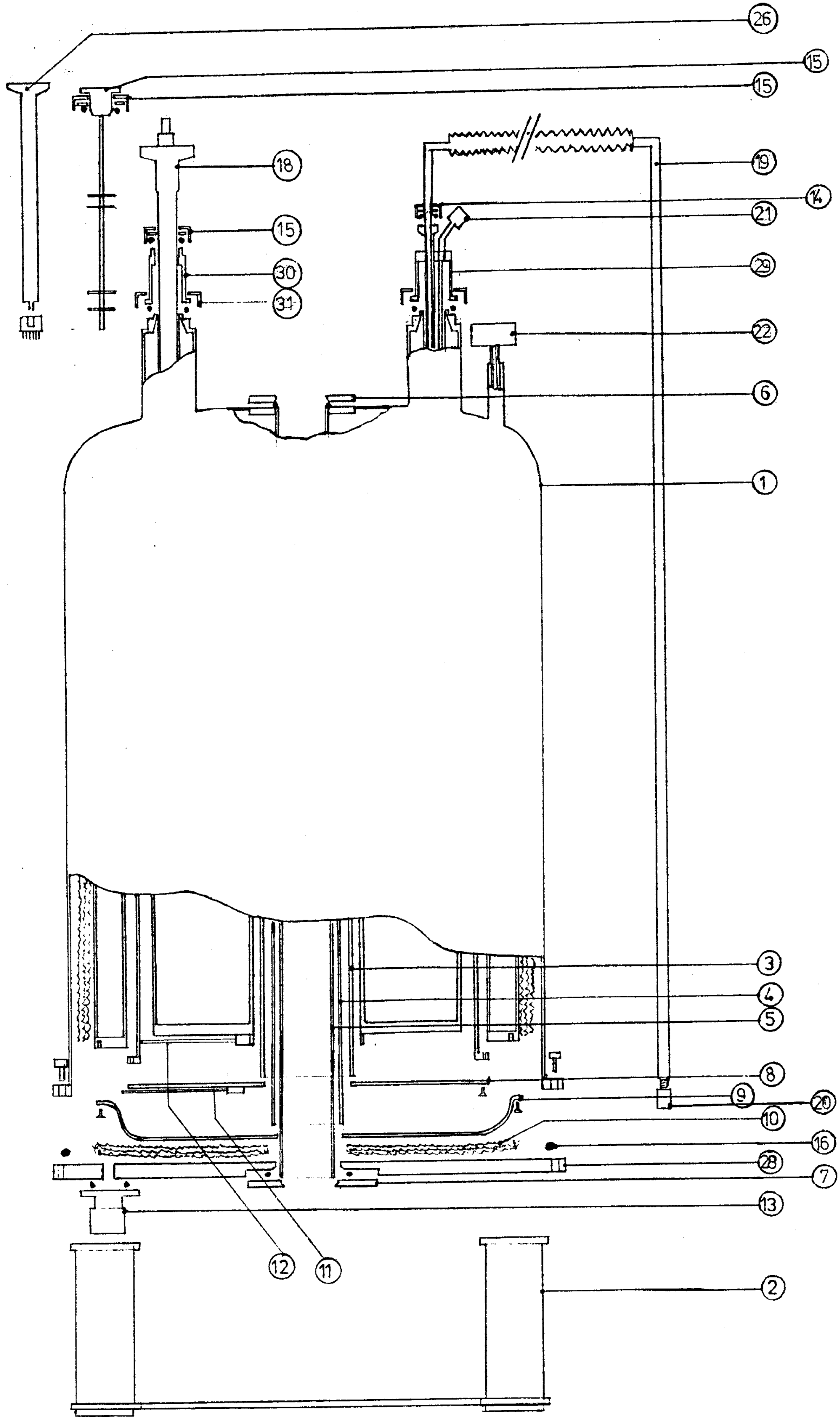
# N.M.R. MAGNET SHIPMENT CHECK-LIST

- [x] 1) Magnet and Cryostat
- [x] 2) Cryostat stand (3 off legs)
- [x] 3) Intermediate shield bore tube
- [x] 4) Nitrogen bore tube
- [x] 5) Room temperature bore tube
- [x] 6) Top closure flange
- [x] 7) Bottom closure flange
- [x] 8) Intermediate shield base-plate + 12 x M4 screws
- [x] 9) Nitrogen base-plate
- [x] 10) Superinsulation pad
- [x] 11) Fibre-glass spacer rods (3 off nitrogen shield-intermediate shield)
- [x] 12) Fibre-glass spacer rods (3 off intermediate shield to helium can)
- [x] 13) Evacuation valve
- [x] 14) Plug, washer, 'O' ring and nut for syphon entry port 2 off (one set spare)
- [x] 15) Radiation baffles, nut, 'O' ring and washer, (2 off each nut 'O' ring and washer)
- [x] 16) Large 'O' ring for base plate (R81200)
- [x] 17) Snorkel pressure relief valve (1 or 2 off as appropriate)
- [x] 18) De-mountable current leads (30 way or 18 way as appropriate)
- [x] 19) Transfer syphon and brass end nozzle
- [x] 20) Phase separator 2 off (1 spare)
- [x] 21) Helium level probe and meter (110v)
- [x] 22) Nitrogen level probe and meter (110v)
- [x] 23) Nozzle and Leybold (NW25) for evacuation of He can (2 off) and O.V.C. including end caps
- [x] 24) Nitrogen blow out tube
- [x] 25) ST8 syphon evacuation fitting

- [x] 26) Shorting plug and tool (30 way or 18 way as appropriate)
- [ ] 27) Shorting lead for de-mountable leads
- [x] 28) Main base plate
- [x] 29) 'Top hat' assembly including helium level probe and syphon entry
- [x] 30) 'Top hat' assembly, de-mountable lead entry
- [x] 31) 'Top hat' securing nut, 'O' ring and washer (2 off)

Miscellaneous items

- [x] Aluminium tape
- [ ] 3 off plastic/rubber tube for nitrogen exhaust
- [ ] 1 off plastic/rubber tube for helium exhaust
- [x] Manuals
- [x] Pressure relief valve - nitrogen exhaust
- [x] Check valve - helium exhaust
- [x] Dip stick.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# Fitting of Charcoal Sorb

A charcoal sorb is supplied with cryostats and should be fitted as follows.

a) The sorb consists of a layer of activated charcoal fixed to a 2" aluminium disc. The disc has a bolt through the centre which is approximately 1" long. A spacer is held onto the disc so that a circular gauze pad is trapped beneath it over the top exposed surface of the charcoal. To activate the charcoal it is necessary to bake it in an oven at approximately 110°C for several hours. For best results the sorb should be evacuated while warm. Adequate performance is usually obtained if the sorb is not evacuated while warm, but used very soon afterwards.

   Do not open the plastic container until ready to use.

   Avoid damp conditions, water will very quickly degrade the performance of the charcoal.

b) When filling the sorb to the base of the helium can first remove the nut securing the spacer onto the central stud.

   Do not invert the sorb as the charcoal is loosely held and may fall from the disc.

   Leave the spacer and gauge in place and screw the stud into one of the trapped holes used for the transit bungs.

c) Assemble the cryostat in the usual manner and begin pumping as soon as possible.

If the cryostat vacuum is ever to be broken it is important to use <u>dry</u> nitrogen gas so that water does not condense onto the sorb.

SAFETY PROCEDURES

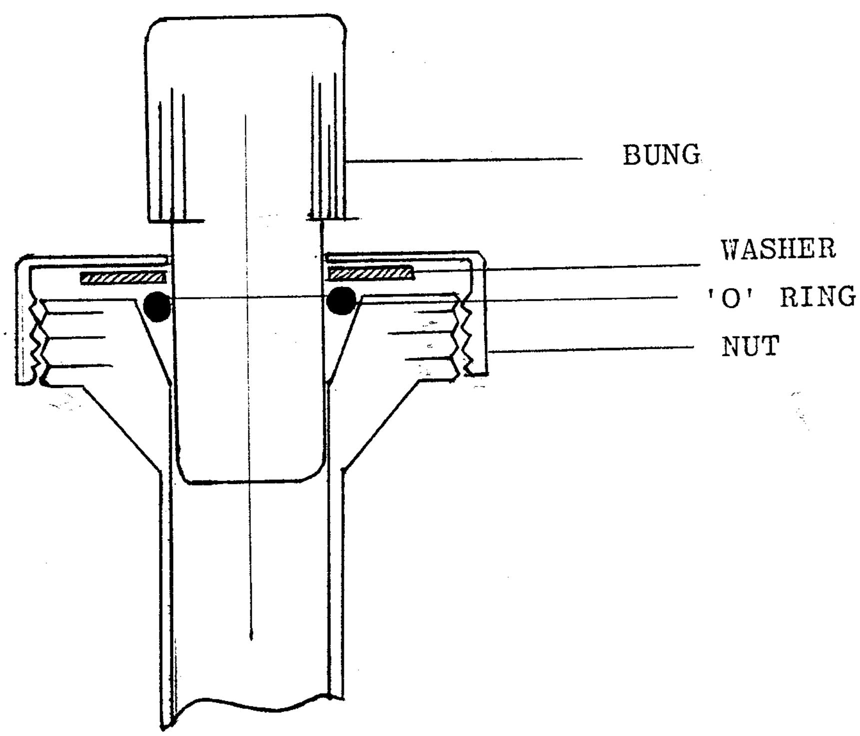
The following procedures must always be followed to ensure efficient and safe operation of the cryogenic system.

a) Assemble the syphon bung correctly, that is the rubber 'O' ring should be below the washer (see diag.)

b) Always ensure that the escaping gas has an exit route so that pressure does not build up inside the cryostat. Periodically check that there is some boil off. Zero boil off could indicate an ice blockage which must be attended to immediately. If in doubt contact your local agent or Oxford Instruments.

c) Periodically check the helium level, excessive boil off could indicate a leak into the vacuum space which may require running down the magnet and warming the cryostat before a cure can be effected.

d) Do not let the level of liquid nitrogen fall below about 10%.




e) Do not attempt to insert the demountable leads with the helium level below 50% and the magnet energised.

f) Do not attempt to refill the cryostat with liquid helium when the magnet is energised unless the helium level is greater than 10%.

## UNPACKING - REMOVAL OF SHIPPING FIXTURES AND REASSEMBLY

(It is strongly recommended that final assembly be done where the magnet is to operate. Movement of the cryostat after assembly can cause deterioration of performance.)

To prevent damage during transit, the cryostat has its internal parts secured together by special fixtures. The existence of these"transit bungs" can be recognised by the bore tube being blocked by aluminium bungs.

Before putting the system into operation, these bungs must be removed, the bore tubes inserted and the superinsulation wrapped around the nitrogen base spinning.

To do this the following equipment will be required:

1. A hoist of minimum capacity 300 kg
2. Assorted spanners and tools (metric)
3. Vacuum pumps, with guages sufficient to reduce the pressure to $10^{-5}$ torr at the pump
4. Silicone vacuum grease
5. Connecting lines from pump to cryostat plus fittings
6. Acetone or some similar solvent
7. Metal polish
8. Clean cotton gloves and soft cloths for cleaning bore tubes.

Proceed as follows:-

Position the cryostat beneath the hoist. Remove the twelve screws (A Figure 1) around the base flange and raise the cryostat leaving the base flange on the floor. Set the base flange aside and remove the hardboard covering from the bottom.

Observe the cryostat from below. The base-spinning of the nitrogen can is now exposed revealing three bosses, whose function is to prevent movement of the base-spinning with respect to the O.V.C base-flange. Remove the bosses (D Figure 2). The nitrogen base-spinning can now be removed by undoing the screws (E Figure 1) around the edge. Remove the three studs from the helium can (G Figure 2).

