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3

A CRYOGENIC LABORATORY.
ITS DESIGN AND EQUIPMENT

A description of the liquid helium and liquid nitrogen installation in the School of Physical Sciences at St. Andrews, including cost analysis, liquid and gas handling, pumping systems and cryostat manipulation.

A THESIS

presented by

ROBERT HARLEY MITCHELL

to the University of St. Andrews
in application for the Degree of
Master of Science, April, 1970.



Th 5729



Aerial view of North Haugh Science Development with cryogenic area on left hand front corner of Physics block

I hereby declare that this thesis is composed by myself, that it is a record of work carried out by myself, and that it has not been previously presented for a higher degree. The work was carried out in the Physical Laboratories of St. Andrews University under the supervision of Professor J. F. Allen.

.....

I certify that Robert Harley Mitchell has spent the equivalent of seven terms as a research student in the Physical Laboratory of the United College of St. Salvator and St. Leonard in the University of St. Andrews, and he has fulfilled the conditions of Ordinance No. 51 of the University Court of St. Andrews and that he is qualified to submit the accompanying thesis in application for the Degree of Master of Science.

(Supervisor)

ACKNOWLEDGEMENT

The author wishes to record his special thanks to Professor J. F. Allen for his assistance and encouragement during the course of this work.

Among the many others who have contributed in some way are, Mr. H. M. Barkla who allowed me free access to his heat exchanger design figures for the St. Andrews H_2/He liquefier, Professor D. V. Osborne who gave freely of his time during the early operational days of the St. Andrews H_2/He liquefier and encouraged my interest in low temperature work, Dr. D. M. Finlayson who gave valuable help in checking the Collins-A.D.L., Philips layout and this thesis, Mrs. M. Cunningham who has done such an excellent typing job, and Mr. D. Speight who produced the photographs.

I particularly wish to thank Mr. T. Marshall, senior technician in charge of gas liquefaction, without whose willing co-operation and assistance the installation and hence this thesis would never have been completed.

SUMMARY

In the following chapters a short history of gas liquefaction is given from the mid nineteenth century to the present day, together with the development of the cryogenic installation at St. Andrews.

The costs of bulk liquid purchase and local liquefaction are compared and related to variations in consumption and demands on technical manpower.

Building design and plant layout are discussed with a view to making local liquid production as efficient and economical as possible. As liquid consumption increases and the demands on manpower become heavier, methods of automatic monitoring and control of helium gas recovery are suggested together with their effects on overall liquid costs.

Types of pumps and pumping lines suitable for lowering pressures in cryostats are reviewed and optimum sizes and throughputs required for various experimental temperatures determined.

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CHAPTER 1

"An artificial fountain to be turned like an hour glass by a child, in the twinkling of an eye, it holding great quantity of water, and of force sufficient to make snow, ice and thunder, with a chirping and finging of birds, and fhewing of several shapes and effects usual to fountains of pleasure." 'Century of Inventions' Marquis of Worcester 1663.

1.1 Early Experiments in Gas Liquefaction

It was not until more than two centuries after the celebrated Marquis constructed his "cooling engine" that oxygen was liquefied in 1877, for the first time, thus marking what might be called the beginning of cryogenics.

When Faraday in 1828 heated chlorine hydrate in one end of a closed test tube and produced liquid chlorine at the other, cool end, very little was known concerning the relation between a liquid and its vapour. After a few gases had been liquefied at room temperature by simple compression, scientists failed to understand why this technique should not work when applied to air and hydrogen. This failure gave rise to the expression "permanent gases", though Faraday was himself convinced that all gases could be liquefied given suitable conditions. As it became evident that temperature had some bearing on the liquefaction process, attempts were made to cool the gas as well as increase the pressure.

Slowly, after numerous experiments by many investigators, the laws of phase equilibrium became apparent, culminating in the dis-

covery of the critical phenomena by Cagniard de La Tour. His simple experiment consisted of heating a liquid in a closed tube, the disappearance of the meniscus indicating that the tube contained only vapour. As a result of this and numerous other experiments it appeared that a temperature could be reached above which liquid formation was impossible no matter what pressure was applied.

Andrews' (1863) determination of the isothermals of CO_2 contain virtually all that is necessary to understand liquid vapour equilibrium in a one component system.

Following Andrews, we can infer that:-

- (a) Every gas has a definite critical temperature.
- (b) At any temperature below the critical temperature the gas will be liquefied by a compression, and that during liquefaction the pressure will remain constant. The coexistent phases therefore appear as horizontal straight lines in the isothermal on a p-v diagram.
- (c) The lower the temperature below the critical point, the lower the pressure required for liquefaction.
- (d) Above the critical temperature there is no formation of a coexistent liquid phase when the gas is compressed isothermally, even though the density may be increased above that of the liquid.

Having established these facts the problem remained of how to

establish the conditions under which the permanent gases could be liquefied.

To Cailletet (1877) must go the credit for first liquefying oxygen, the technique he used being derived from the accidental liquefaction of acetylene. While compressing acetylene a valve was accidentally opened. The sudden expansion of the gas produced a mist in the glass tube, which he correctly deduced to be liquid acetylene. On repeating the experiment with oxygen compressed to 300 atmospheres and cooled to the temperature of evaporating sulphur dioxide he again observed the mist of condensing liquid which appeared on expansion through the valve.

The cascade process devised by Pictet (1877) was, however, a much more practical method of producing liquid. It depended on several stages of cooling, each with a different gas, where progressively lower temperatures were obtained. The first stage used SO_2 , a gas easily liquefied at room temperature, condensed under pressure and expanded into a separate vessel. By rapid evaporation, the temperature in this vessel was reduced below the critical point of the second gas, CO_2 . This was compressed at the SO_2 temperature, expanded and evaporated to produce solid CO_2 at -195°K . A tube containing oxygen at high pressure was immersed in the solid CO_2 and when the pressure was released the oxygen liquefied.

It is interesting to note that the sublimation temperature of

CO_2 is 195°K and the critical temperature of oxygen is 154°K , so the liquefaction actually took place in the manner of Cailletet although that was not the interpretation which Pictet put upon it at the time.

By 1887 Wroblewski and Olzewski at Cracow had improved techniques and had liquefied all the gases, except hydrogen and helium, and had obtained much information relating to boiling points, critical pressures, critical temperatures, etc. They pioneered the use of ethylene in the cascade process, the advantage being that ethylene can be liquefied at just below room temperature but when pumped to a pressure of 10 mm Hg can be reduced in temperature to 125°K . The first air liquefier of any size used the cascade principle; it was constructed by Kamerlingh Onnes at Leiden in 1894, Fig. 1 and produced liquid at the rate of 14 litres/hr. It remained in use at that laboratory for many years.

1.2 Liquefaction of Hydrogen and Helium

Experimenters were now faced with the difficulty that while they could produce a mist of liquid hydrogen in a tube by rapid expansion they could not actually produce liquid by the cascade method. The latter depends on having a range of substances such that the critical point of one is lower than the triple point of the other. There were no gases to span the gap in temperature between liquid nitrogen and the expected critical temperature of

hydrogen.

Dewar's brilliant but essentially simple invention in 1892 of the vacuum insulated vessel was probably the turning point in the struggle to liquefy hydrogen. Using double walled glass vessels, with silvered surfaces to reduce radiation losses, he was able to keep liquid gases for periods which had hitherto been impossible.

In 1852 Joule and Thomson first reported the cooling of a gas by expanding it through a porous plug. This cooling occurs for all gases provided they are below their characteristic inversion temperature prior to expansion. The effect can be most simply described as an internal work process, since the work is done in separating molecules against their attractive forces. Thus an ideal gas will show no change in temperature when passing through a porous plug. The effect of interatomic forces in a real gas can be described in terms of departures from both Joule's law and Boyle's law. The departure from Joule's law produces a cooling at all inlet temperatures but the departure from Boyle's law produces cooling at low temperatures and heating at high temperatures. The latter dominates at high temperatures. Since these departures are characteristic for each gas, there is an inversion temperature characteristic of each gas. If one uses the van der Waals equation of state, then the inversion temperature is equal to twice the Boyle temperature; and

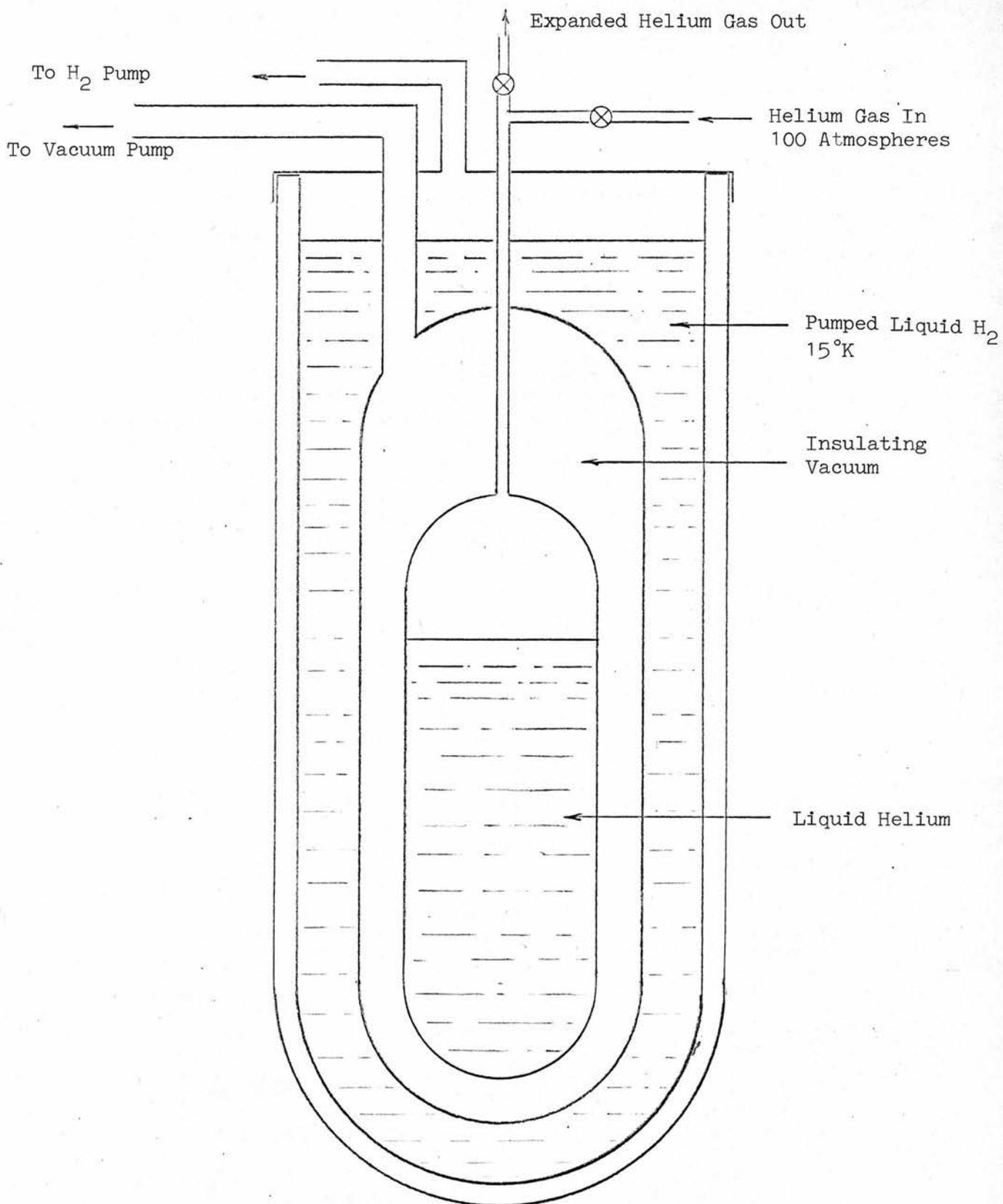
experimentally this is true for the cryogenically important gases to within about 10%.