The central bungs should next be removed. These are contained on a connecting rod held at the top by a retaining plate. Release the retaining plate by undoing the central screw; the bungs should fall down the bore. Lower them gently and remove them completely.

## Re-assembly of the Cryostat

Before fitting of the bore tubes, the internal spacings must be checked and adjusted if necessary. Spacer rods are fitted (L and J Figure1) for adjustment. First adjust the spacing of the helium can and intermediate shield. To do this, view from beneath and adjust the three spacer rods J outwards to obtain an even annulus between the intermediate shield and helium can. Fit the intermediate shield base-plate (K Figuer 1). The intermediate shield base-plate and helium can should be seen to be concentric when viewed down the bore from the top. Re-adjust spacers J if necessary.

When the correct spacing has been achieved, slacken each of the three spacers by about 10 thousandths of an inch and lock the locking screws. Finally, check again for an even annulus and concentricity.

The centre tube can now be fitted. Remove the intermediate shield base-plate. Clean the intermediate shield bore tube with a de-greasing agent. Avoid greasy finger marks by wearing clean cotton gloves. Insert from below so that it engages at its upper end into the top of the intermediate shield (N Figure 1). The bore tube has a shoulder machined at either end for this purpose. Hold in position while the intermediate shield base-plate is re-fitted.

Next adjust the spacing of the helium can/intermediate shield with respect to the nitrogen can by adjustment of the three rods (L Figure 1). The intermediate bore tube should be in place for this procedure. In a similar manner to the previous adjustment, move the spacers outwards to obtain an even annulus between the nitrogen can and intermediate shield. Replace the nitrogen can base-spinning and view down the bore from above. You should see the intermediate shield and nitrogen can base-spinning forming a concentric set. Adjustment of spacers L moves the narrowest hole (in the nitrogen base-spinning) with respect to the other.

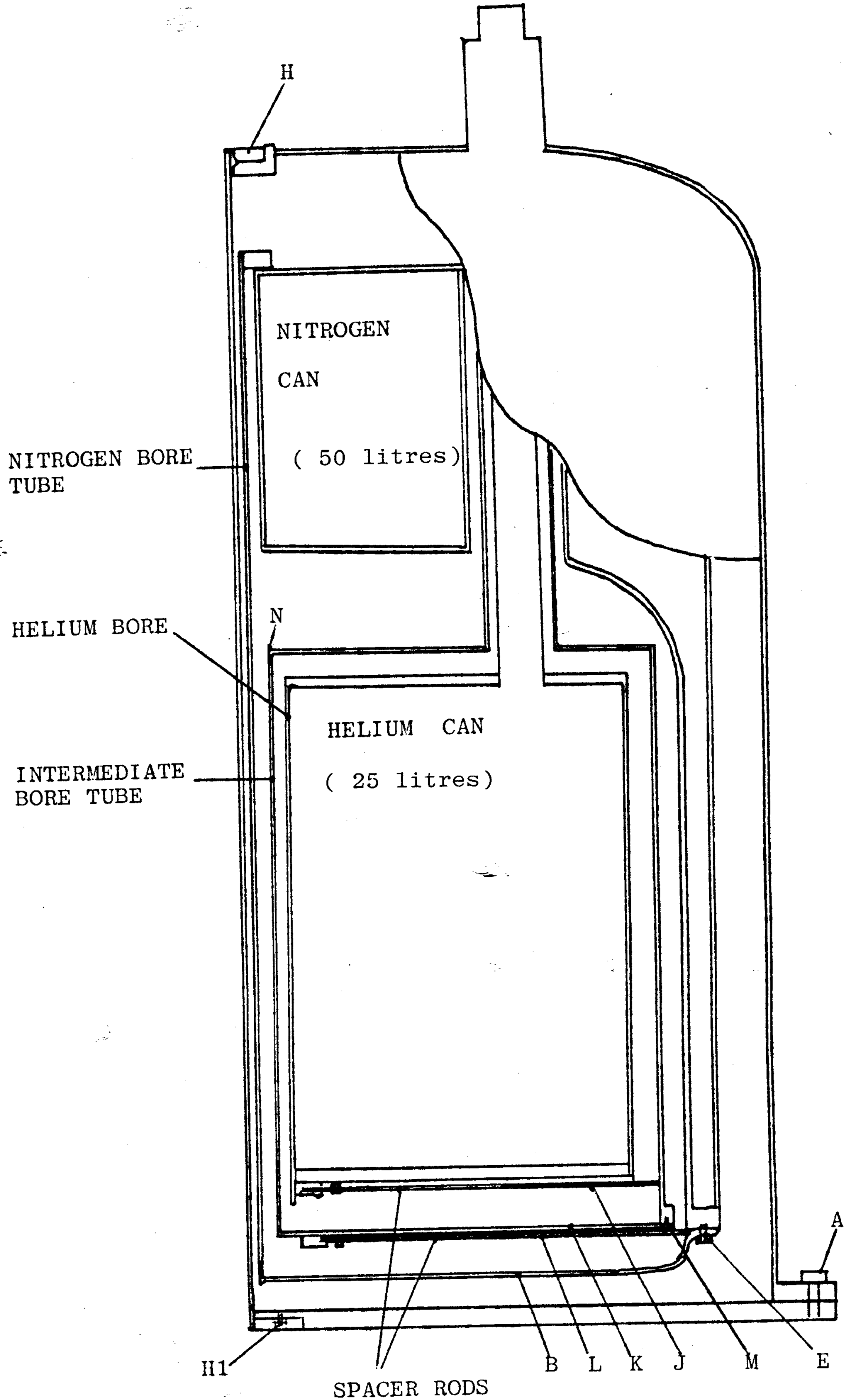
The nitrogen shield bore tube should next be fitted. Clean it with a de-greasing agent as previously. Remove the base-spinning and insert the bore tube from velow so that it engages in the top of the nitrogen jacket. Hold in position while the base spinning is replaced.

Next fit the superinsulation over the nitrogen shield base-spinning. The superinsulation will be tucked up round the outside of the nitrogen can. It may be held in place with aluminium sticky tape. Carefully release the superinsulation so that it hangs down below the bottom of the O.V.C. The superinsulation will already be cut to facilitate holding across the base-plate. Fit the base-pad of superinsulation and inter-leave it every layer with the superinsulation at the sides. Make sure that the insulation will not foul the room temperature bore tube and trim back where necessary so that the outermost layers are shorter than the inner-most. This minimises the inner layers receiving conducted heat from the outer layers if two aluminised surfaces come into contact. Fix the layers in place with small pieces of aluminium tape and remove the tape each time to fix the next layer so that when all layers have been folded over the tape is on the outside only.

Clean the '0' rings on the base plate and lightly grease them. Ensure the channels are clean and free of debris. Remove flange H1 Figure 1 and place the base plate below the cryostat. Lower the cryostat onto the base plate with the large '0' ring in place and screw it down firmly with the twelve screws A. Raise the cryostat again and insert the room temperature bore tube from below, so that it engages in the top flange (H Figure 1). Replace H1 securing the room temperature bore in place. Ensure that the '0' rings are clean and lightly greased.

Finally lift the cryostat onto its legs and screw to the base plate. Fit the vacuum valve,ensuring that all '0' rings are clean and lightly greased.

The cryostat is now ready for evacuation and cool down.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NITROGEN BORE
TUBE
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( 25 litres)
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BORE TUBE
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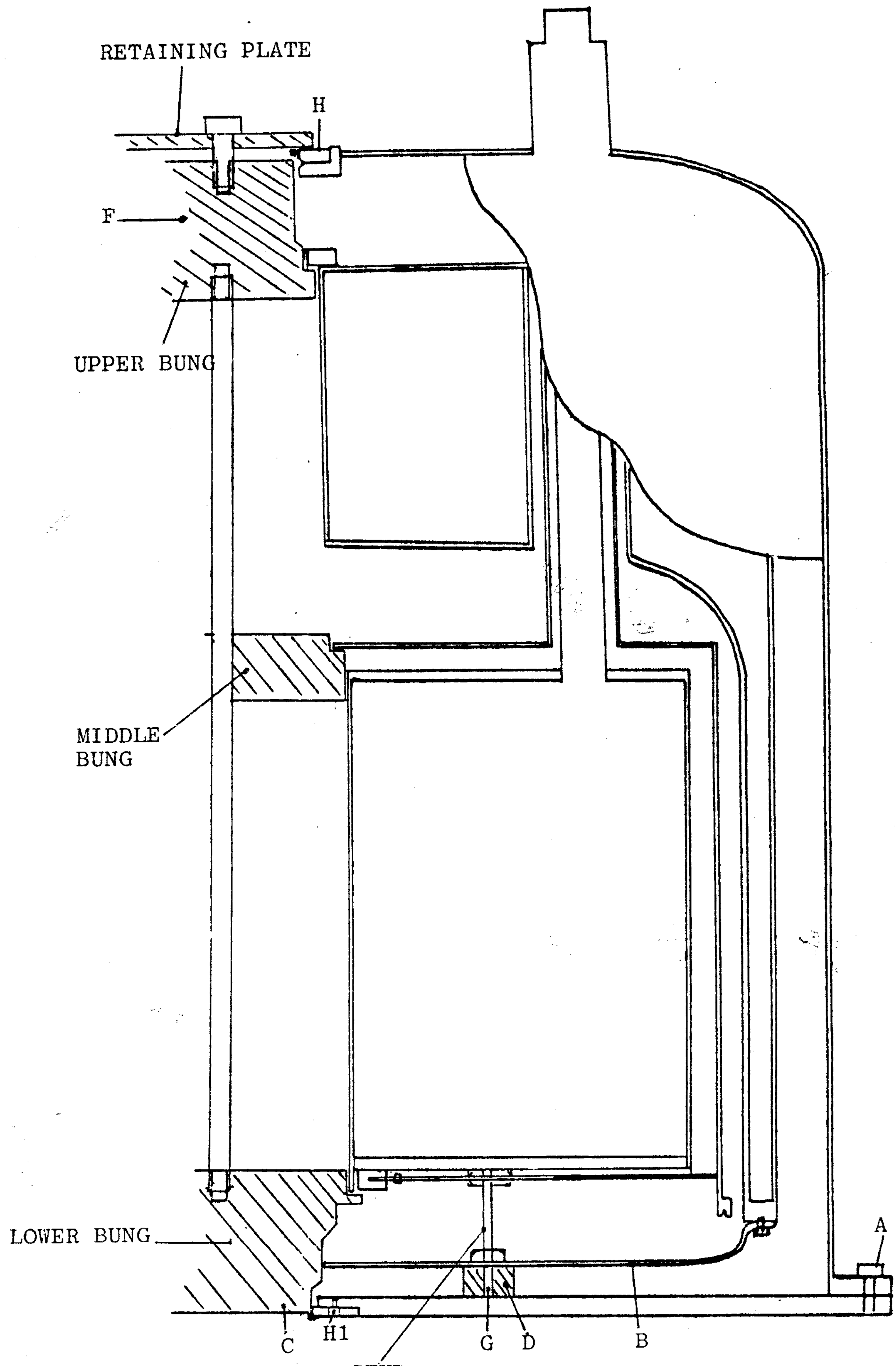
SPACER RODS