Dewar first liquefied hydrogen at the Royal Institution in 1898 and collected about 20 ccs before the apparatus became blocked by impurities. A combination of Linde circuit and Joule-Thomson expansion was used but little information on the machine was ever published.

In the Linde cycle the gas which has been cooled by Joule-Thomson expansion is passed back over the pipe which contains the incoming high pressure gas, thus cooling it before it expands. By the use of this heat exchange process the temperature before the Joule-Thomson expansion can be reduced sufficiently to bring about the liquefaction of air, although precooling of hydrogen is necessary, to take it below its inversion temperature of 205°K .

Applying the same principle as had been applied to hydrogen liquefaction by Dewar, Kamerlingh-Onnes succeeded in liquefying helium at Leiden in 1908. As the inversion temperature of helium is 51°K it was not sufficient to provide liquid air under reduced pressure as the only precooling medium.

The first stage of cooling was accomplished by using air but a bath of hydrogen boiling under a pressure 60 mm of mercury had to be incorporated to bring the helium temperature down to 14°K . This, the first liquefaction of helium, was a tremendous achievement



Schematic Diagram Simon Single Expansion Helium Liquefier

Fig. 1.2

when one considers the limited range of materials and technical resources available at the time.

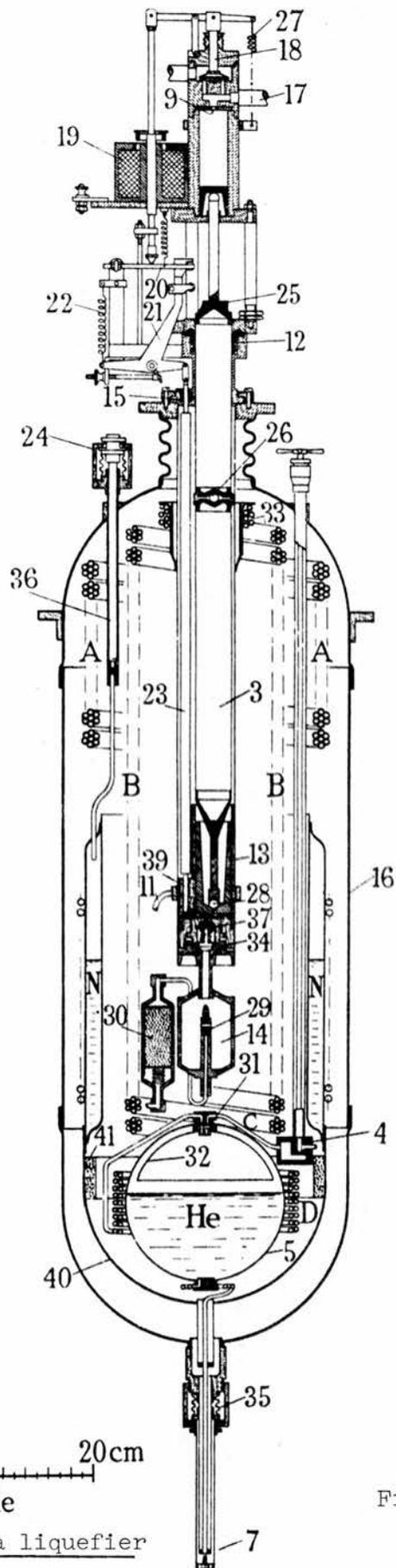
In 1908, the total stock of helium gas available for liquefaction was 360 litres at NTP, or a little over 0.5 litre of liquid making no allowance for the gas retained in the system of liquefier and compressor.

No further helium liquefiers were constructed outside Leiden until McLennan built a machine at Toronto in 1923 from drawings furnished by Kamerlingh-Onnes.

The Simon (1932) single expansion liquefier was the next successful development in helium liquefaction. In this elegant device helium was compressed isothermally into a chamber to a pressure of 100 atmospheres and maintained at a temperature of 15°K; heat being transferred from the vessel containing the compressed helium to the hydrogen bath through the medium of helium exchange gas. After removing the exchange gas the compressed helium was allowed to expand slowly through a valve to a pressure of one atmosphere. The gas in the chamber cooled adiabatically, leaving a substantial amount of liquid helium in the chamber. Fig. 2.

1.3 Expansion Engine Liquefiers for Helium

Kapitza (1934) accomplished the considerable engineering feat of constructing an external work helium liquefier with an expansion engine working below the temperature of liquid hydrogen. As no liquid



0 10 20cm
Scale

Fig. 1.3

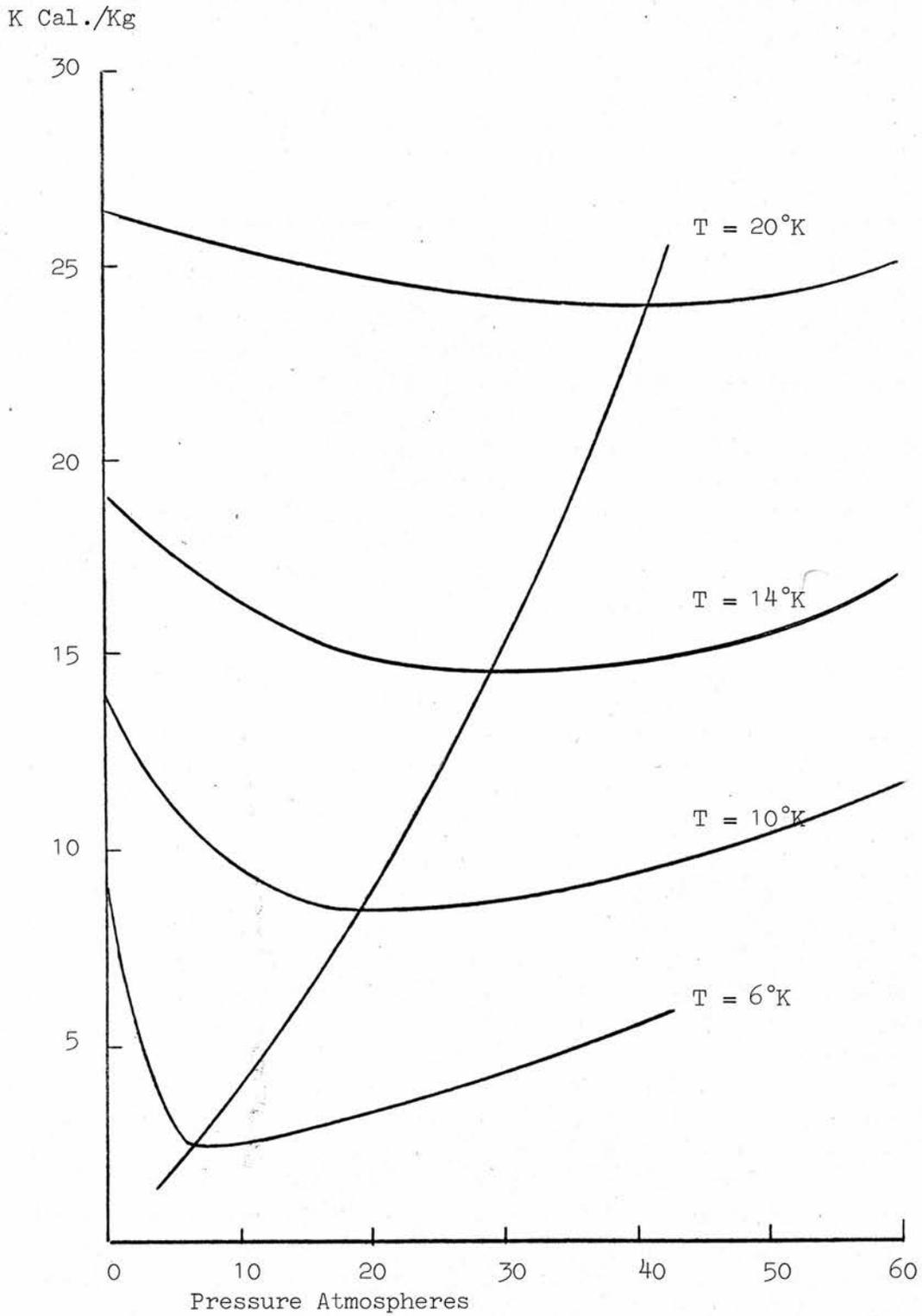
Section through Kapitza liquefier

FIG. 3—Detailed drawing of the liquefier

lubricants exist at these temperatures a diametral clearance of 0.05 mm was left between the piston and cylinder. Pressure equilibrium grooves were machined on the piston to keep it central in the cylinder and so avoid metal to metal friction. The expansion stroke of the single engine varied between 3.5 and 5 cm and was controlled by a hydraulic piston and cylinder mounted external to the liquefier. To reduce gas blow-by past the piston to negligible proportions the cycle was made non-sinusoidal; the expansion stroke being completed in about 1/10 second and the compression stroke more slowly, giving a working rate of 100-120 strokes per minute.

Referring to Fig. 3, compressed helium at 30 atmospheres was first cooled by outgoing helium and nitrogen vapour before it was cooled by contact with liquid nitrogen at 65°K. A further helium exchanger provided cooling for the incoming helium before part was fed to the expansion engine at 20°K. After expansion, the gas left the engine at 10°K cooling the rest of the incoming helium before it was expanded at the Joule-Thomson valve where a fraction liquefied.

The maximum liquefaction rate attained was 1.9 to 2 litres per hour although the expansion engine efficiency was such that 4.7 litres per hour should have been possible. In his paper, Kapitza (1934) suggested that the performance was restricted by the outlet temperature of the expansion engine reaching a tempera-