RETAINING PLATE
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MIDDLE BUNG
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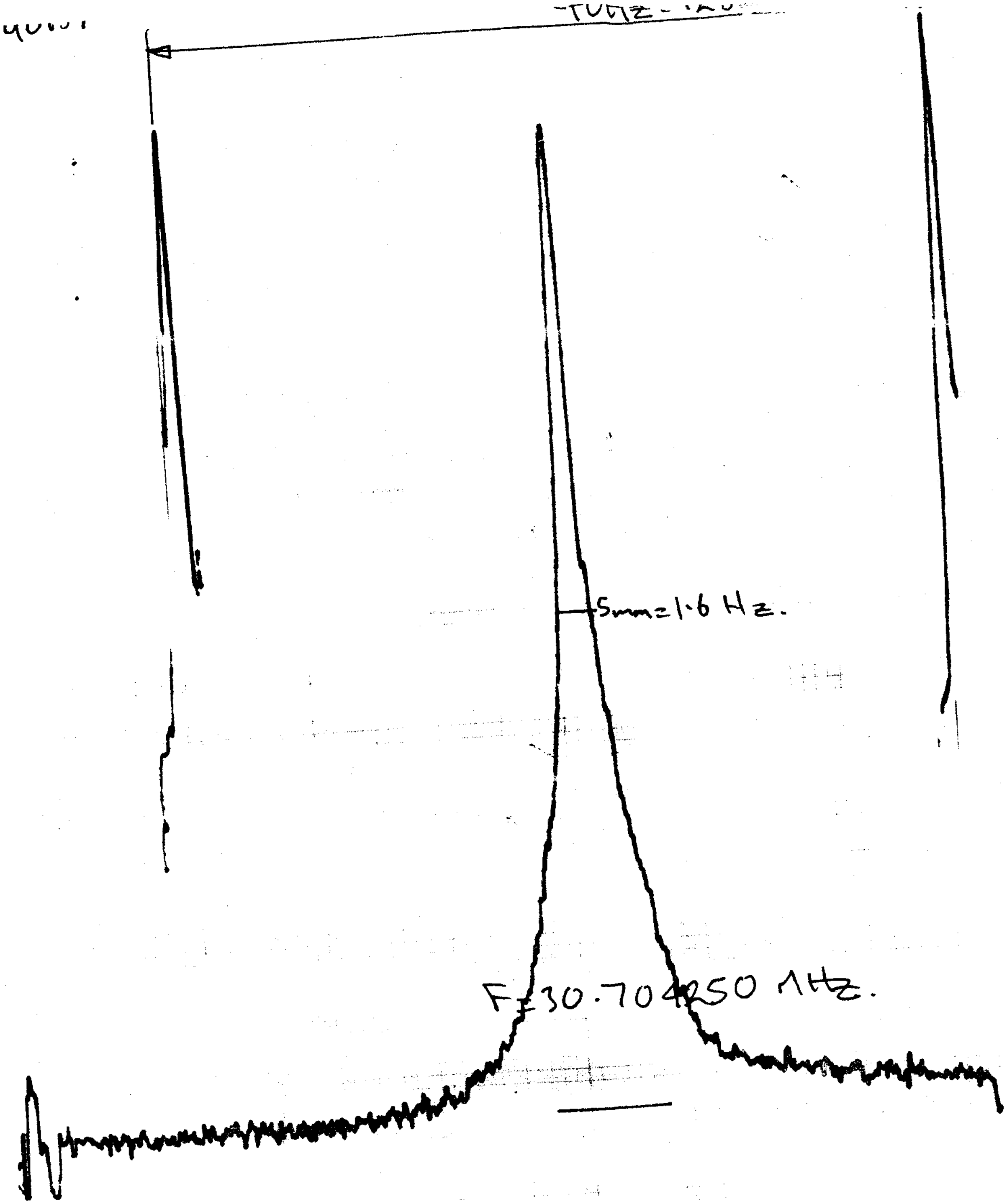



1.6 Hz (shimmed acetone d6 10x10mm sample)

| | S/C | 7/30/82 |
|---|---|---|
| $Z_1$ | 399 | 431 |
| $Z_2$ | 583 | 591 |
| X | 723 | 705 |
| Y | 587 | 600 |
| ZX | 394 | 442 |
| ZY | 493 | 627 |
| XY | 533 | 586 |
| $X^2-Y^2$ | 297 | 376 |
| $Z_0$ | | 485 |

SUPERCONDUCTIVE MAGNET SYSTEM

Project number: W23462
Magnet number: 90157
Cryostat number: D/1500B/4/17

CENTRAL FIELD: 4.697 Tesla (200Mhz)
CURRENT FOR CENTRAL FIELD: 69.78 34.89 Amps
FIELD/CURRENT RATIO: 0.1346 T/A
SUPERCONDUCTING TYPE: Nb--Ti
BORE DIAMETER: 54 mm
INDUCTANCE: 50 Henry
SWITCH "OPEN" RESISTANCE: 10 Ohms.
SWITCH HEATER RESISTANCE: 100 Ohms.
SWITCH HEATER CURRENT: 60 mA
RECOMMENDED POWER SUPPLY TRIP VOLTAGE: 4 Volts.

SWEEP PROGRAMME

(70.13 mT WITH 1.005)

| A/minute | Sweep time setting | from | to (Amps) |
|---|---|---|---|
| MAXIMUM CHARGING RATE - CONSTANT CURRENT | | | |
| 1.5 | | 0 | 15 20 |
| 0.75 | | 15 | 25 33.3 |
| 0.375 | | 25 | 30 40. |
| 0.150 | | 30 | 33 44.0 |
| 0.075 | | 33 | (47) FIELD 46.75 |
| MAXIMUM DISCHARGING RATE - CONSTANT CURRENT | | | |
| REVERSE | ABOVE | | |

| Charging voltage: at power supply | at magnet | from | to (Amps) |
|---|---|---|---|
| MAXIMUM CHARGING RATE - CONSTANT VOLTAGE | | | |
| | | | |
| MAXIMUM DISCHARGING RATE - CONSTANT VOLTAGE | | | |
| | | | |

PROJECT NO: W23462
MAGNET NO : 90157

SUPERCONDUCTING SHIM SYSTEM

Jan/19/1994
Dial Value (×7)

| Dial Value (×7) | Coil | Switch heater resistance | Switch heater current | Recommended Operating current |
|---|---|---|---|---|
| 408 (~~470~~) | Z1 | 100 ohms | 60mA | -2.02 Amps |
| 584 | Z2 | " | " | +1.66 Amps |
| | Z3 | NOT FITTED | | ———— |
| 798 | X | 100ohms | 60mA | +4.46Amps |
| 524 | Y | " | " | +1.74 Amps |
| 465 | ZX | " | " | -2.12 Amps |
| 527 | ZY | " | " | -0.14 Amps |
| 556 | XY | " | " | +0.66 Amps |
| 425 | $X^2-Y^2$ | " | " | -4.06 Amps |
| 470 | $Z_0$ | " | " | |

CRYOSTAT

Liquid helium evaporation rate with leads removed : < 16 c.c./hr

Liquid nitrogen evaporation rate : < 150 c.c./hr

Position of Magnetic Centre : ~ 325 mm. from base flange

NOTES

1. Insert shorting plug immediately on removal of the de-mountable leads.

2. Ensure that the 10 pin seals 'A' and 'B' are connected to the correct cables.

3. Short the current terminals on the top of the de-mountable

1.2 Cryogenic Short Form Instructions

1.2.1 Commissioning Cryostat (at room temperature)

It is assumed that the cryostat has been unpacked, the bore tubes inserted and the

z −1.3 −0.9 −1.2 −1.6 −1.5

$z^2$ 0 +0.8 +1.6

x +4.8 +4.7

y +2.~~0~~ +1.6

xz −1.2 −1.3

yz

xy

$x^2-y^2$ −3.0 −2.8

30 − 10

## 1.2 Cryogenic Short Form Instructions

### 1.2.1 Commissioning Cryostat (at room temperature)

It is assumed that the cryostat has been unpacked, the bore tubes inserted and the helium gas recovery system is ready for connection.

1) Evacuate the outer vacuum case (OVC) to better than 5 x $10^{-5}$ torr. Use a fast rotary pump and diffusion pump after flushing twice with DRY nitrogen gas ($<$1 torr).

2) Ensure that OVC valve is closed and cover the pumping orifice.

3) Insert demountable current leads and helium level probe.

4) Flush and pump the liquid helium tank several times with dry helium gas. The tank should quickly reach a pressure of 1 torr using a 50 l/min. rotary pump. (N.B. OVC must be at vacuum)

### 1.2.2 Cooling to 4.2K

1) Fill the nitrogen tank and helium tank up the neck tubes until full with liquid nitrogen ($LN_2$)

2) After 1 hour completely refill the helium reservoir and leave overnight to precool. In the morning use dry helium gas to blow the liquid nitrogen out. ($LN_2$ out via tube inserted through syphon hole and into cone).

3) Allow helium gas to flow for a few minutes after $LN_2$ stops flowing.

4) Connect a capsule gauge, rotary pump and source of dry helium gas to helium tank.

5) Pump out and watch for a pause in the pressure in the range 80 to 100 torr, (indicates liquid nitrogen).

6) Flush and pump out (at least twice) until certain that no $LN_2$ remains (less than 5 torr). Then fill with helium gas and connect to recovery system.

7) Insert helium transfer syphon ensuring that it is firmly located in the cone (inside the cryostat).

8) Gently transfer until liquid collects ($\sim$1 hour and 25 litres of liquid).

9) a) If the magnet is to be energized and shimming performed transfer until at least 50% full.

1.2.2 (continued)

9) b) Remove demountable leads if energizing later, and insert the gas plug.

10) Check that all holes into the helium tank, except the recovery system, are blocked.

11) The final boil-off figure for the liquid helium will not be known until about a week after initial filling. This is a result of the long thermal time constant of the gas cooled shield.

### 1.2.3 Refilling with liquid helium

1) Unless a special refilling syphon has been supplied, mount the helium transport dewar on a lifting truck and insert transfer syphon (NOT into cryostat).

2) Pressurize transport dewar and wait until liquid comes out of the syphon.

3) Insert syphon into cryostat but stop when its end is about 50 mm above the cone.

4) Transfer liquid

5) Reduce pressure on transfer dewar and remove syphon.

6) Ensure that all holes are closed..

### 1.2.4 Warming up the Cryostat

1) Insert transfer syphon into cone, close off recovery system and transfer as much liquid as possible to a transport dewar. (He tank pressure $<$ 0.5 Atm.)

2) Insert a tube to the bottom of the nitrogen tank and blow out the $LN_2$ using helium gas applied to the other boil off tubes ($\lesssim$ 0.5 atmospheres).

   DO NOT overpressurize.

3) Circulation of dry warm helium gas round the magnet will accelerate the warm up.

4) For fast warm up soften the OVC with dry nitrogen gas and warm the outside using a 1 kW fan-heater.

5) IMPORTANT

   If the cryostat temperature rises above 77K then the OVC MUST be repumped to better than $5 \times 10^{-5}$ torr.

## 1.3 Magnet Short Form Instructions

(If in doubt refer to main instruction section)

### 1.3.1 Running the Magnet up from zero

1) Check that the helium and nitrogen levels are adequate.

2) Insert demountable current lead.

3) Check for continuity of current lead etc.

4) Connect power supplies and set all currents to zero.

5) Decide upon final magnet current and calculate a suitable over-field value (+ 0.5% to 0.7%).

6) Chose constant voltage or constant current mode of energizing.

7) Find the helipot setting to give the overfield current and the final current (The current flows through the superconducting switch when the heater is off)

8) Follow instructions for mode of energizing.

1.3.1 (continued)

9) Check that main current is initially zero.

10) Activate heaters on Main Coil, $Z_0$,
This will happen automatically if the Oxford instrument shim power supply is used, when the main coil heater is activated.

11) Begin to increase the current.

12) Every 5 Amps, dump any induced currents in the other shim coils (i.e. open and close their switches).