Variation of Enthalpy with Pressure for Helium at Several Temperatures

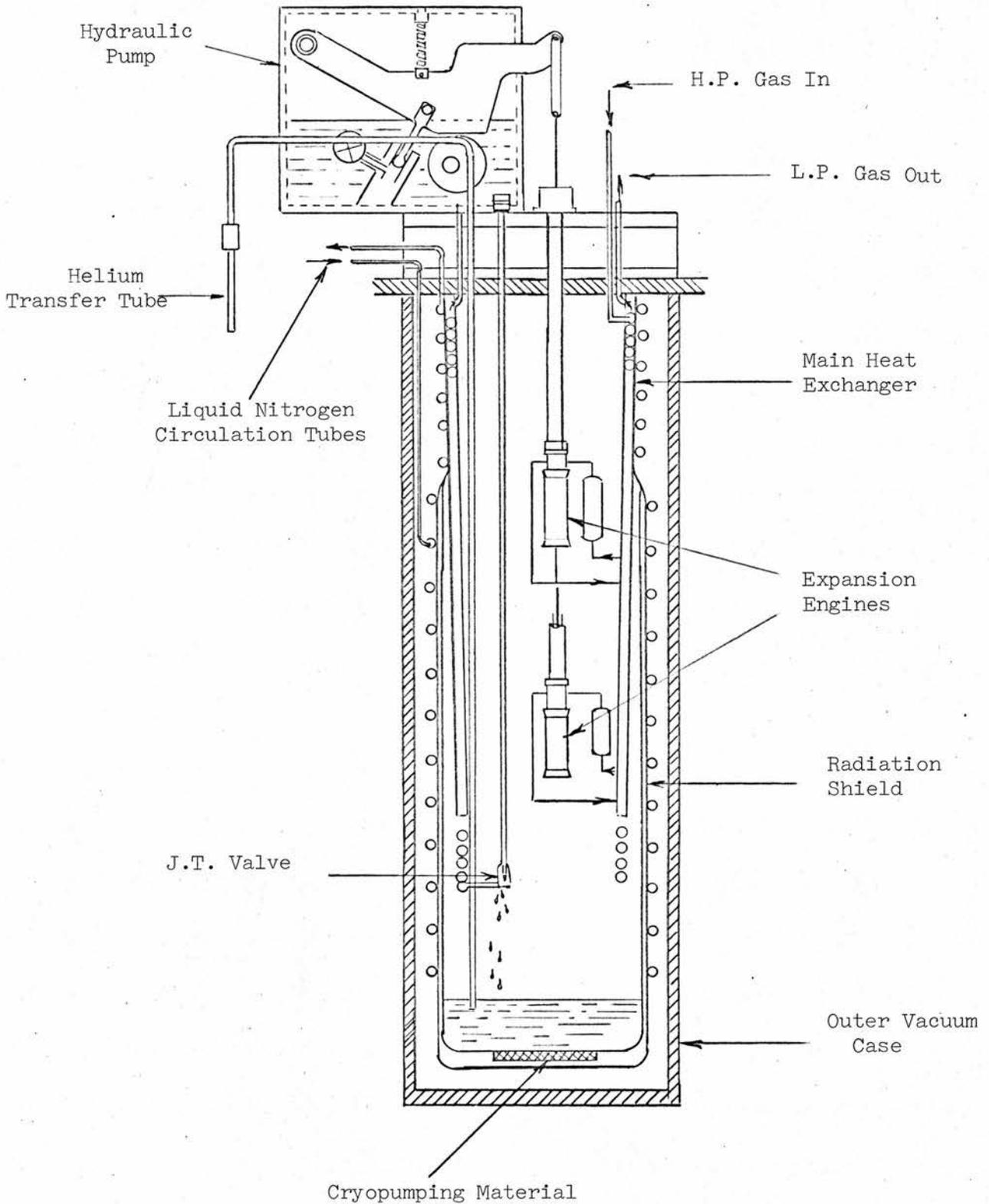
Fig. 1.4

ture of 6°K and an inversion of the Joule-Thomson effect if the incoming gas pressure was not reduced from 30 atmospheres to 17 atmospheres. It is true that the enthalpy of helium at 10°K is at a minimum at 17 atmospheres, Fig. 4 and hence the liquid produced by Joule-Thomson expansion is at a maximum in these conditions but this does not seem to account for all the reduced yield. It may be that some considerable fraction of the reduced yield arose from the geometry of the liquid collecting vessel and the positioning of the liquid transfer tube. The transfer tube was connected directly to the bottom of the spherical liquid helium container⁵ and passed through the vacuum space to a needle valve on the outer case of the liquefier. This meant that immediately liquid began to collect it would run down into the transfer tube, evaporate, be replaced by more liquid, evaporate, and so on. Due to the very low density and viscosity of liquid helium, it is also possible that a Taconis (1949) type oscillation could have been set up. It has been observed that when a tube of suitable dimensions is placed in liquid helium and the other end closed and maintained at room temperature or at any rate warm compared with 4°K , spontaneous oscillations may be set up in the gas column in the tube. Any fluctuation in pressure in the tube will cause the helium level in it to rise or fall. If it rises and reaches a warm part of the tube some liquid will evaporate and increase the pressure and so the level is depressed. On depression gas will condense and lower the pressure.

The system is now in a state of positive feedback. This causes heat pulses to be transmitted down the tube into the liquid so boiling it off. (Bannister 1966, Norton and Muhlenhaupt 1967.) In the Kapitza liquefier Fig. 3 helium liquid was withdrawn from the bottom of the container and the bottom (valve) end of the transfer tube was warm when closed. In his explanation of low liquid yield Kapitza states that improved making rates could be obtained by bleeding gas through the transfer tube valve and thereby lowering the pressure before the Joule-Thomson valve. This action could also have had the effect of greatly reducing or eliminating any possible oscillations both by providing a small opening in an otherwise closed tube and also by diminishing the temperature gradient down it. Processes of the types described could easily have resulted in large amounts of heat being fed into the liquefier, evaporating liquid; thus causing the expansion engine to run at too low a temperature for maximum efficiency.

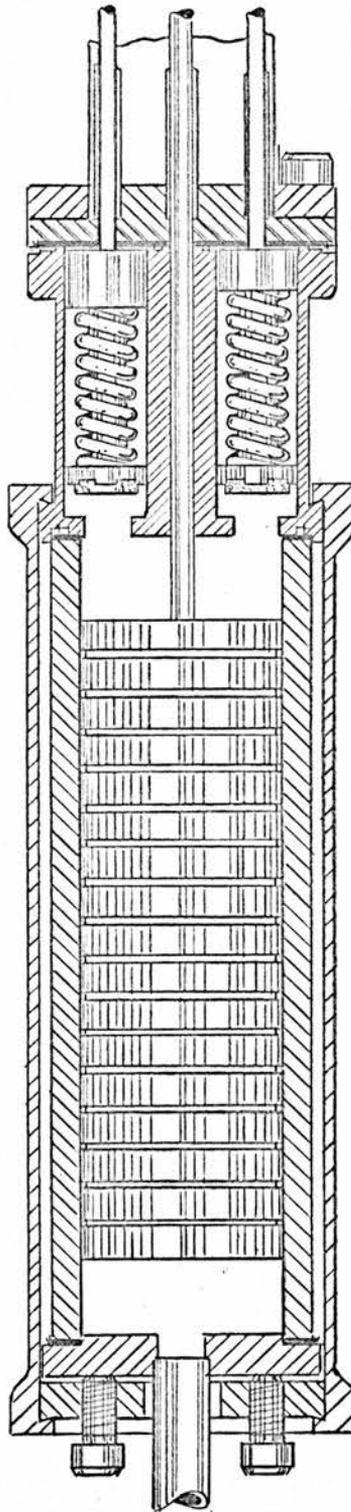
During the years 1930-1940 a few more hydrogen and helium liquefiers were built in various research laboratories in Russia, Europe and America. These were mainly small machines using Joule-Thomson expansion to produce liquid in what was very often the experimental space.

Probably the greatest single stimulus to the development of low temperature research was the adaptation by Collins (1939) of



Schematic Section through A.D.L. Collins Helium Liquefier

Fig. 1.5



Section through Collins Expansion Engine

Fig. 1.6

the Kapitza liquefier. There were three main features in the Collins machine which transformed liquefaction from cranky laboratory apparatus to commercial technology:- (Figs. 5 and 6)

1. By using two expansion engines, one extracting heat at 60°K and the other extracting heat at 15°K , helium could be liquefied without the aid of a liquid nitrogen cooling cycle.
2. The main heat exchanger was made by enclosing a helix of cupronickel tubing, fitted with a spiral copper fin, between two closely fitting slightly conical stainless steel moulds of large diameter. This gave a cross flow exchanger with low pressure drop in the returning gas stream whose flow was essentially parallel to the cone axis. The exchanger was highly efficient and comparatively simple to manufacture.
3. The space in which the engines and heat exchangers were hung was filled with helium gas at the pressure existing on the low pressure side of the expansion valve. Minor leaks from gasketed joints on the engines or mechanical couplings in the piping were thus of little consequence and care in making high vacuum sealed plumbing joints was unnecessary. Since the temperature gradient in the gas was negative downwards there was no appreciable convection. The whole assembly was contained within a large metal dewar to isolate it from room temperature.

In the Collins design the expansion engines were so arranged

that the piston rods were always in tension. This had two advantages: the rods could be of sufficiently small cross-section to conduct a negligible amount of heat, and their flexibility allowed the pistons to align themselves precisely in the cylinders. Grooves cut on the piston as in the Kapitza machine assisted with centralisation within the cylinder and with gas lubrication. With this arrangement very small diametral clearances of 0.01 mm could be employed. Similar small diameter rods in tension were used for operating the inlet and outlet valves, which were fitted with leather seats.

Reciprocating motion for the pistons and valves was provided by cams with pivoted arms. The thermal energy of the gas was converted to mechanical work by attaching oil pumps to the other ends of the pivoted arms, and the heat so produced was simply dissipated to the surrounding atmosphere.

Valve and piston rod packing glands were at the room temperature end of the rods so there were no problems with low temperature sliding seals. Simple O-ring packings were used with lubricant-impregnated felt washers.

Further development of the liquefier by Arthur D. Little Inc. has resulted in a complete installation being marketed with compressors, gasholder and gas recovery system.

Unfortunately, while the A.D.L.-Collins machine was very

satisfactory and reliable it was also very costly, and only well-endowed laboratories could contemplate its purchase. This meant that a substantial number of Joule-Thomson, Linde liquefiers continued to be built in various laboratories. These included not only machines requiring an external source of liquid hydrogen but also combined liquefiers where both systems were contained within the one case, liquefaction of helium and hydrogen being conducted simultaneously.

Included among the first type were the liquefiers at Leiden (15 litres/hr), Ohio State University (Daunt and Johnston, 1949, 7.5 litres/hr), Clarendon Laboratory Oxford (Croft and Bligh, 1961, 14 litres/hr) and R.R.E. Malvern (Parkinson, 1960, 9 litres/hr).

Of the second type Ashmead (1950) at Cambridge constructed a combined helium-hydrogen liquefier with a helium production rate of 4 litres/hr. Similar machines were constructed at Paris (Spoendlin, 1954, 5 litres/hr), St. Andrews (3.6 litres/hr) and Grenoble (Lacaze and Weil, 1955, 7 litres/hr). The Lacaze and Weil liquefier was put into commercial production by T.B.T. Grenoble and several of these machines were sold to French universities and industrial laboratories as well as one to Nottingham University. They were complicated to operate but had a reasonable record for reliability in spite of the exchangers being insulated by a high

vacuum.

A larger helium liquefier working on the Joule-Thomson Linde principle, designed by Mann, Bjorklund and Macinko, was built at the National Bureau of Standards Laboratory at Boulder Colorado, and produced 15 litres/hr.

Further development by Collins (1952) utilising three expansion engines resulted in a production rate of between 25 and 32 litres/hr. Much larger machines were designed by Collins for Navajo, Arizona and with this unit a production rate of 120 litres/hr was obtained.

1.4 Liquefaction by Turbine Engines

At present what is probably the largest helium liquefier in the world is at Otis Kansas and has a production rate of 800 litres of liquid helium per hour. It was designed and built by Sulzer Brothers Ltd., Switzerland for NASA and contains two cold gas turbines operating at 32°K and 10°K. The small size of turbines compared with piston machines of similar capacity is attractive, as is the case in power stations and large prime movers, but the technical problems are formidable.

The first published account of a successful turbine operating at low temperature was due to Kapitza (1939). His turbine was used in an air liquefier which produced about 30 litres/hr.

The Sulzer, Ergenc and Trepp (1964) turbine is similar to the

Kapitza turbine being of the single stage inward radial flow type. The oil lubricated bearings are located in a region away from the cold gas and the energy is absorbed by an oil brake.

Further development is likely in the field of turboexpanders probably by the use of gas bearings. Oil lubricated bearings cannot of course be run in the cold zone and the maximum permissible speed is around 120,000 r.p.m. Using gas bearings speeds of over 300,000 r.p.m. are attainable, which would again make for much higher efficiencies.

1.5 Conclusion

Between the first liquefaction of helium in 1908 by Kamerlingh-Onnes and 1940 there were probably less than a dozen laboratories throughout the world where helium was regularly liquefied. In 1965 there were many hundreds of laboratories where low temperature research was being actively conducted and liquefaction of helium was almost taken for granted. Much of the credit for this state of affairs must go to Professor S. C. Collins, and it is indicative of the success of his machines as produced by Arthur D. Little, that by 1965 three hundred of the standard units had been produced and sold throughout the world.

CHAPTER 2

2.1 A Joule Thomson Cascade Hydrogen-Helium Liquefier

From 1952 until 1956 helium liquefaction at St. Andrews was confined to a small cryostat/liquefier using straightforward Joule-Thomson expansion of helium precooled by liquid hydrogen from a small Linde type liquefier. This hydrogen liquefier produced about 4.2 litres of liquid/hour and the helium liquefier 750 ccs/hour. Increasing interest in low temperature research made it imperative that liquid helium should be made available in comparatively large quantities and that the facility for liquefying hydrogen should be retained. As no money was available for the purchase of new compressors and vacuum pumps, existing equipment had to be utilised in the new machine. The existence of the small H₂ liquefier in running order and with all the attendant services for gas recovery, purity monitoring, high pressure gas storage etc., made it reasonable to examine helium liquefaction processes which require supplies of liquid hydrogen.