13) When overfield is reached leave current flowing for about 10 minutes.

14) Reduce field to final value; wait 5 minutes.

15) Close all magnet switches and wait 2 minutes.

16) Reduce main power supply current to zero.

17) Dump any currents in all other shims.

18) Insert NMR probe and adjust shim currents for best line.

19) RECORD MAGNET MAIN CURRENT AND POLARITY plus shim current values.

20) Remove leads and replace the gas plug

(N.B. Short the magnet terminals before disconnecting the main leads or before switching the power supply off).

1.3.2 Running down an Energized Magnet

1) Look up the operating current and polarity
If there is any doubt about these, then follow the emergancy discharge procedure.

2) Insert current leads and connect to power supplies.

(N.B. Short the main power supply terminals before connecting to magnet).

3) Remove the short circuit.

4) Turn Main current up to operating value.

5) Open the superconducting switches in the following order.

$Z_0$, $Z_2$, $Z_1$ and Main Coil

6) Set current to run down in the required mode and at the recommended rate.

7) When discharged switch off all heater c-rrents and remove leads.

## 1.4 Shimming with Superconducting Correction Coils

(DO NOT operate the main coil heater during this procedure)

1) Set all potentiometers to 5.00 (zero current) and operate all shim heaters.

2) If magnet centre is unknown, move the probe about the approximate centre to find a position where the resonance is independant of the $Z^1$ current. (At a later stage of shimming it may be better to go to the centre of the ZX and ZY shims)

3) If room temperature bore shims are present set all their currents to zero.

4) Adjust $Z_1$ shim for best line

5) Adjust all superconducting shims, in turn, to give maximum signal height. Finally adjust to give best line shape (or longest ringing on a medium sweep rate). Note all currents.

6) Remove leads and close the entry port.

## 1.5 Shimming with Room-Temperature-Bore Correction Coils

1) Adjust $Z^1$, X, Y then $Z^2$, $Z^1$ then ZX, ZY then $Z^1$, X, Y again.

2) Adjust XY, $X^2-Y^2$ and $Z^1$, X, Y.

3) Repeat (1) and (2) for best line.

4) Spin sample at ~ 30 Hz and adjust $Z^1$, $Z^2$, $Z^3$, $Z^4$ ($Z^3$ may contain $Z^1$ impurity and $Z^4$ may contain $Z^2$ and $Z^1$ impurities)

5) Stop sample spinning and adjust ZX, ZY then X, Y, ZX, ZY, XX and $X^2-Y^2$.

6) Repeat (4) and (5) for best results.

7) Check for spinning side-bands. Minimum side-bands will occur for the best line shape. Typically ~ 0.5% amplitude (maximum permitted 2%).

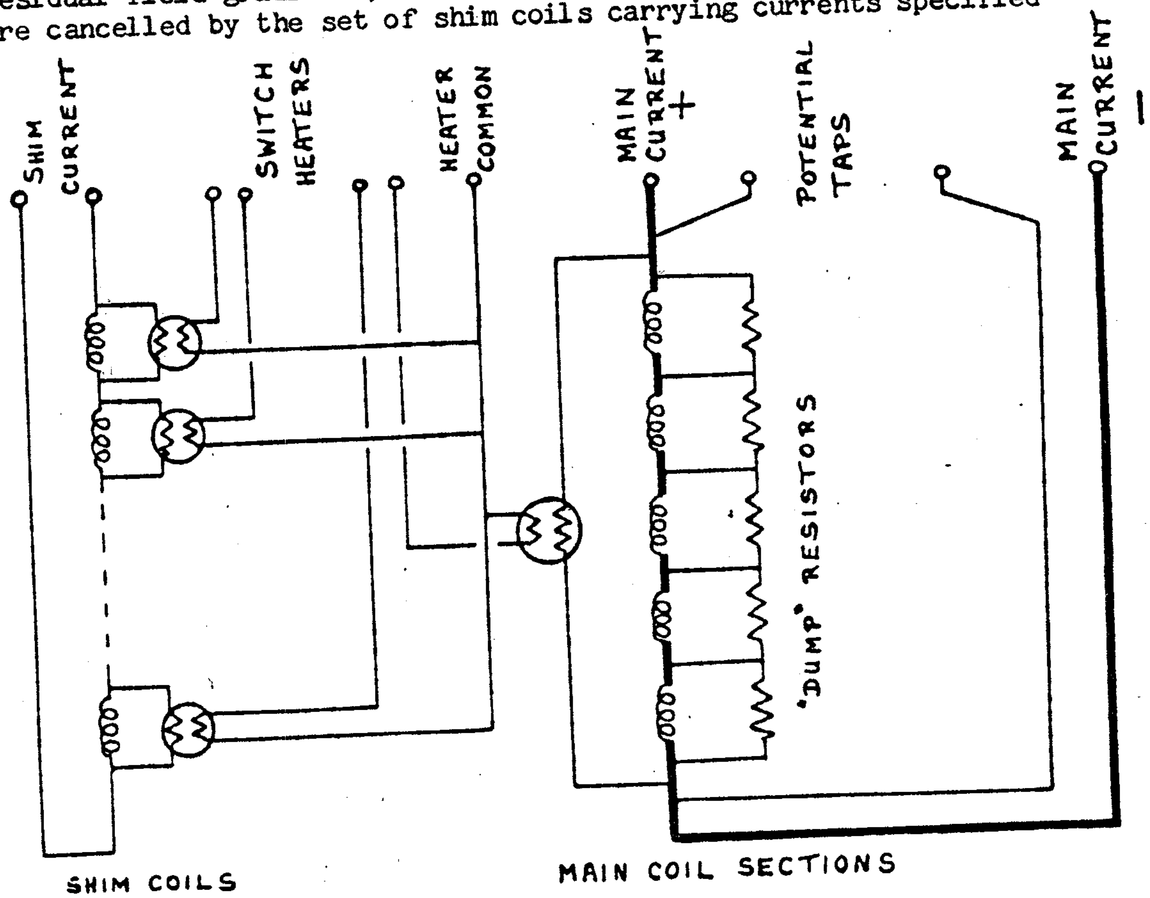
SECTION 2.1- GENERAL PRINCIPLES OF OPERATION

2.1.1 The Magnet.

The magnet is wound from superconducting wire which is NbTi alloy or intermetallic $Nb_3Sn$ filaments in a protective matrix of copper and/or bronze. It consists of a number of concentric solenoid sections together with compensating coils. The latter cancel the residual second and fourth order axial gradients which result from the finite length of the solenoid sections. All the sections are connected in series and a superconducting switch is put in parallel with the whole assembly.

(A superconducting switch consists of a length of superconducting wire thermally isolated from the liquid helium bath. By raising its temperature, using an inbuilt electrical heater, it is made resistive. It is then in its "open" state and current from the power supply will flow through the superconducting magnet rather than through the resistive switch element. When the heater is turned off, the switch element becomes superconductive again and the switch is closed. At this point the current is circulating through the magnet and power supply only. As the current from the power supply is reduced the magnet current remains constant and the difference current flows through the switch. Eventually all the magnet current is going through the switch and full persistant mode has been reached.)

Residual field gradients, caused by winding inhomogeneities etc., are cancelled by the set of shim coils carrying currents specified




MAGNET CIRCUIT SCHEMATIC

in part 1.1. The circuit diagram above shows the arrangement of the various coils and switches.

Above the magnet, at the top of the helium can, is mounted a terminal plate carrying the demountable lead connector, a syphon connector and protection circuits. The protection consists of low value resistors across each coil section.

In the event of the magnet quenching (becoming resistive) these resistors prevent the development of high voltages which could cause insulation break down. They also dissipate some energy during the quench and thus reduce the energy dissipated in the magnet windings.

### 2.1.2 The Cryostat

The magnet is operated at a constant temperature of 4.2K, obtained by immersing it in a bath of liquid helium-4 which is boiling at normal atmospheric pressure.

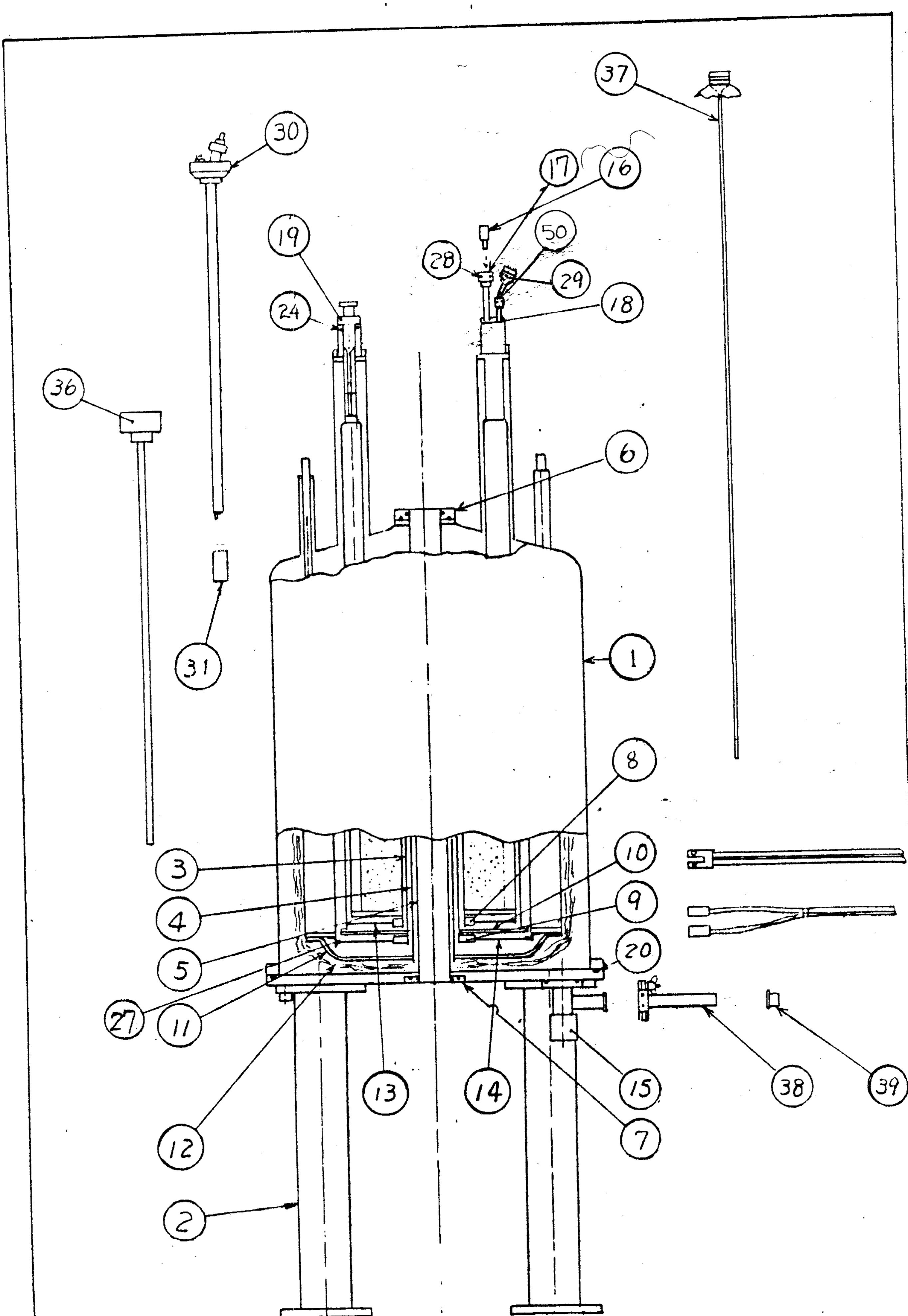
The liquid helium is held in the cryostat which is shown schematically in figure 2.2. The helium vessel consists of two parts (I) a well containing the magnet, and (II) a reservoir. This assembly is suspended by two neck tubes which also provide service access. Latteral support is given by radial struts at the bottom of the helium tank. The helium vessel is surround by a liquid nitrogen tank and shield, which is in turn enclosed by the outer vacuum case. Both the helium tank and nitrogen tank are superinsulated.

### 2.1.3 Cryostat Top Plate

The arrangement of the fittings on top of the cryostat is shown in figure 2.2. The neck tubes, which project above the top of the main cryostat body, carry the inlets for the liquid helium transfer tube, level indicator and dip-stick together with the inlet for the demountable current lead.

All electrical contacts to the magnet are made via this demountable lead which carries a multiway plug on its lower end. The plug mates with a socket on the magnet terminal plate thus allowing repeated insertion and removal. The wiring details are shown in figure 2.3.

The top plate also carries three fill/vent tubes for the liquid valve nitrogen container and the valve for evacuating the outer vacuum case (OVC). This valve incorporates an over-pressure relief valve which opens at approximately 0.1 atmosphere over pressure.



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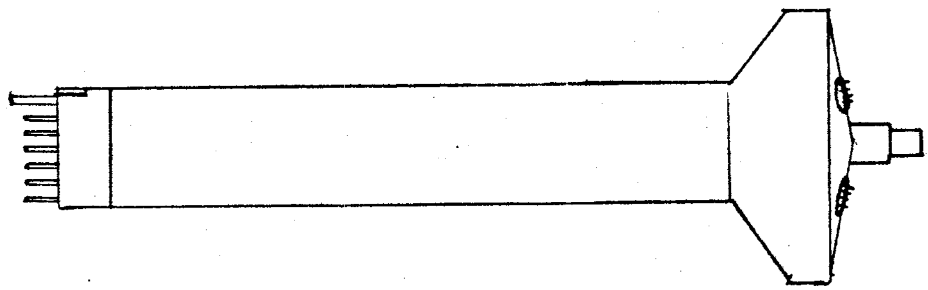
1. Cryostat body
2. Support stand
3. Intermediate shield bore tube
4. Nitrogen shield bore tube
5. Room temperature bore tube
6. Top closure flange
7. Bottom closure flange
8. Fibre glass spacer rod assembly ring ($H_c$can)
9. Fibre glass spacer rod assembly ring ($N_z$can)
10. Intermediate shield base plate
11. Nitrogen shield base plate
12. Superinsulation
13. Fibre-glass spacer rod He/intermediate shield
14. Fibre-glass spacer rod Nz/He
15. Evacuation valve
16. Syphon entry plug
17. Syphon entry port
18. Safety valve (helium can)
19. Radiation baffles, (assembled into stack)
20. Cryostat base plate

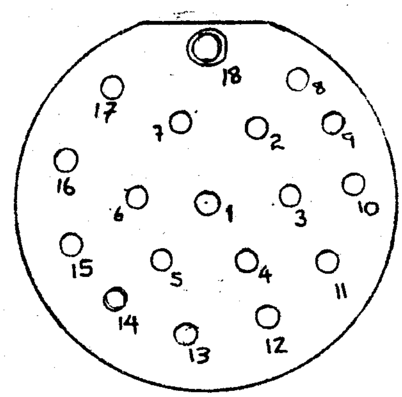
24. 'Top hat' stack assembly
27. Spacer rod bracket
28. 'Syphon entry' 'O' ring
29. Helium level probe
30. De mountable lead
31. Pin protection cap
35. Helium level probe
36. Nitrogen level probe
37. Dip stick
38. Evacuation valve adaptor
39. Dust cap for evacuation port

KEY FIG 2-2

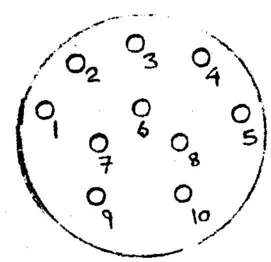
WIRING OF DEMOUNTABLE LEADS



1 8-way lead

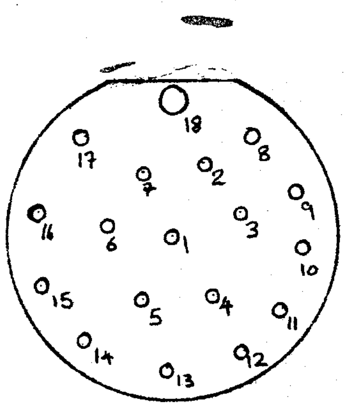



Top view, 18-way connector




Top view of 10 pin seal.

18 WAY CONNECTOR. INSIDE CRYOSTAT TO MAGNET

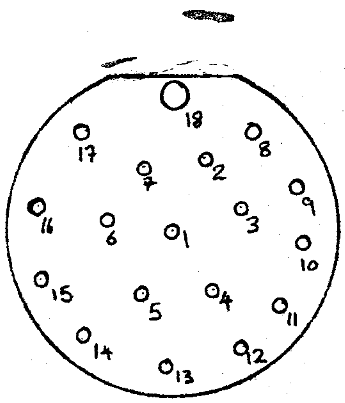



1 8-way connector, Top view

| PIN | FUNCTION |
|---|---|
| 1 | Heater & resistor common |
| 2 | Main |
| 3 | $Z^0$ |
| 4 | Z1 |
| 5 | X |
| 6 | Y |
| 7 | ZX |
| 8 | Main coil start |
| 9 | |
| 10 | Shims (+ ve) |
| 11 | ZY |
| 12 | XY |
| 13 | $X^2 - Y^2$ |
| 14 | $Z_3$ |
| 15 | Shims (- ve) |
| 16 | Main coil end |
| 17 | |
| 18 | $Z_2$ |

Figure 2.4

18 WAY CONNECTOR. INSIDE CRYOSTAT TO MAGNET




1 8-way connector, Top view

| PIN | FUNCTION |
|---|---|
| 1 | Heater & resistor common |
| 2 | Main |
| 3 | $Z^0$ |
| 4 | Z1 |
| 5 | X |
| 6 | Y |
| 7 | ZX |
| 8 | Main coil start |
| 9 | |
| 10 | Shims (+ ve) |
| 11 | ZY |
| 12 | XY |
| 13 | $X^2 - Y^2$ |
| 14 | $Z_3$ |
| 15 | Shims (- ve) |
| 16 | Main coil end |
| 17 | |
| 18 | $Z_2$ |

Figure 2.4

WIRING OF 18 WAY DE-MOUNTABLE LEADS

DECEMBER 1979

| 10 pin seal 'A' | 18 pin No. | Wiring | Function | 10 pin seal 'B' | 18 pin No. | Wiring | Function |
|---|---|---|---|---|---|---|---|
| 1 | 2 | ↑ | Main | 1 | 10 | 24 s.w.g. | Shims |
| 2 | 3 | | $Z^0$ | 2 | 10 | | (+ ve) |
| 3 | 4 | Switch | $Z^1$ | 3 | | | |
| 4 | 18 | Heater | $Z_2$ | 4 | 15 | 24 s.w.g. | Shims |
| 5 | 5 | | X | 5 | 15 | | (- ve) |
| 6 | 6 | 36 s.w.g. | Y | 6 | | | |
| 7 | 7 | | ZX | 7 | 8&9 | 36 s.w.g. | Main start |
| 8 | 11 | | ZY | 8 | 16&17 | 36 s.w.g. | Main end |
| 9 | 12 | | XY | 9 | 14 | 36 s.w.g | $Z_3$ heater |
| 10 | 13 | ↓ | $X^2 - Y^2$ | 10 | 1 | 24 s.w.g. | Common |

## SECTION 2.2 CRYOGENIC FLUIDS AND COOL-DOWN PROCEDURE

### 2.2.1 Liquid helium and liquid nitrogen

The cryogenic fluids used with this system are liquid helium and liquid nitrogen. The relevant properties of these liquids are shown in the table overleaf.

(a) Liquid nitrogen is a colourless liquid obtained from the liquefaction of air. It is generally stored in vacuum-insulated containers, but its relatively high latent heat of evaporation permits short term storage in foamed plastic vessels. It may be trans ferred from one container to another by pouring, or by the use of a rubber or plastic tube. The following safety precautions should be observed.

(1) Flexible materials become brittle when cooled to liquid nitrogen temperature. Rubber tubes used for transferring liquid nitrogen will break easily if strained.

(11) Open vessels containing liquid nitrogen should be kept covered to prevent frost formation by the condensation of water vapour from the atmosphere, and also to prevent oxygen enrichment by condensation from the air. This can present a fire risk.

(111) Vessels containing liquid nitrogen should not be sealed so that evaporating gas can escape and prevent a pressure build-up.

(1V) Liquid nitrogen spilled on vacuum or cryogenic equipment will freeze "O" rings and cause loss of vacuum. Care should be taken when pouring to prevent excessive spillage.

(V) Liquid nitrogen spilled on the body will cause tissue damage, similar to a severe burn. Rubber gloves and boots should be worn where necessary.

(b) Liquid helium is a colourless liquid produced from the naturally occuring gas deposits in the earth. The density of the gas is so low that the concentration in the atmosphere is minute. Where possible, evaporated gas should be recovered and re-liquefied, both to economise in operating costs and to conserve the earth's resources.

| Cryogen | Normal Boiling Point (K) | Latent Heat (Joules/g) | Amount of Liquid Evaporated by 1 Watt (L/1 hour) | Liquid Density (g/ml) | Gas Density at NTP g/ml | Liquid to NTP Gas Volume Ratio | Enthalpy Change (gas) B.P. to 77 K (J/gm) | Enthalpy Change (gas) 77 to 300 (J/gm) |
|---|---|---|---|---|---|---|---|---|
| Liquid Helium | 4.2 | 20.9 | 1. 38 | 0.125 | $1.79 \times 10^{-4}$ | 1 : 700 | 384 | 1157 |
| Liquid Hydrogen | 20.39 | 443 | 0.115 | 0.071 | $8.99 \times 10^{-5}$ | 1 : 790 | 590 | 2900 |
| Liquid Nitrogen | 77.55 | 198 | 0.023 | 0.808 | $1.25 \times 10^{-3}$ | 1 : 650 | - | 234 |
| Liquid Oxygen | 90.19 | 212.5 | 0.015 | 1. 14 | $1.43 \times 10^{-3}$ | 1 : 797 | - | From BP 193 |

Liquid helium is stored in vacuum insulated containers which also include some form of radiation shield to intercept thermal radiation from the room-temperature environment. It is transferred between storage vessels, or from a storage vessel to a cryostat by means of a vacuum insulated transfer tube (sometimes called a syphon).

2.2.2 Evacuating the outer vacuum case.
(It is not recommended that this be done if the cryostat is below room temperature).

In order to maintain the thermal isolation of the liquid helium it is necessary that a high vacuum be obtained in the outer case of the cryostat (OVC). Traces of air will be condensed when the cryostat is filled with liquid helium but helium gas will spoil the vacuum. Over a long period of time, helium gas may percolate past O-rings from the atmosphere. The OVC should not need pumping if the cryostat contains liquid helium.

A slow increase in the boil-off rate of liquid helium indicates that the dewar is going soft (helium gas has entered the OVC).

If the OVC goes soft, de-energise the magnet (section 2.3.5), warm up the cryostat and try to locate the leak. Assuming that the problem was helium gas diffusion, pump out the OVC and follow the usual commissioning procedures.

The pumping equipment needed should consist of an oil diffusion pump of 5o mm (2in.) diameter fitted with a liquid nitrogen cold trap. The diffusion pump should be backed by a rotary pump of speed not less than 25 1/min. The rotary pump should have a gas ballast facility. If plastic or rubber link tubes are used these must NOT have been used previously to carry or pump helium.