In terms of thermodynamic efficiency the Simon (1932) single expansion helium liquefier lends itself well to these cases where the day's supply of helium can be produced in a single expansion. If, however, a number of compressions and expansions have to be made, then the continuously running liquefier will have a higher average yield.

A larger model of the Joule-Thomson expansion cryostat/

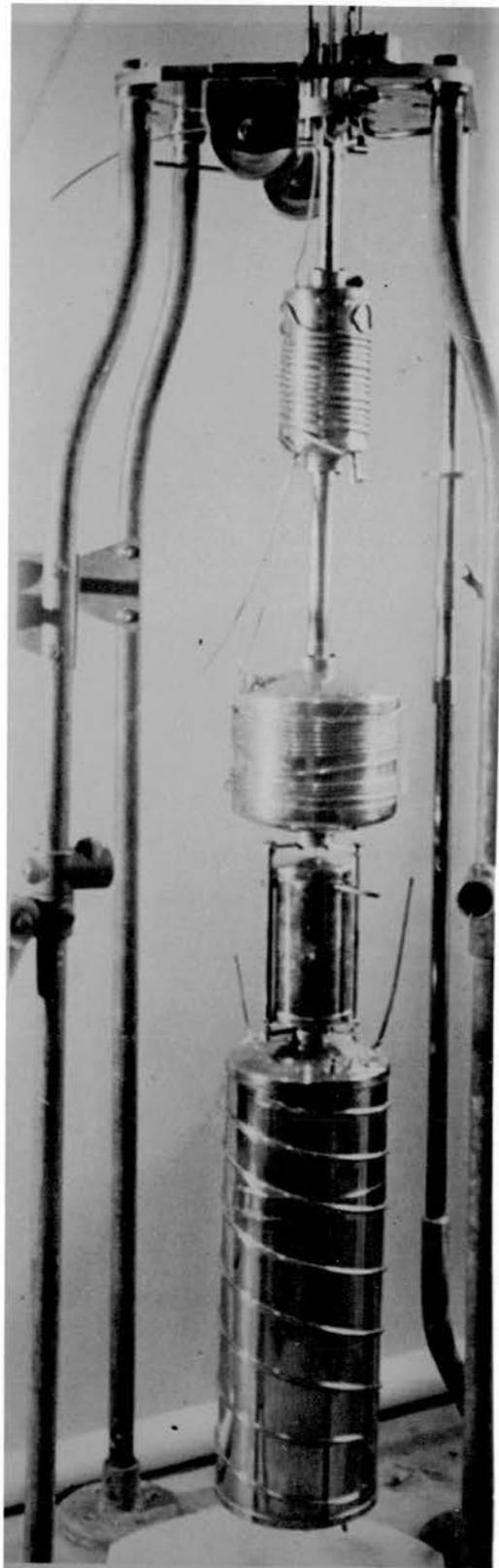


Photo 2:1 St. Andrews Liquefier Central Support Arrangement.

liquefier previously mentioned was an attractive alternative but the inherent dangers involved in handling large quantities of liquid hydrogen in an unsuitable building outweighed the comparative simplicity of this solution. Also two starting times are required for such a technique, and unless storage facilities exist for previously made liquid, two operators are also needed.

Following the success of the Ashmead (1950) liquefier in Cambridge it was decided that a somewhat similar machine would suit St. Andrews. Two liquefiers would be combined within the same outer case. When in use as a hydrogen liquefier, the helium circuit would remain empty, and when in use as a helium liquefier a constant amount of liquid hydrogen would remain in the machine to provide the necessary cooling of the helium below its inversion temperature. In the latter duty cycle the hydrogen circuit could be closed and since the liquid need not be handled or transferred the explosion hazard was greatly diminished. Combining the two machines in this way meant that the starting time was longer than for either single liquefier separately, but not as long as the sum of the two.

2.2 General Design and Gas Circuits

The complete liquefier assembly was supported from the top plate using a central spine arrangement (Photo 1). The spine consisted of thin wall stainless steel tube at the top, followed

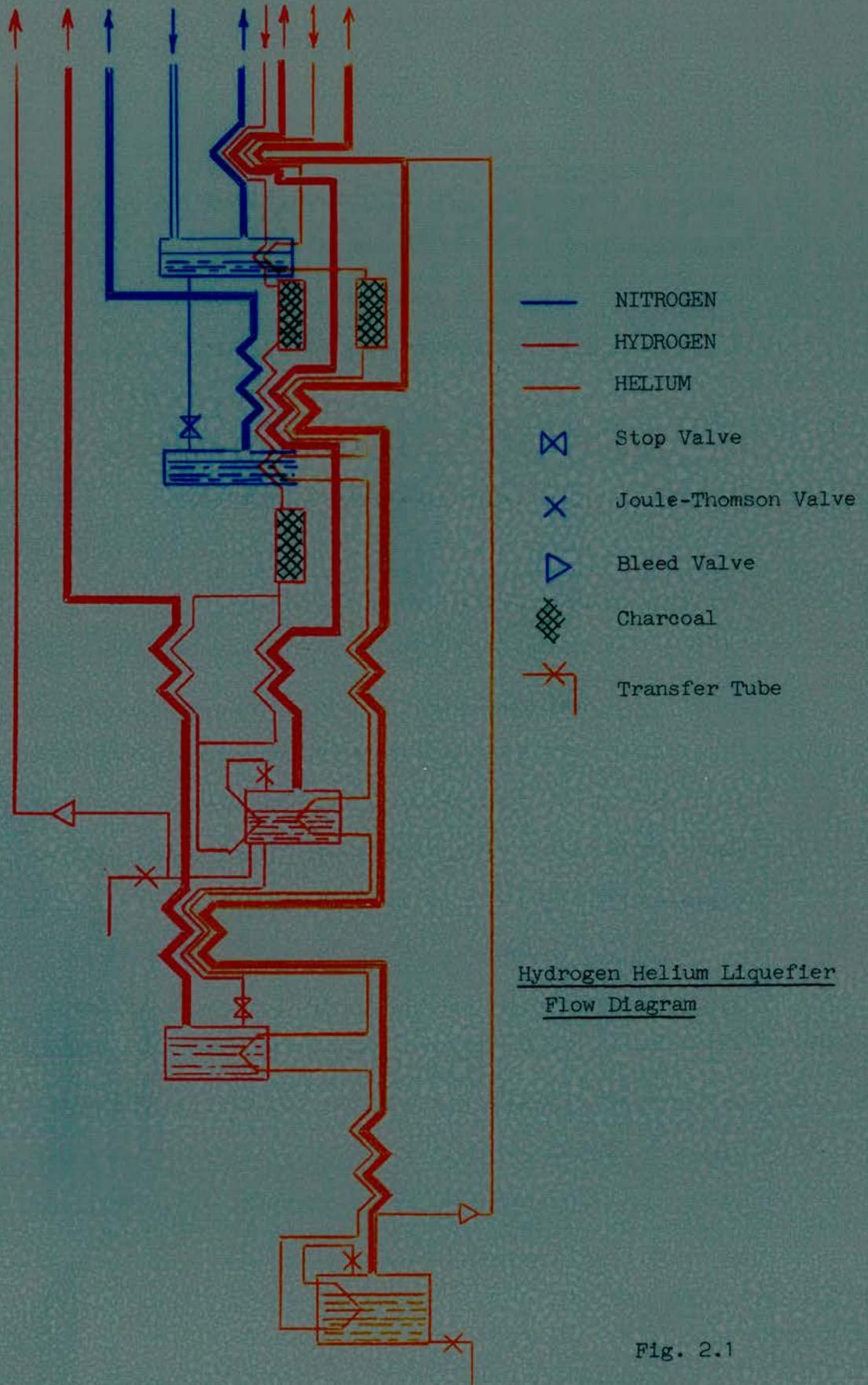


Fig. 2.1

downwards in sequence by the 65°K pumped nitrogen can, another stainless steel tube, the 23°K hydrogen can and finally a tubular cage surrounding the 14.5°K pumped hydrogen can supported the helium can. This meant that the 65°K N₂ can and 23°K H₂ can formed load bearing members as well as being liquid containers. While this form of construction resulted in a fairly long liquefier (Photo 2), about 150 cms from top plate to floor level, it provided a rigid axial framework to build on.

Referring to the flow diagram shown in Figure 1, the incoming streams of high pressure hydrogen (120 atmospheres) and helium (25 atmospheres) were cooled by returning low pressure H₂, He and nitrogen from the radiation shield standpipe. Extending from top to bottom of the liquefier this 50 mm I.D. brass tube contained liquid N₂ boiling at 1.3 atmospheres, 80°K. It formed the reservoir for the radiation shield and source of liquid for the pumped N₂ can.

Leaving the bottom of the first exchanger the two high pressure gas streams passed through two vertical 6 mm O.D. x 5 mm I.D. german silver tubes soldered to the standpipe, before entering two similar activated charcoal purifiers. The charcoal was contained in stainless steel tubes 25 mm I.D. x 100 cms long. Cooling of the charcoal being simply accomplished by the flow of cold gas, no cooling of the purifiers by mechanical contact or liquid

immersion was necessary. Passing to the next exchanger, the high pressure streams were cooled by returning H_2 , He and N_2 from the pumped $65^\circ K$ can, and before being passed to the third series of exchangers were directed through tubes soldered to the outside of this can. A further activated charcoal trap (25 mm I.D. x 30 cms long) at this point in the high pressure hydrogen line removed any remaining impurities and acted as a partial ortho-para convertor. Immediately following this trap the high pressure H_2 stream was split, one branch taking about 20% of the gas exchanged with low pressure hydrogen gas returning from the $14.5^\circ K$ can, the rest being cooled by hydrogen gas returning from the $24.5^\circ K$ can. The two streams were recombined in a coil soldered to the outside of the $24.5^\circ K$ can before expanding at a Joule-Thomson valve to a pressure of 2.8 atmospheres into the can. Here 35% of the gas was liquefied and collected, the remainder passed back up the heat exchangers for recycling. A small fraction, 15% of the liquid was transferred intermittently through a valve into the lower H_2 can where it was reduced in pressure and temperature by means of a rotary vacuum pump to 80 mm Hg and $14.5^\circ K$, close to the triple point. This liquid transfer line formed one element of a small exchanger with gas being pumped from the $14.5^\circ K$ can. This pumped H_2 gas passed only through the exchanger immediately above the $24.5^\circ K$ can before going direct to the top plate through a large

diameter tube. While this resulted in a certain loss of thermal efficiency it solved the difficulty of maintaining a high pumping speed at the 14.5°K can.

Returning now to the high pressure He gas, it exchanged only with returning low pressure He before passing through a coil soldered to the outside of the 24.5°K can. It was passed through a combined exchanger with returning low pressure He and pumped H₂ before the coil soldered to the outside of the 14.5°K can and so to the final exchanger. Before reaching the Joule-Thomson valve, the He passed through a coil soldered to the lower part of the liquid He can. It was then expanded at the Joule-Thomson valve to a pressure of 1.42 atmospheres and a fraction liquefied.

Soldering the final part of the high pressure tube to the outside of the can resulted in the gas liquefying before it reached the Joule-Thomson valve. This technique, which is attributed to Simon meant that liquid was expanded rather than the gas which resulted in much quieter conditions, and hence more efficient liquid collection in the can.