(a) Connect the valve on the top of the cryostat to the pumping equipment using a short tube of not less than 15 mm ($\frac{3}{4}$ in) internal diameter. Using the rotary pump, evacuate the cryostat (OVC) until the pressure is less than 1 torr, then admit an atmosphere of DRY nitrogen gas and pump out again - repeat several times. Finally pump to less than 0.05 torr.

(b) Switch over to the diffusion pump and evacuate the cryostat to less than $5\times10^{-5}$ torr and continue pumping for at least 24 hours (preferably at least 48 hours) before sealing the OVC valve. This ensures that residual gas trapped in the superinsulation is removed, and cannot impair the reflecting properties of the superinsulation. (On filling the cryostat with cryogenic liquids, the pressure should fall to less than

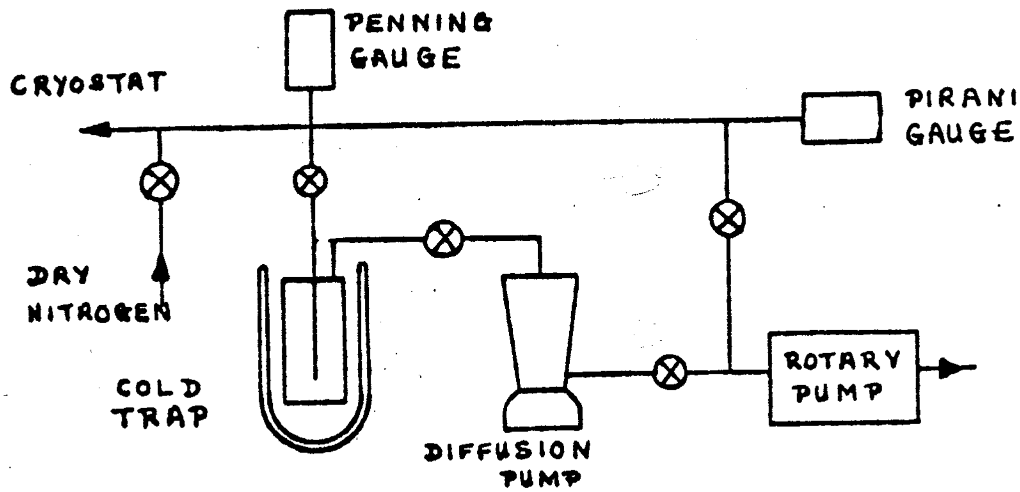



FIG 2.5 CRYOSTAT PUMPING SYSTEM.

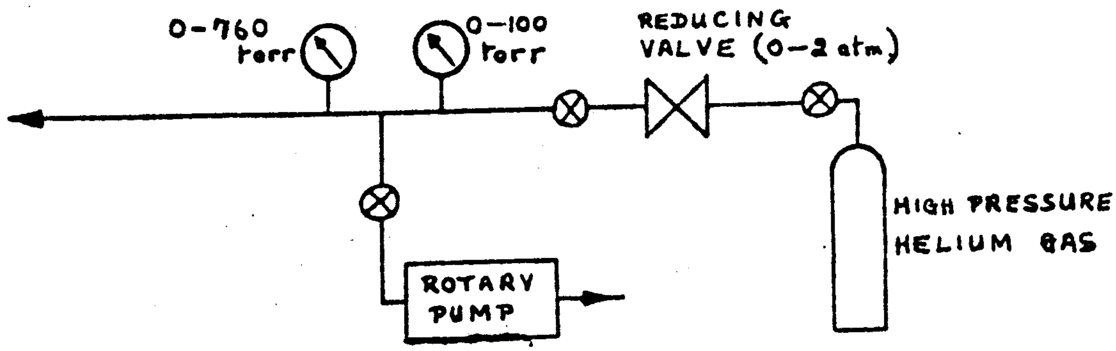



FIG. 2.6 FLUSHING & NITROGEN "BLOW-OUT" SYSTEM

$10^{-6}$ torr). Place a dust cap over the pumping port.

It has been assumed that the evacuation procedure started from atmospheric pressure. If the cryostat is already evacuated and it is desired to inspect the pressure only, the pumping tube should be already evacuated and the diffusion pump operating before the OVC valve is opened.

The recommended equipment for evacuating the outer case is shown in figure 2.5.

WARNING NEVER PUMP HELIUM CAN WITHOUT A VACUUM IN THE O.V.C. (Failure to observe this rule will result in collapse of the helium reservoir).

### 2.2.3 Filling the liquid nitrogen container.

Connect one of the three filler/vent tubes of the liquid nitrogen container to a storage vessel using flexible plastic pipe. Transfer the liquid nitrogen by pressurising the storage vessel to approximately 0.25 atm. (4 P.S.I.G.) Violent boiling will occur initially until the radiation shield has cooled down. When liquid nitrogen sprays out of the filler tubes release the pressure on the storage vessel to stop the transfer.

The storage vessel can be pressurised using a high-pressure gas cylinder fitted with a reducing valve. By using an electrically operated valve between the gas cylinder and the storage vessel, the liquid nitrogen container can be filled and the level maintained using a Liquid Nitrogen Level Controller.

Inspect the liquid nitrogen level daily.

The problems cause by ice formation in the filling tubes can be prevented by slipping 0.25 m (10in.) lengths of plastic tubing over them. These tubes also prevent any overflow of liquid nitrogen from cooling the top flange and its O-ring. This can be important if an autofilling system fails to stop the nitrogen transfer when the tank is full.

### 2.2.4 Precooling the magnet.

Before filling the cryostat with liquid helium, the magnet must be cooled to a temperature below 100K. To do this, fill the liquid helium container with liquid nitrogen, completely above the magnet. Use a length of 10 mm (3/8 in.) diameter stainless steel tubing inserted into the transfer tube entry port. Allow the liquid nitrogen to remain for one or two hours and then fill it completely again. Leave the system overnight to precool correctly and then remove it as follows.

Insert the 10 mm stainless steel tube into the transfer entry fitting and ensure that it is firmly fitted into the socket on the top of the magnet. Blow out all the liquid nitrogen by pressurising the liquid helium container to not more than 0.25 atm. (4 p.s.i.g.)

It is most important that all the liquid nitrogen is removed. Failure to do this properly will make filling with the liquid helium difficult, and may impair the performance of the magnet. Evacuate the liquid helium container using a rotary pump, (if during pump down a pause is seen in the range 70-100 torr then liquid nitrogen is still present) and then fill it with helium gas. Repeat this procedure at least two times in order to thoroughly purge the magnet of nitrogen. As an indication that all the liquid nitrogen has been removed, check that it is possible to evacuate the liquid helium container to a pressure less than 10 torr.

The recommended equipment for performing this operation is shown in figure 2.6.

### 2.2.5 Initial Filling with liquid helium.

Connect the cryostat to the helium recovery system or put a one-way valve on the cryostat exhaust tube. Position the liquid helium storage vessel so that the transfer tube can be inserted easily. Ensure that the transfer tube is not blocked by blowing helium gas through it.

Remove the plug from the transfer tube entry port and also from the top of the storage vessel. Insert the transfer tube slowly, allowing it to cool gradually. Ensure that the end of the transfer tube is fitted into the socket on top of the magnet. In this way, liquid is introduced at the bottom of the magnet which is then cooled by the enthalpy of the gas as well as by the latent heat of evaporation.

Start transferring the liquid helium by pressurising the storage vessel. (This is generally done by gently squeezing a rubber bladder). The transfer rate should be such that the vent pipe is frozen for not more than 2 m (6 ft.) of its length. The initial transfer rate should be equivalent to about 10L. of liquid per hour. This rate can be increased as the magnet cools.

When the magnet resistance drops to zero, the transfer rate can be further increased in order to fill the liquid helium container. This should occur when 15 to 30 litres of liquid have been transferred, and a further 30 litres are then required to fill the cryostat.

When the liquid helium reservoir has been filled, stop the transfer by releasing the pressure in the storage vessel. Remove the transfer tube and replace the plug.

Inspect the liquid helium level at least daily.

### 2.2.6 Refilling with liquid helium.

The cryostat should be refilled before the level reaches the 10% mark. In refilling, care should be taken not to evaporate the liquid in the cryostat with the hot gas which initially comes through the transfer tube. (N.B. - Failure to take care can cause the magnet to quench).

The correct procedure is as follows:-

(a) Insert one leg of the transfer tube into the storage vessel but leave the other one outside of the cryostat. Pressurise the transport dewar in the normal way, as if transferring helium. After about a minute liquid will issue from the transfer tube, indicated by a blue tongue of vapour. (Prior to this a white vapour plume will have been seen for about 20 seconds).

(b) Quickly release the pressure in the transport dewar and insert the open end of the transfer tube into the cryostat.

(c) Lower the transfer tube until it reaches the bottom of the necktube. DO NOT push the tube into the socket on top of the magnet. Transfer liquid helium in the usual way.

If the helium level has fallen below 5% and the magnet is still energized there are two courses of action open:

(1) If the level is below 0% or if the user is not certain that a careful transfer can be done - DE-ENERGIZE THE MAGNET. - refill and then re-energize the magnet.

(11) Refill the dewar but be careful as the syphon is introduced and as the transfer starts.

### 2.2.7 Closing down and warming up the cryostat.

Having de-energized the magnet, the system can simply be allowed to run out of liquid helium and nitrogen, and left to warm up. If a rapid warm up is desired either transfer the helium out of the cryostat into a transport dewar or insert the blowing-out tube into the transfer tube entry port and gently pass DRY helium gas through it. This will boil-off the remaining liquid. Remove the liquid nitrogen by passing a stainless steel tube through one of the filler tubes and blocking off the other two fillers. This will pressurise the container and blow out the liquid.

Having removed all the cryogenic liquids the system can be warmed up by softening the vacuum. Leave for 1 hour to let the magnet warm towards 77K. Slowly allow DRY nitrogen gas into the OVC until 1 atmosphere is reached - leave the valve open with 1m of 20 mm diameter tube connected. The nitrogen gas can be obtained from the neck of a container of quickly boiling liquid nitrogen. Non-preferred method: With the vacuum valve closed, blow some helium gas into the pipe attached to the valve. Place a rubber bung on the end of the pipe, then open the valve and close it again. This technique ensures that only a small amount of helium gas enters the vacuum space, so that the warming up process is not too violent. Ensure that the relief valve is unobstructed. - This technique is very effective, but afterwards great care must be taken to flush the helium out of the superinsulation.

### 2.2.8 Helium gas recovery.

The use of a gas recovery system is desirable for four reasons,

1) Financial saving;
11) Conservation of the earth's supply of helium;
111) Prevention of the cryostat becoming contaminated with ice and air;
1V) Prevention of the air becoming contaminated with helium gas to the detriment of vacuum seals.

A typical recovery system consists of low-pressure gas storage, in the form of a gas-holder or gas-bag, connected to the cryostat vent; a compressor; and high-pressure gas storage. The compressor should be specifically made for helium because of the large amount of heat produced when compressing this gas.

IMPORTANT NOTE.

To prevent air entering the cryostat the recovery system should be maintained slightly above atmospheric pressure. A pressure of a few centimetres of water is sufficient.

Failure to observe this precaution may lead to ice and solid air forming on the connectors for the demountable leads, preventing reinsertion of the leads.