To facilitate initial cooling, bleed valves were fitted to both H₂ and He circuits short circuiting the upper exchangers. In the case of the H₂ circuit the bleed valve was placed in the liquid transfer line immediately above the 24.5°K can and the gas carried directly to the top plate. The He bleed valve was fitted

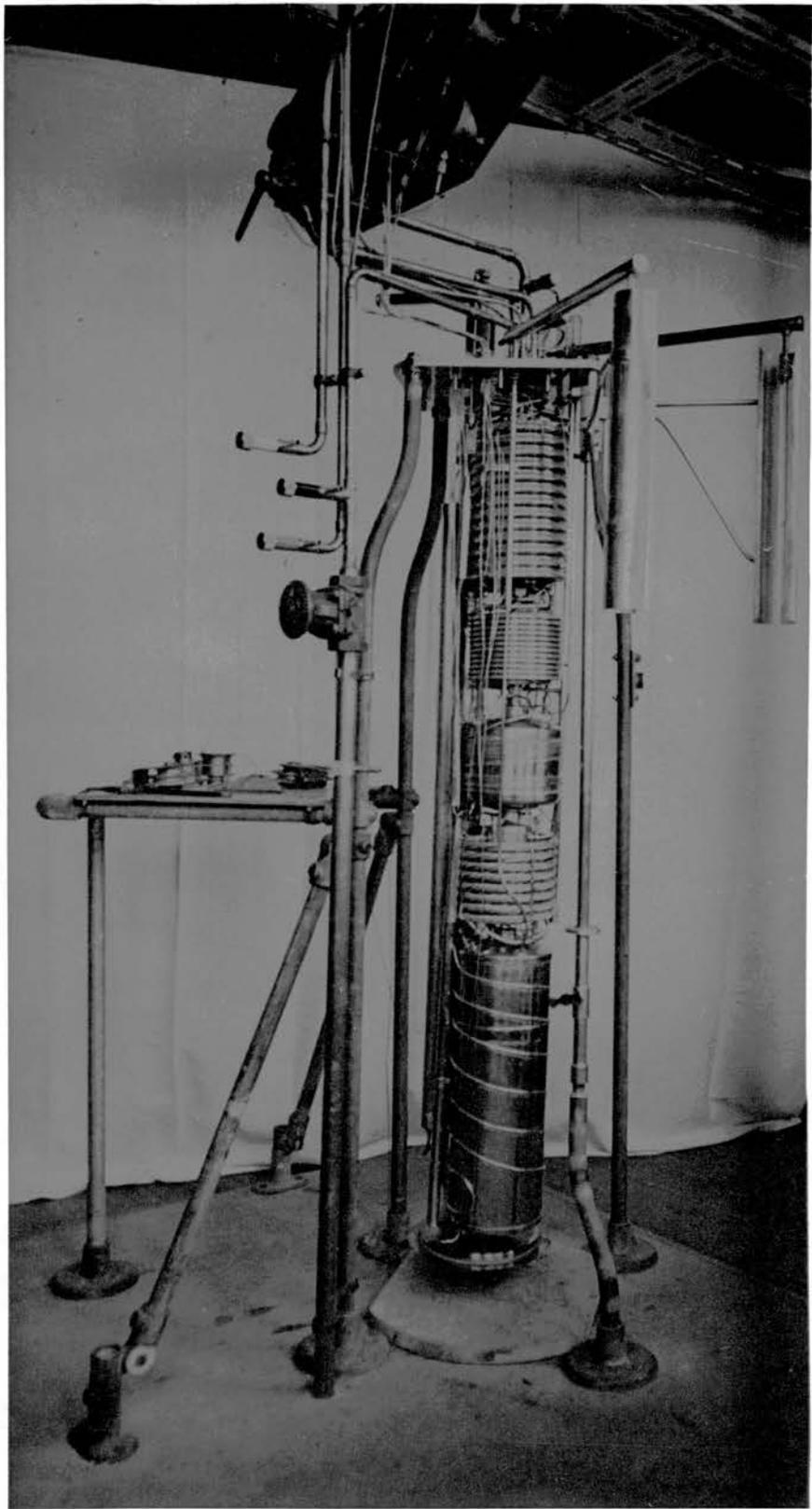


Photo 2:2 St. Andrews Liquefier with Radiation Shield Removed
Showing Heat Exchangers.

at the cold end of the final exchanger but due to lack of space at the top plate it was not possible to bypass the first exchanger and take the gas directly to room temperature.

2.3 The Heat Exchangers

Construction of the heat exchangers followed mainly the solder-bonded parallel tube pattern, great care being taken to ensure that the tubes fitted snugly together before being soldered (Photo 2).

Originally the final exchanger was made from six 3.25 mm I.D. x 4.9 mm O.D. copper tubes, the high pressure gas passing through the centre tube and the returning low pressure gas through the five others twisted round it. The low pressure tubes were soldered to the centre tube to provide good thermal contact and the whole rope-like bundle, 4 metres long, wound into a helix 15.6 cms O.D. The complete exchanger weighed about 2.4 K.G. but a substantial fraction of this was surplus solder due to the unavoidably open twisted construction.

After four years of operation it was decided to replace this exchanger with one of very much lighter construction; firstly the wall thickness of the tubing was reduced from 0.8 mm to 0.2 mm with the bore size maintained at 3.25 mm, and secondly it was constructed in the form of a seven tube cluster. In this form the

Flexible Metal
Strip Hinges

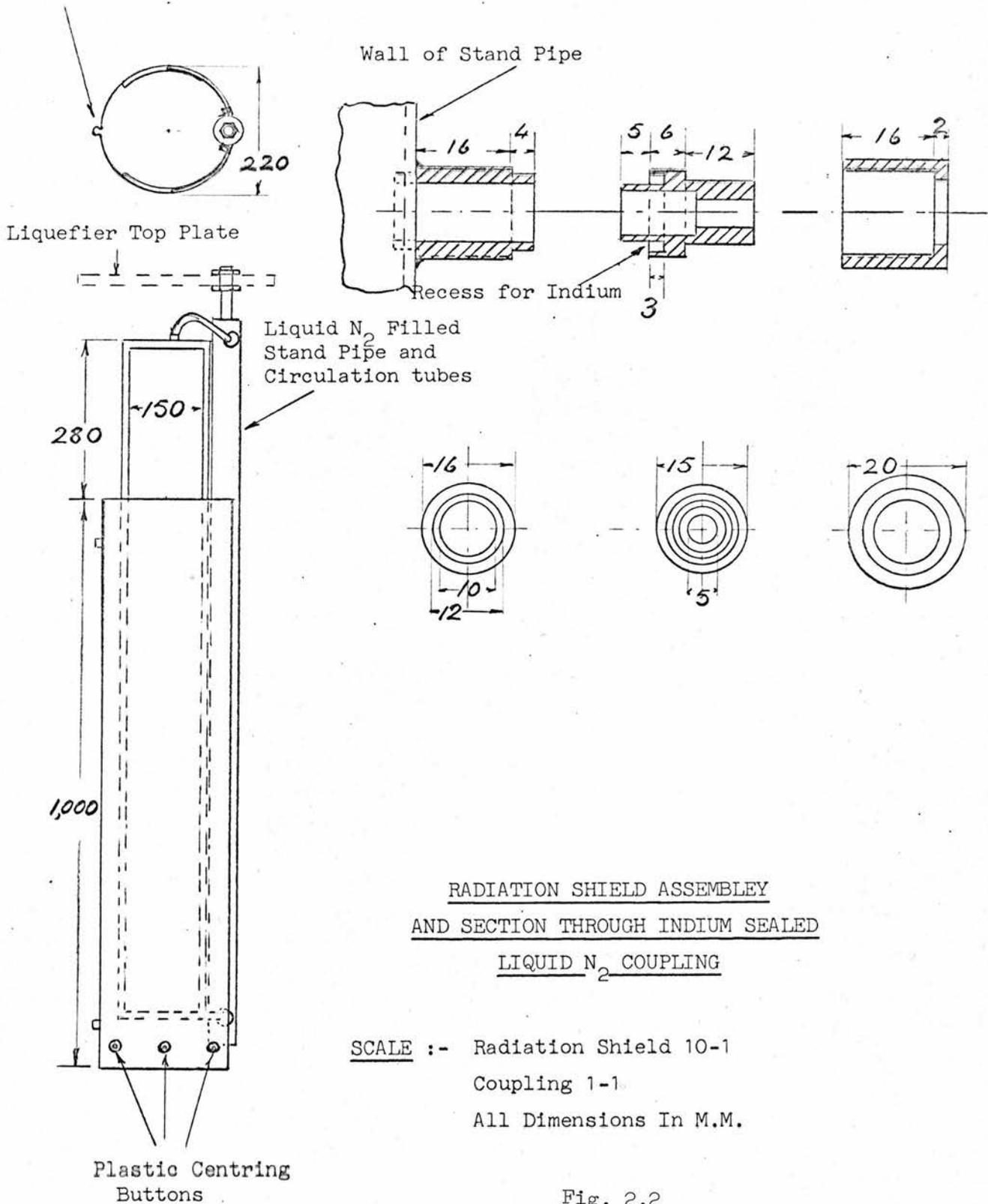
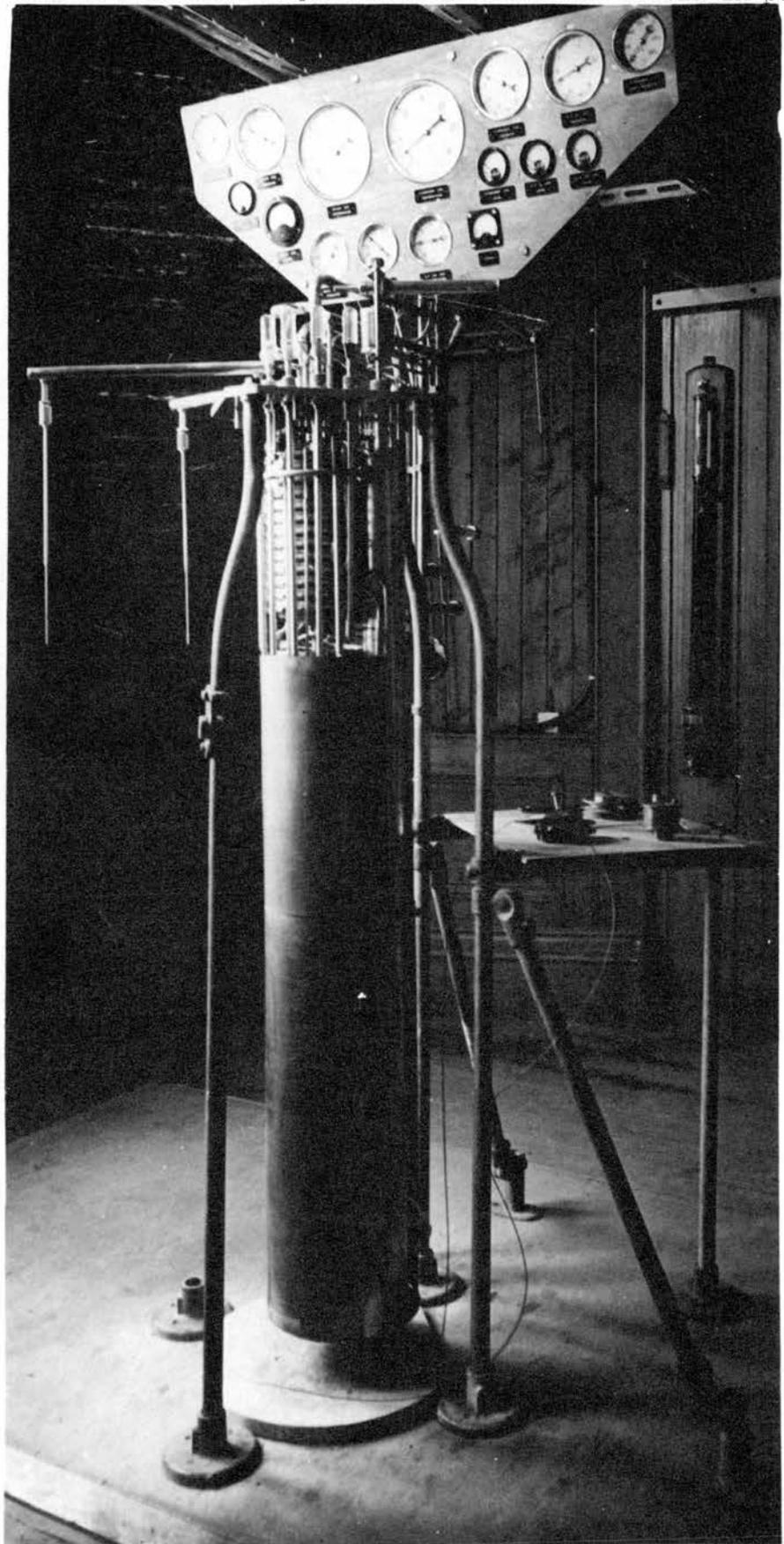


Fig. 2.2

Photo 2.3 St. Andrews Liquefier with Radiation Shield in position



high pressure gas was passed through the centre tube as before but the six identical low pressure tubes lay snugly parallel to one another so reducing the amount of solder required to establish thermal contact. With the same length as before the new final exchanger weighed 0.71 K.G.