SECTION 2.3 MAGNET OPERATION

## 2.3.1 Insertion of leads.

Remove the plug in the current lead entry port by slackening the knurled nut. *Insert the current leads and lower them gently, allowing adequate time for them to cool down, until the lower end is felt to enter the conical socket. Rotate the lead until the location pin on the connector is felt to engage, and then gently push the connectors together. Care should be taken to avoid bending the pins. Tighten the knurled nut to ensure a gas tight seal. Check that the resistance between the two main leads is less than 0.1 ohm, and test for continuity and short circuits. Connect the flexible leads to the current supply, observing the polarity and ensuring that all connections are tightly clamped.

Note the polarity in the system log-book.

Consult figures 2.3 and 2.4 for the arrangement of the leads.

## 2.3.2 Energising the Magnet.

### CONSTANT VOLTAGE MODE.

When using an Oxford Instruments, or similar power supply, to run the magnet up from zero field, the supply can be used in its constant voltage mode. This provides a smooth charging rate which decreases at higher currents as the voltage drop down the leads subtracts from the set voltage level on the supply. Operating current and charging voltages are given in Part 1.1. Small changes in current are best made with the fine current control on the power supply. Set all the supply controls to zero.

(a) With the main superconducting switch closed (heater "OFF") set the positive voltage limit control of the supply to maximum Set the "FINE" current control to mid-range. Increase the current to the desired value (over-field value - see next section) using the "COARSE" current control.

(b) Wait for one minute then measure the voltage at the supply terminals. Add to this the recommended magnet charging voltage (part 1.1) and note this total voltage ($V_T$)

(c) Now turn the voltage limit control to zero leaving the current control set at the desired level. The output current will now fall to zero.

* If connectors are "iced-up" so that the leads can not be inserted, see section 2.4.2

(d) Open the superconducting switch by switching on the heater supply. Also open the superconducting switches of the $Z_o$ and $Z_2$ shim coils.

(e) Wait for 30 seconds and then set the voltage control to $V_T$. The magnet current will now increase at a rate determined by

$$\frac{LdI}{dt} = V_T - (R_L I)/\ (1+R_L/R_S)$$

where I is the magnet current (not equal to the supply current $I_o$)

$R_L$ is the lead resistance.

L is the magnet inductance.

$R_S$ is the combined resistance of the switch and the protection resistors 2 to 6 ohms.

After the supply reverts to the constant current mode, the magnet current will come to equilibrium with a time constant $L/R_s$ seconds. Thus a few minutes wait is necessary before any other action is taken.

(f) Reduce the current slowly by hand down to the operating value. Wait several minutes and then close the superconducting switch (plus $Z_o$, $Z_2$ etc.)

(g) Wait a few more minutes then smoothly reduce the power supply current to zero. Short the terminals on top of the demountable current lead and disconnect the flexible leads from the power supply.

CONSTANT CURRENT MODE.

In this mode the power supply current is change by external means - either manually or using a sweep generator. The magnet will only charge at the programmed rate if the "set voltage" level (at the output terminals of the supply) is large enough to overcome the back e.m.f. of the magnet. Set all the controls to zero. (It is assumed that an external sweep unit is used).

(a) With the main superconducting switch closed set the sweep unit output to maximum. Set the positive voltage limit to maximum, then increase the power supply control until the full current (overfield value) is flowing in the leads. Lock the control in this position.

(b) Reduce the sweep unit output to zero and check that the main current falls to zero. Wait one minute.

(c) Open the superconducting switches on the main magnet and the $Z_o$ and $Z_2$ shims. Wait for 30 seconds.

(d) Set the sweep rate to the value shown in part 1.1 and begin the sweep. Change the sweep rate at the specified current values.

(e) Stop the sweep at the full overfield current and wait for a few minutes. Reduce the current to the operating value (either by hand or on the slow sweep rate) and wait for several minutes. Then close the superconducting switch (plus $Z_o$ and $Z_2$).

N.B. NOTE THE POLARITY AND FINAL CURRENT IN THE LOG BOOK.

(f) Wait for a few more minutes and then smoothly reduce the power supply current to zero. Short the terminals on the demountable current lead and disconnect the flexible leads from the power supply.

After an hour the magnet can be shimmed to within 10 times the specified homogeneity. There may be significant field and homogeneity changes over the next two days (or longer if the overfield value was not correct - see section 2.3.3) so that the final shimming should be delayed until the magnet has settled to its final field value. If bore shims are used on the magnet then they can accommodate any changes unless the overfield value was grossly incorrect.

### 2.3.3. Persistent mode operation

Although a superconducting magnet in persistent mode produces great long-term field stability, the superconducting screening currents associated with superconductors take a considerable time to settle to their final distribution and values. During this time, of about one week duration, the field will drift by about a gauss. This effect can be reduced so that the drift is almost eliminated by the following technique.

If the final value of the field is being approached from below (i.e. charging the magnet), increase the current <u>above</u> its final value by between 0.5 and 0.7% (the optimum value will require some experimentation). Then reduce the current to its final value. These operations can be done manually using the fine current control, taking care to operate the control smoothly. Before proceeding further, wait several minutes to ensure that current sharing between the magnet and the protection resistors has ceased, then close the superconducting switch by turning the heater "OFF". Wait for another minute, then decrease the current supply output smoothly to zero using the sweep generator on the ten minute range. When the current reaches zero turn the voltage control to zero.

Short the main terminals, together to avoid voltage transients (which could possibly cause the switch to open) and disconnect the power supply.

NOTE THE POLARITY IN THE LOG BOOK.

on. Short the output terminals.

Insert the demountable current lead into the cryostat and connect to the electronics. TAKE CARE that all switch heaters are OFF

.

Remove the short circuit from the output terminals.

EXTREME CARE must be taken to ensure that the main current leads are connected with the correct polarity. If any doubt exists as to the correct polarity, it is preferable to operate the emergency discharge mechanism, rather than attempt to discharge the magnet in the conventional manner (section 2.3.6.)

Set a suitable voltage limit on the current supply and sweep up the current to exactly the value of the operating current, using the ten-minute sweep range. Discharge the $Z^o$ and $Z^2$ shim coils (which are inductively coupled to the main coil) as follows:-

(a) increase the shim supply current to the value noted in the log book.

(b) open the superconducting switch, wait for 30 seconds then reduce the current to zero. Close the switch and repeat the above for all even order axial shims.

(c) Open all the switches for $Z_o$ and $Z_2$ etc.

Open the main superconducting switch.

Discharge the main solenoid by running down its current at the rate specified in part 1.1. This can be done in constant voltage or constant current mode. Wait until the power supply terminal voltage falls to zero before disconnecting the leads.

Discharge all the shims by opening their switches.

Disconnect all power supplies.

## 2.3.4. Shimming the magnet.

### General Comments.

To obtain the best field homogeneity it is necessary to observe an NMR signal from a nuclear species which has a line width significantly less than the expected magnet line width. It will be assumed that the line is observed in the CW mode using a moderately fast sweep and an unsaturated signal.

With the exception of the Zo shim, all the shims have low stored energy so that no special precautions need to be taken in their operation. The Zo shim is strongly coupled to the main coil, consequently changes in its current should be made carefully.

Initially shimming will be done for maximum signal height. Only in the final stages will the "ringing" after the peak be maximized.

(a) Turn all shim switch heaters "ON" for about 30 seconds then switch them "OFF".
(From here it is assumed that the switch heaters are operated when required and no explicit instruction will be given).

(b) Adjust the NMR probe height until the centre of the $Z_1$ shim is located. (i.e. the line position does not move with $Z_1$)

It is recommended that the shims be adjusted in the following order:-

(c) $Z_1$, X, Y - go round twice or more.

(d) ZX, ZY then return to $Z_1$, X, Y - repeat until best signal is found.

(e) XY, $X^2 - Y^2$

(f) repeat (d) and (e) as often as required.

(g) Sometimes the centre of the ZX and ZY shims does not coincide with the $Z_1$ centre. In the final stages of shimming it helps to move the probe to the centre of the ZX and ZY shims. This movement will not exceed $\pm$ 2mm.

NOTE: It should be remembered that, at these levels of field homogeneity, the susceptibility of the construction materials of the NMR probe can cause problems. This is especially true if the symmetry of the probe construction is low, since then the inhomogeneities cannot be removed using only first and second order shims.

## 2.3.5. Discharging the magnet.

Turn all the controls of the main power supply to zero and switch

2.3.6. Emergency Discharge

In an emergency, for instance when no power supply is available or when the polarity of the current leads in unknown, the magnet may be discharged by connecting a pair of diodes across the terminals as shown in figure 8. Switch on the $Z^0$ and $Z^2$ shim coil switch heaters and then the main coil heater. The magnet will discharge at a rate determined by the forward voltage drop of the diode. The discharge will be slow, e.g. about 100 minutes using silicon diodes. Care should therefore be taken not to disconnect the diodes before the discharge is complete. The diodes must be capable of carrying the full operating current of the magnet and must be fixed to an adequate heat-sink

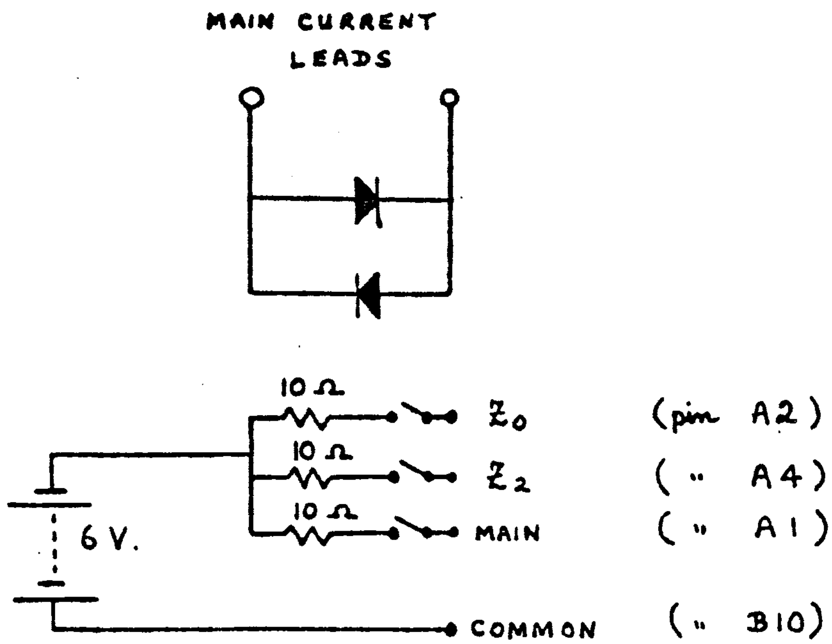



FIG 2.8

SECTION 2.4 TROUBLE SHOOTING

2.4.1. Cryogenic Operation.

| | | |
|---|---|---|
| High impedance pumping line | - | shorten line or use large bore tubing. |
| Defective pump | - | rectify or replace. |
| Condensable vapours in vacuum space | - | pump on gas ballast until clear. Warming the outer case may help |
| Leak into vacuum space | - | prove and locate leak by connecting helium mass spectrometer leak detector to the vacuum valve. Leak located in nitrogen or helium vessels by evacuating the vessel in question, when leak rate should diminish and then filling with helium gas when leak rate will increase. |
| Leak in outer case | - | Inspect "O" rings and replace as necessary or consult Oxford Instruments Company. |
| Leak in nitrogen or helium vessels | - | Consult the Oxford Instrument Co. |

Vacuum case pressure does not decrease on filling with liquid nitrogen.

| | | |
|---|---|---|
| Leak into vacuum space | | see above |

Vacuum case pressure does not decrease on filling with liquid helium.

| | | |
|---|---|---|
| Diffusion pump back streaming | - | disconnect pump |
| Leak into vacuum space | - | see above |

Magnet does not pre-cool.

| | | |
|---|---|---|
| Nitrogen vessel empty | - | fill |

Difficulty in transferring liquid helium.

| | | |
|---|---|---|
| Magnet not adequately pre-cooled | | see section 2.2.4. |
| Transfer tube blocked | - | remove, allow to warm up and blow helium gas through it. |
| Storage vessel empty | - | replace. |
| Transfer rate too high | - | recovery pipes excessively frosted. Frost for 1 m indicates adequate transfer rate. |

| | | |
|---|---|---|
| Excessive heat leak | - | See "high evaporation rate" below. |
| Thermal oscillations in helium vessel neck | - | Place ear to top of cryostat-oscillations can sometimes be heard. Move transfer tube up or down until oscillations dissappear. |
| Syphon does not reach to bottom of magnet | - | When cooling the magnet down to 4.2K it is essential to transfer liquid to below the magnet so that the enthalpy of the gas is used for cooling. Ensure that the transfer tube is inserted into the extension socket on top of the magnet. |

Excessive liquid helium evaporation rate

| | | |
|---|---|---|
| Leak into vacuum space | - | Indicated by condensation of water vapour or frost on the cryostat's outer wall. |
| Helium vessel touching radiation shield | - | Indicated by reduction in nitrogen evaporation rate. Consult Oxford Instrument Company. |
| Radiation shield insufficiently cold. | | Either the liquid nitrogen vessel is empty - refill, or the radiation shield is touching the outer case indicated by a cold spot on the outer case - consult Oxford Instrument Company. |

N.B. Normal liquid helium evaporation rate is given in Section 1.1 This is with the magnet in persisten mode and the leads removed. The evaporation rate will be higher than this immediately after transferring.

Syphon entry port blocked with ice.

Remove the top cap from chimney and cover the aperture. Warm and dry the cap.

## 2.4.2. MAGNET OPERATION.

Leads cannot be inserted

| | | |
|---|---|---|
| Entry ports blocked with ice | - | See "Syphon entry port blocked with ice" above. |
| Connectors blocked with ice or solid air | - | With the liquid helium level at approx. 50% insert a 10 mm dia. stainless steel tube into the leads entry port. (The blowing out tube provided can be used). The tube should be connected to a supply of helium gas before inserting it, and should be sealed with a bored-out rubber stopper to prevent further ingress of air. Lower the tube until its lower end reaches the connector on the terminal plate and then play a jet of helium gas over it. This will vaporize the solid air condensed on the plate. Remove the tube and try to insert the leads again. |

N.B. By maintaining a slight pressure above atmospheric in the recovery system, ice formation will be prevented.

| | | |
|---|---|---|
| Connectors bent or damaged | - | consult Oxford Instrument Company. |

<u>Magnet does not reach correct current when charging.</u>

| | | |
|---|---|---|
| Current limit incorrectly set | - | check setting. |
| Excessive lead drop | - | check connections. |

<u>Superconducting switch does not open</u>

| | | |
|---|---|---|
| Inadequate heater current | - | increase to maximum of 100 mA |
| Heater connection open circuit | - | check continuity |

<u>Superconducting switch does not close</u>

| | | |
|---|---|---|
| Wrong control operated | - | check controls |
| Inadequate time for switch to cool | - | wait longer. |
| Liquid helium level too low | - | check and refill |
| Switch burnt out | - | consult Oxford Instrument Company. |

<u>Homogeneity poor</u>

| | | |
|---|---|---|
| Shim coils interacting | - | ensure all switches are open during adjustment. |

| | | |
|---|---|---|
| Probe not central | - | check and adjust |
| Magnetic materials present | - | check and remove |

<u>Magnet discharges spontaneously</u>

| | | |
|---|---|---|
| Helium level low | - | refill and re-energise |
| Helium level indicator faulty | - | disconnect and check. (applies when level indicator is interlocked to switch heater unit.) |

<u>Magnet Quenches</u>

| | | |
|---|---|---|
| Helium level low | - | refill, check magnet and safety mechanisms. |
| Leads replaced with incorrect polarity | - | refill, and check magnet. |

### 2.4.3 Combined Over-pressure relief valve and bunsen valve

This device is intended to protect cryostats against a large increase in pressure which could arise should a superconducting magnet quench. When used as a relief valve it forms a positive seal preventing the flow of gas in either direction unless the internal pressure exceeds approximately 0.5 at m. A simple adaption enables the valve to function as a bunsen valve, while still retaining its use as a relief valve. In this condition it is used with cryostats which are not connected to a gas recovery system, and enables the natural boil-off to vent to atmosphere without permitting air to enter the cryostat and cause icing.

<u>Method of operation (a) as a relief valve.</u>

The valve seat (1) is normally closed by a ball (2) sealing against the O-ring (3). The ball is pressed against the seat by the compression of the O-ring (4). If the pressure rises to about 0.5 atm. the ball is forced past the O-ring (4) allowing the gas to vent. To reseat the valve the ball is pressed back against its seat through the hole in the top of the body.

<u>(b) as a combined Bunsen valve and relief valve</u>

In this case, the spacer (5) is inserted between the body of the valve, and the seat. The ball is only held on the seat by its own weight so that gas can exit past it through the annular channel (6). A pressure in the reverse direction however, seals the valve, preventing the ingress of air. A large flow of gas, or a high pressure will force the ball past the O-ring (4), so that its function as a relief valve is retained.

When the cryostat is connected to a recovery system the spacer (5) is discarded so that the gas does not escape. If the cryostat is not connected to a recovery system, the spacer should be fitted and the normal vent blocked with a stopper.

<u>Maintenance</u>

Unscrew the body of the valve from the seat and inspect both O-rings. Clean them and lightly smear them with grease. Reassemble. This operation should be carried out every 6 months.

| | | | [illegible] | BELOW | MIDDLE | ABOVE | BELOW | MIDDLE |
|---|---|---|---|---|---|---|---|---|
| 36.8 | 16.3 | -11.8 | 0 | 20.6 | 20.1 | -28.0 | 48.8 | 20.1 |
| -6.1 | 1.4 | -20.6 | 30 | -14.2 | -4.7 | -28.9 | 1.3 | [illegible] |
| -45.0 | 0.5 | 10.2 | 60 | -42.6 | 2.7 | 12.4 | -11.0 | 7.7 |
| -69.0 | 3.5 | 58.2 | 90 | -57.0 | 15.5 | 70.1 | -17.4 | 15.5 |
| -97.9 | 15.4 | 24.2 | 120 | -79.4 | -16.9 | 42.6 | -42.6 | -26.9 |
| -51.8 | 17.3 | -4.6 | 150 | -31.8 | -27.2 | 15.2 | -7.6 | -27.3 |
| 53.3 | 4.8 | 16.2 | 180 | 69.5 | 21.0 | 32.4 | 74.5 | 21.0 |
| 98.4 | 27.9 | 25.2 | 210 | 106.4 | 15.9 | 33.2 | 90.9 | 35.9 |
| 60.5 | 5.0 | -0.8 | 240 | 56.2 | 2.7 | -3.1 | 26.4 | 2.7 |
| 3.5 | -17.0 | -34.8 | 270 | -8.4 | -28.9 | -46.8 | -48.0 | -28.9 |
| -7.4 | -2.9 | -39.8 | 300 | -25.6 | -21.3 | -58.3 | -62.7 | -21.3 |
| 24.7 | 31.2 | -20.8 | 330 | 4.7 | 11.2 | -40.8 | -19.5 | 11.2 |

TYE 1 FOR MORE RESIDUALS, 0 FOR NO MORE
-00 1 1 5 3

TRANSVERSE VARIATION IN A SAMPLE AXIALLY 1.00 BY 3.00 DIAMETER

MAGNET AT 30.712 MHZ, 46.991 KILOGAUSS

| | HERTZ | DEGREES | GAUSS | PPM |
|---|---|---|---|---|
| X AND Y | 13.4 | 323.6 | 0.021 | 0.44 |
| ZX AND ZY | 13.3 | 97.2 | 0.020 | 0.43 |
| X2-Y2 AND 2XY | 4.5 | 23.2 | 0.007 | 0.15 |
| Z2X AND Z2Y | 4.9 | 187.5 | 0.008 | 0.16 |
| ZC2 AND ZS2 | 2.6 | 105.8 | 0.004 | 0.09 |
| X3 AND Y3 | 1.5 | 89.0 | 0.002 | 0.05 |
| Z2C2 AND Z2S2 | 0.4 | 31.9 | 0.001 | 0.01 |
| ZX3 AND ZY3 | 0.1 | 110.4 | 0.000 | 0.00 |
| C4 AND S4 | 0.2 | 88.1 | 0.000 | 0.01 |

TRANSVERSE VARIATION IN A SAMPLE AXIALLY 2.00 BY 3.00 DIAMETER

MAGNET AT 30.712 MHZ, 46.991 KILOGAUSS

| | HERTZ | DEGREES | GAUSS | PPM |
|---|---|---|---|---|
| X AND Y | 40.3 | 323.6 | 0.062 | 1.31 |
| ZX AND ZY | 79.9 | 97.2 | 0.122 | 2.60 |
| X2-Y2 AND 2XY | 40.3 | 23.2 | 0.062 | 1.31 |
| Z2X AND Z2Y | 59.0 | 187.5 | 0.090 | 1.92 |
| ZC2 AND ZS2 | 50.8 | 105.8 | 0.078 | 1.66 |
| X3 AND Y3 | 41.2 | 89.0 | 0.063 | 1.34 |
| Z2C2 AND Z2S2 | 15.0 | 31.9 | 0.023 | 0.49 |

```
11.00  9870.  1.000     127.92    106.15     -4.78      4.11

11.50  7700.  1.000     -66.26    -61.46      2.45     -0.97

CHOOSE HIGHER DEGREE, ELSE RANGE OF RESIDUALS, NEGATIVE INTEGER ESCAPES TO EVALUATION
-6 -1 6 -6 -1 5 -6

REDUCED DERIVATIVES EVALUATED AT RO=   0.50 FROM  7TH DEGREE POLYNOMIAL FIT OF 17 DATA POINTS EXTEND

POSITION   FIELD  ORDER 1        2         3         4         5         6         7

    7.10  11922.94     -2.52     -1.17      0.49     -0.08      0.02     -0.01      0.00

    7.30  11921.77     -3.25     -0.66      0.37     -0.07      0.01     -0.01      0.00

    7.50  11920.39     -3.62     -0.29      0.23     -0.12     -0.04     -0.01      0.00

    7.70  11918.91     -3.78     -0.17     -0.04     -0.22     -0.06     -0.01      0.00

    7.90  11917.36     -3.99     -0.47     -0.50     -0.37     -0.09     -0.01      0.00


REDUCED DERIVATIVES EVALUATED AT RO=   0.50 FROM  6TH DEGREE POLYNOMIAL FIT OF 17 DATA POINTS EXTEND

POSITION   FIELD  ORDER 1        2         3         4         5         6

    7.10  11919.57      1.38      0.19     -0.08     -0.16      0.04     -0.01

    7.30  11920.15      1.46     -0.03     -0.29     -0.14      0.01     -0.01

    7.50  11920.70      1.26     -0.48     -0.46     -0.11     -0.01     -0.01

    7.70  11921.10      0.80     -1.15     -0.67     -0.17  [illegible]   -0.01

    7.90  11921.12     -0.67     -2.16     -1.03     -0.28     -0.07     -0.01


REDUCED DERIVATIVES EVALUATED AT RO=   0.50 FROM  5TH DEGREE POLYNOMIAL FIT OF 17 DATA,POINTS EXTEND

POSITION   FIELD  ORDER 1        2         3         4         5

    7.10  11850.41     31.47     22.70      3.13     -1.17     -0.01
```