At the same time a new He can was made, the capacity reduced from 7.5 litres to 5 litres, the reduction being mainly in diameter to give increased clearance between the can and the radiation shield.

2.4 The Radiation Shield Figure 2 and Photo 3

The 1 mm thick copper radiation shield was of unusual design being split vertically and hinged to make removal, for internal access, and replacement a simple operation. The liquid nitrogen reservoir for this shield was the 50 mm diameter brass tubular stand pipe extending from top to bottom of the liquefier. Two pairs of indium sealed couplings were connected to loops of pipe soldered to the radiation shield. In this way liquid was distributed over the area of the shield.

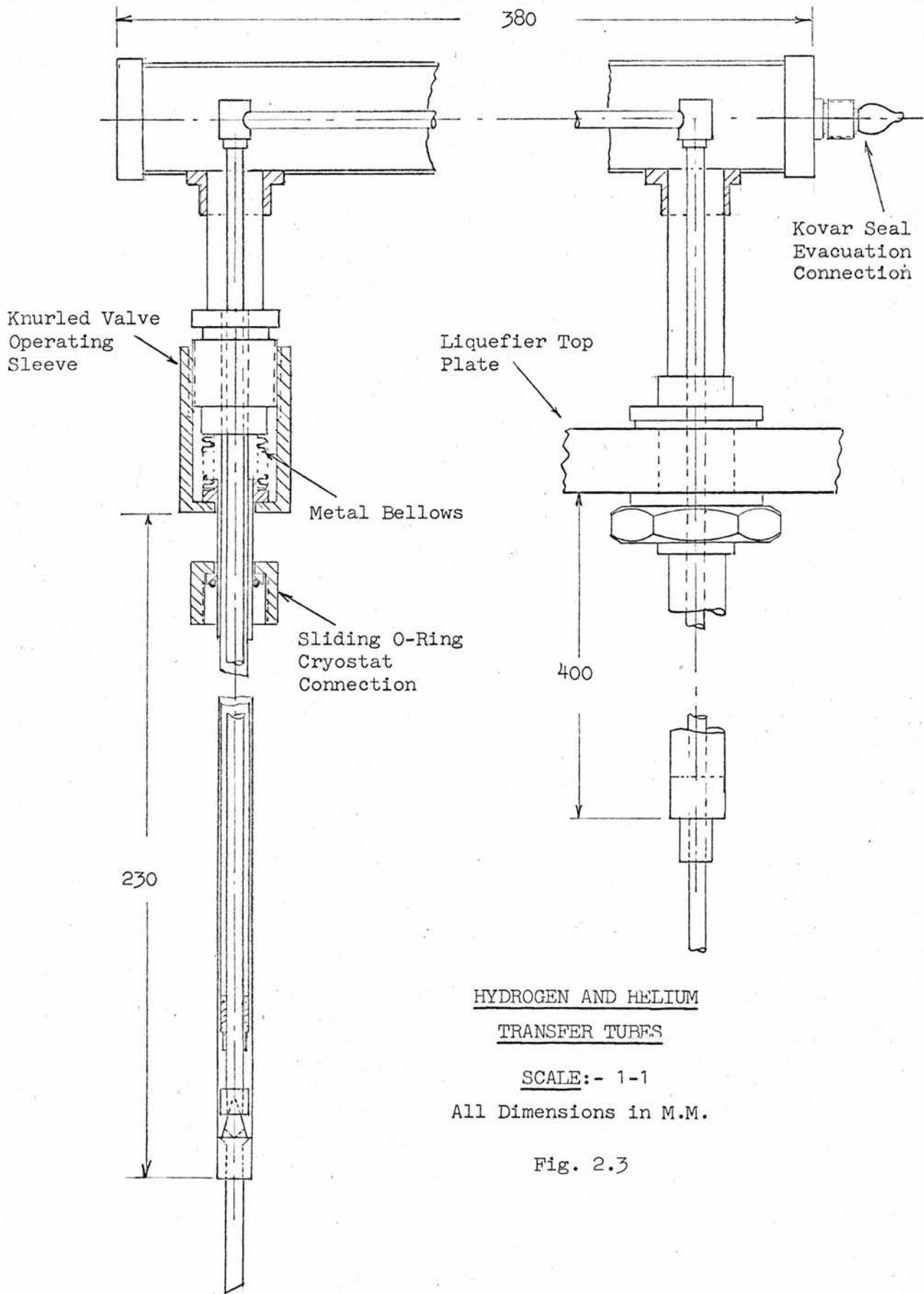
A certain amount of trouble was experienced at first with these seals but a procedure for reconnecting was finally devised which made them very reliable. The seals were first tightened carefully and evenly at room temperature, the outer case evacuated and the radiation shield filled with liquid N_2 . After the N_2 had

evaporated and the seals returned to room temperature the outer case was removed. The nuts could now be tightened a further eighth to quarter turn before going solid. Treated in this way the joints proved very satisfactory, remaining tight for long periods without any further attention.

2.5 Control Valves

The Joule-Thomson expansion valves for both H₂ and He were identical. Both needle and seat were constructed from copper nickel with a 40° taper on the needle. Fine control of pressure was achieved by screwing the 1/4" diameter needle body sixty threads per inch. While the screw thread was at the cold end of the assembly this never gave rise to any trouble due either to freezing or wear caused by the absence of lubricant. A rubber O-ring seal at the room temperature end of the tubular shaft provided the necessary sealing on the low pressure side of the valve. If it was necessary to remove the valve for any purpose the high pressure and low pressure tubes were unsoldered and the complete unit withdrawn through the top plate.

The transfer valves were very similar in construction to the Joule-Thomson valves using O-ring low pressure seals at room temperature and tubular shafts to the needles. The taper on the needle was increased to 60° and the needle body screwed 1/4"-20 T.P.I. to make for more rapid operation.



A much simpler form of construction was used in the bleed valves. Here, a quarter inch diameter stainless steel cylinder one inch long with a 45° included angle taper machined on one end was attached to a Bowden cable and pushed into contact with a brass seat. Friction in the room temperature sliding O-ring seal was sufficient to hold the valve in the open or shut position. Adequate sealing was achieved by this method, no major leakage ever being evident.

2.6 Liquid Helium and Hydrogen Transfer Tubes Figure 3

The vacuum jacketed transfer tubes were identical, the construction following closely the system originally devised by Kapitza (1934). The sliding outer tube carrying the male needle part of the control valve was sealed by a metal bellows and the valve seat soldered to the 4 mm O.D. thin walled german silver tube carrying the liquid. This valve assembly was at the exit end of the tube and remained at room temperature except during liquid transfers.

The vacuum jacket was carried down through the top plate into the liquefier a distance of 40 cms. A separate N_2 cooled radiation shield was not considered necessary in these tubes since the transfers occupied such a short time, about one minute/litre transferred; and we found in practice that very economical transfers were achieved.

2.7 Pressure Gauges and Gas Thermometers

High pressure H_2 and He pressures were measured immediately before the Joule-Thomson valves and low pressures directly on the cans using ordinary bourdon tube gauges connected by capillary tubes sufficiently narrow to prevent rapid pressure fluctuations from being transmitted to the gauge mechanism. The reduced pressure N_2 and H_2 gauges were of the barometrically compensated type so that pumping speeds could be set to the same reading during every run. The N_2 gauge had a range of 0-760 mm Hg and the H_2 gauge a range of 0-100 mm Hg, both being provided with shut off valves to avoid damage should it have been necessary to test a circuit by means of an overpressure.

Gas thermometer bulbs were fitted to the bottoms of the H_2 and He cans both to enable cooling rates to be measured and to know when liquefaction was taking place.

2.8 Liquid Level Indicators

Monitoring of liquid levels in liquefiers is frequently a problem since variations in pressure due to liquid transfer, changes in Joule-Thomson settings or pumping speed cause wide fluctuations and instability in any kind of gauge dependent on pressure of the liquid head. Furthermore indicators of this type are rather insensitive when used for low density liquids, especially helium where liquid and vapour densities are comparable.

In an attempt to provide a uniform system of level indication for all liquids, concentric tube capacitors were built into 20 mm diameter pipes fitted to the side of each can with 2 mm bore connector tubes at top and bottom. These small bore tubes effectively prevented any surging of the liquid within the capacitor. Any change in liquid level caused a change in the capacity of the capacitor which produced a variation in anode current in a crystal oscillator. The inner german silver tube of the capacitor was 6 mm O.D. x 0.2 mm wall and the outer 8 mm O.D. x 0.2 mm wall.

For all systems except helium this system worked very well indeed and was completely reliable but the extremely small dielectric constant for helium of 1.05 meant that with an empty capacitor capacity of 100pF the capacity when full was only 105 pF. This very small change in capacity made the simple circuit unstable and inaccurate.

In 1963 a commercial capacitance level indicator built by Deber Kontrol, Stockholm, became available in this country and the home made circuit, for the He indicator only, was replaced with this instrument. This proved to be entirely satisfactory and gave results reproducible from day to day without need for adjustment.

2.9 Compressors

The first H₂ compressor used on this liquefier was a Reavell

Type SAT4 with a rated throughput of $10.1 \text{ m}^3/\text{hr}$ at 120 atmospheres. While reasonably reliable from the mechanical point of view major modifications of the shaft sealing arrangements were required to prevent the entry of contaminating air.

A Broome and Wade air compressor with a throughput of $15.3 \text{ m}^3/\text{hr}$ at 25 atmospheres was modified in our workshops to make it suitable for helium. Unfortunately, as it was a poor quality machine in the first place, it gave constant trouble with porous castings and similar fundamental defects.

Early in 1961 these units were both replaced by identical three stage horizontal compressors with all stages on the same axis, built by Andreas Hofer, Mulheim, Germany, with a throughput of $36 \text{ m}^3/\text{hr}$ at 120 atmospheres. This very large throughput was not utilized completely, some of the gas being bled through a bypass circuit and in the case of the He through a pressure reducing valve to 25 atmospheres. It was hoped these machines would solve our compressor problem, as similar machines had been used with success elsewhere and were highly recommended. While one performed very well and gave no trouble the other had to be returned twice to the manufacturer for repair within a period of four years.

2.10 Vacuum Pumps

A pressure of 140 mm Hg was maintained in the reduced pressure N_2 can be means of a 120 l/min rotary vacuum pump; manual pressure

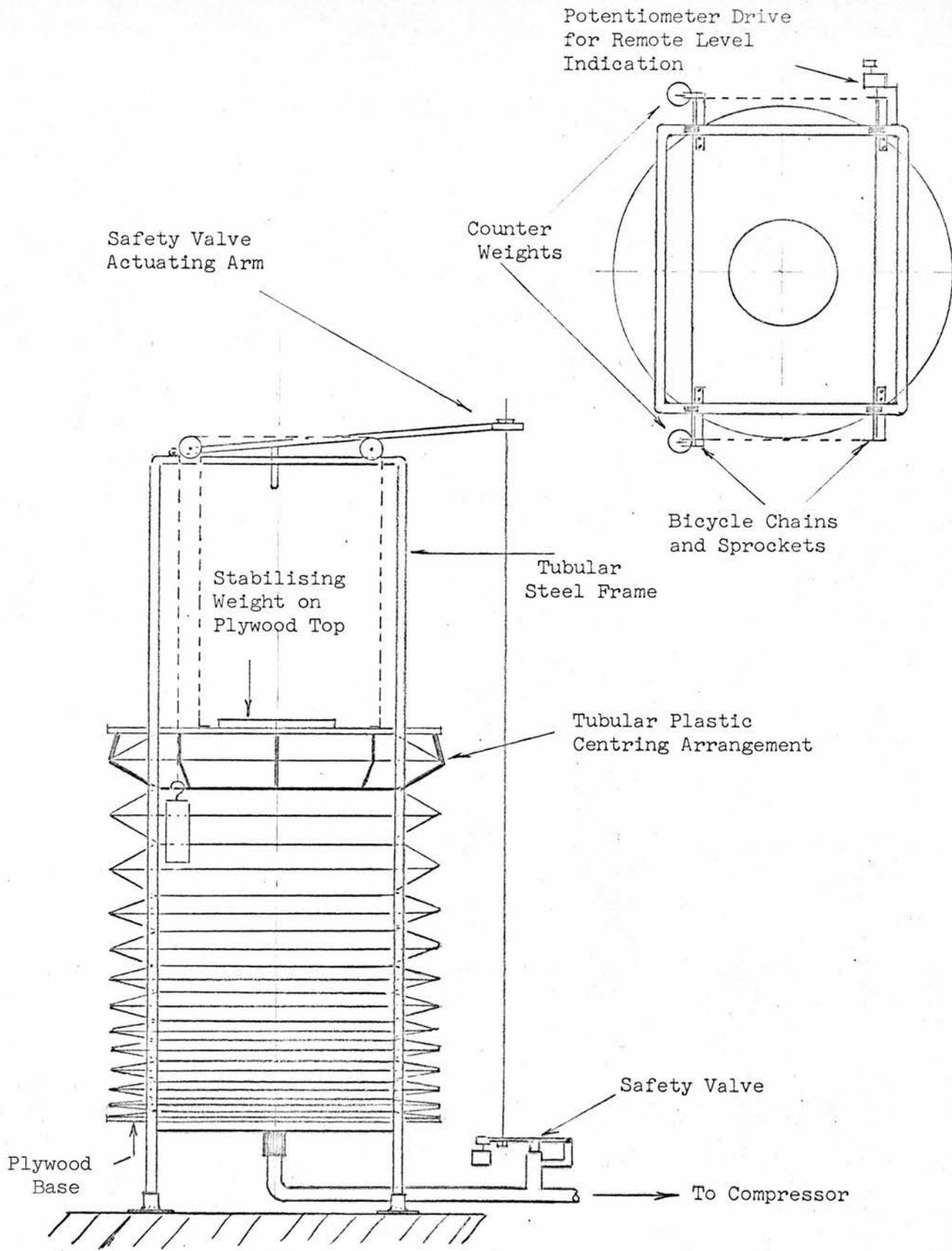


Fig. 2.4

BUTYL RUBBER GAS HOLDER

SCALE:- 17-1

control being by means of a 1" Saunders diaphragm valve. This pump was filled with tri-tolyl phosphate rather than rotary vacuum pump oil, to enable liquid air to be pumped as this was more readily available at St. Andrews. Also liquid nitrogen in storage tends to condense an appreciable fraction of oxygen, and, as liquid is evaporated from the can during the run, the remainder tends to become oxygen-rich, which is not a happy circumstance in close proximity to high pressure hydrogen.

The hydrogen vapour pump was an Edwards single stage rotary piston type with a pumping speed of 1,500 l/min which easily maintained the pressure at some 80 mm Hg, or 14.5°K. Again pressure control was by a 1" Saunders diaphragm valve and once set, with the liquefier in a steady state condition, required no further attention.

2.11 Gas Holders Figure 4

Due to the liquefier being in what had been the back parlour of a rather run-down Victorian residence, the load which the floor would safely stand was strictly limited. Since an oil filled gas-holder having a capacity 1 m^3 weighs about 270 kg, two such gas-holders being required for convenient operation of the system, a lighter type of reservoir had to be found. The solution was a large rubber bellows 1 m diameter and 2 m high, gas being drawn from the bottom and smooth movement of the top ensured by a system

of chains and counterweights. The increase in height was not strictly proportional to the change in volume since the weight of the lower convolutions made them rise non-uniformly. Calibration of volume against height was therefore necessary, as well as corrections for ambient temperature and pressure. A system of alarm bells and safety valves was required, as unlike an inverted bell rigid gasholder there was no inherent safety device; overpressure would result in the bellows assuming a spherical shape and ultimately bursting.

These gasholders were originally made from natural rubber but the rate of diffusion particularly by oxygen through this material was rather high and they were replaced by bellows made from butyl. Unfortunately no reliable gas diffusion figures were taken from the original gasholders but accurate measurements were taken on the later version. From these, it was established that while the total volume of gas remained effectively constant, the purity dropped from 99.72% to 98.94% in a period of 150 hours. While there was effectively no temperature or pressure gradient across the rubber membrane, the differences in gas concentration will provide a driving force for diffusion. Since diffusion rate is a function of the collision cross-section of the gas molecule and the diameter of He is very much smaller than O_2 or N_2 the measurements on butyl rubber require further explanation. However

while molecules of a larger diameter diffuse more slowly, gases of a higher boiling or critical temperature, particularly oxygen, dissolve better in the rubber (van Amerongen, 1964). A certain permeability in a given rubber may therefore be a combination of high diffusivity with low solubility, as with He or low diffusivity with high solubility as with O_2 .

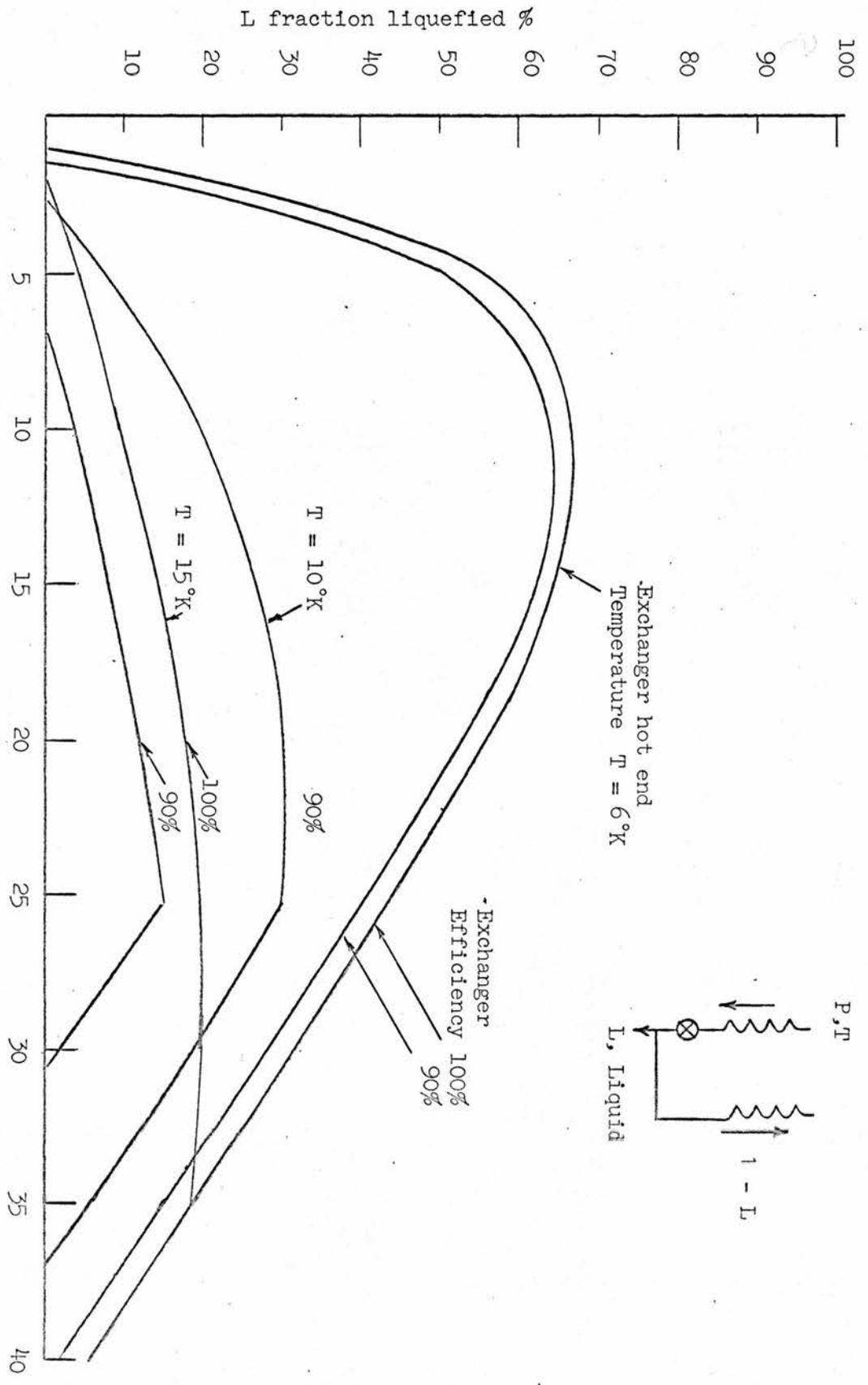
Taking these various points into account it was shown that for the material in question the permeability to O_2 was 1×10^{-10} cc at N.T.P./cm²/cm thickness/sec/cm Hg; the figure for He being very similar. This figure compared very favourably with experimental results while the rough measurements on natural rubber were in agreement with predicted performance (Izod, 1968).

2.12 Performance

The liquefier was in operation for almost exactly nine years and during that time liquid was made and withdrawn on 400 separate occasions, though a large number of these were concentrated into the last two years of use.

With the original bottom exchanger and He can, liquid H_2 was produced after seventy minutes and liquid He seventy minutes later. When the more efficient exchanger and smaller He can were fitted H_2 appeared after forty minutes and He after sixty minutes. These cooldown times could have been considerably reduced if the object had been merely to establish good cooling rates, but it was standard

Fraction He liquefied with J-T heat exchanger efficiencies 100% and 90% as a function of inlet temperature and pressure



J-T Pressure Atmospheres P
Fig. 2.5

practice to produce sufficient liquid H_2 to maintain He production at its maximum rate as soon as liquefaction had commenced.

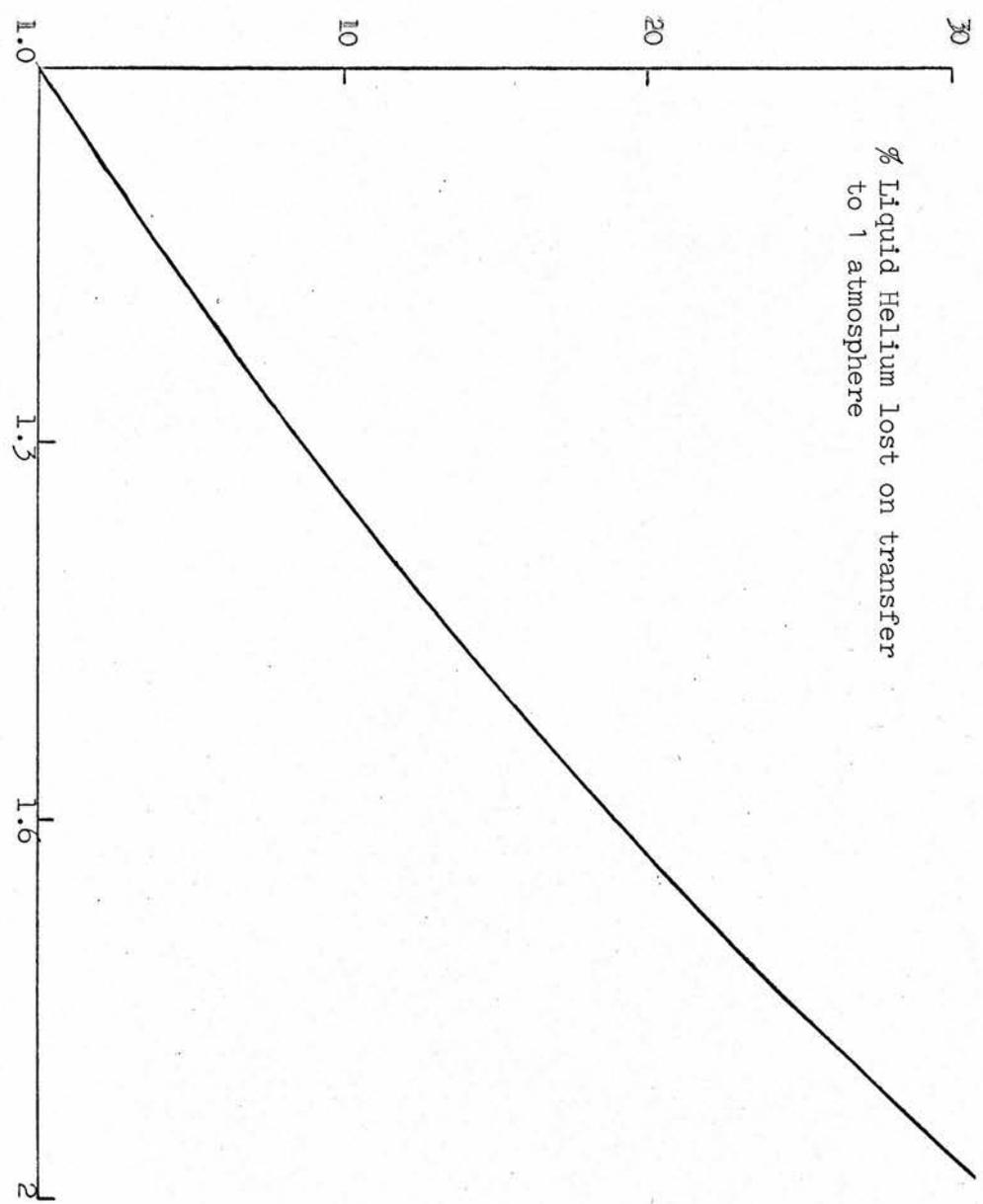
The normal production rate for He was about 3.6 litres/hr with a Joule-Thomson pressure of 25 atmospheres and with the previously quoted figure of $15.3 \text{ m}^3/\text{hr}$ throughput, this figure infers a fraction liquefied of 17.4%, which we considered to be a very satisfactory figure (see Figure 5).

However, later accurately measured throughput of the compressor was found to be $23 \text{ m}^3/\text{hr}$, so that the real fraction liquefied amounted to 11.6%. This figure was even further reduced in terms of useful liquid transferred; since the pressure in the He can was 1.4 atmospheres absolute, some 11.75% of liquid made was lost in transferring the liquid into the cryostat at atmospheric pressure.

Part of the unusually high pressure drop was accounted for by the flow of returning He being severely restricted at the point where the pipe diameter was reduced from 8 mm I.D. to 5 mm I.D. for the final 23 cms. There was no reason for this reduction in diameter other than ease of assembly and it is interesting to calculate the fraction liquid lost on transfer due to this section alone.

As previously stated the actual throughput of the compressor was $23 \text{ m}^3/\text{hr}$ and the maximum making rate 3.6 litres/hr or $2.66 \text{ m}^3/\text{hr}$,

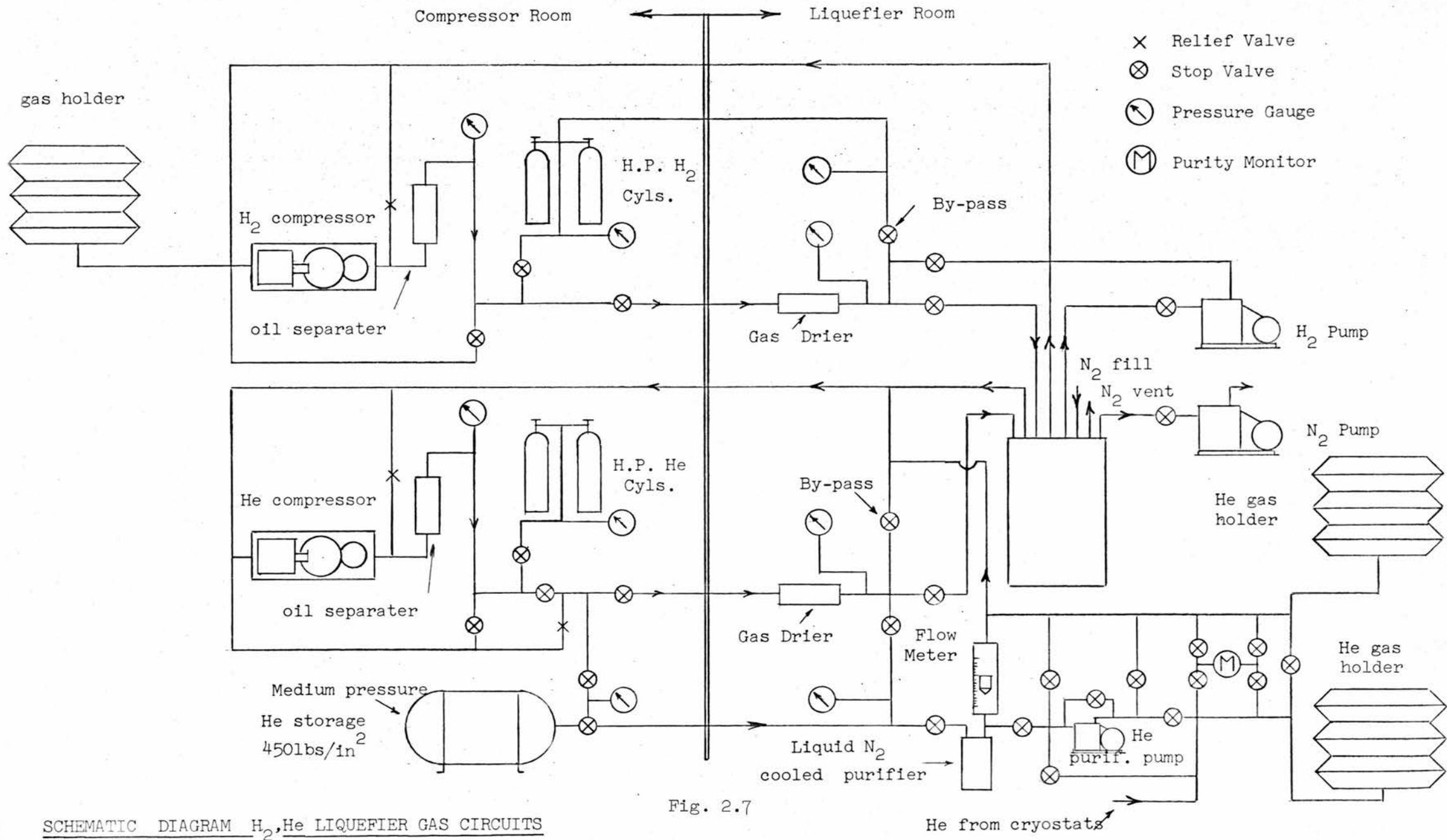
% Liquid Helium lost on transfer
to 1 atmosphere



Absolute pressure atmosphere

Fig. 2.6

Fig. 2.7



SCHMATIC DIAGRAM H₂, He LIQUEFIER GAS CIRCUITS

Fig. 2.7

FIG 7

so the actual volume of gas passing through this part of the exchanger was $20.34 \text{ m}^3/\text{hr}$. This added almost exactly 0.1 atmospheres to the pressure in the He can, sufficient to increase the fraction of liquid lost on transfer by nearly 2.9% (see Figure 6).

2.13 Liquefier Operating Procedure Figure 7

Operation of the liquefier was fairly complex and it is worth describing the procedure in some detail. The sequence of operations was as follows; being described as in an operation manual:-

1. Close valves on internal hydrogen (H_2) and helium (He) purifiers and stop pump. Start rotary pump and diffusion pump on outer case and open case evacuation valve after ten minutes. Pressurize liquid nitrogen (N_2) storage dewar.
2. Fill external He purifier dewar and cold trap on outer case diffusion pump with liquid N_2 .
3. Fill He gasholder through external purifier with gas from the medium pressure tank, and fill H_2 gasholder from high pressure (H.P.) cylinders.
4. Check that H_2 and He high pressure valves to the liquefier are shut and bypass valves fully open. Start compressors and build H_2 pressure up to 66 atmospheres, using bypass at liquefier. Increase He pressure at compressor to 100 atmospheres (while the operating pressure of the liquefier is 25 atmospheres the higher pressure keeps the gas flow through the oil separator down to a

reasonable rate thus ensuring efficient oil separation. Adjust bypass at liquefier to 25 atmospheres.

5. Open H_2 and He return line valves and with Joule-Thomson bleeds and transfer valves fully open circulate both gases through the system. Check transfer tube valves are opening and free from moisture, reduce gas circulations to a slow trickle.
6. Start pump on reduced pressure N_2 can, open valve on liquid N_2 storage dewar and after about ten minutes both radiation shield standpipe and N_2 can will be filled. The case vacuum should be reading 5×10^{-5} or better at this stage.
7. Increase gas circulations, H.P. H_2 to 3 atmospheres before Joule-Thomson and 1.75 atmospheres absolute in can, H.P. He to 4 atmospheres before Joule-Thomson and about 1.3 atmospheres absolute in the can. This process is continued until the H_2 is below its inversion temperature a reading of 6-7 in. Hg on the H_2 can gas thermometer.
8. Close H_2 Joule-Thomson, close H_2 bleed, open H.P. H_2 valve to liquefier fully and adjust Joule-Thomson and liquefier bypass valve to maintain a pressure of 120 atmospheres before the Joule-Thomson and 2 atmospheres absolute in the can.
9. This part of the process cools the whole of the He circuit and is continued until the gas thermometer on the He can reads 5-6 in. Hg. Reduce He circulation to a trickle and when H_2 gas thermo-

meter reads 14-15 in Hg close reduced pressure H_2 can transfer valve increasing can pressure to about 2.6 atmospheres absolute.

10. Liquid H_2 starts to collect at this stage and while making liquid H_2 fill He gasholder once more and refill H_2 gasholder as required.

11. When there is sufficient liquid H_2 in the main can (about three quarters full) make sure the H_2 gasholder is nearly empty and fill the reduced pressure (R.P.) H_2 can through the liquid transfer valve.

12. Shut reduced pressure H_2 can pumping valve, start H_2 pump and open pumping valve gently adjusting pressure to about 80 mm Hg. Open He Joule Thomson gently to avoid loading the H_2 suddenly and increasing the H_2 can pressure to a dangerous level. Continue opening Joule-Thomson until He can pressure about 1.15-1.2 atmospheres absolute and keep this setting until He is below its inversion temperature a reading of about 10 in Hg on the gas thermometer.

13. Close He Joule-Thomson and bleed, re-open Joule-Thomson and adjust L.P. to 1.4 atmospheres. The H.P. has to be watched carefully for the next ten minutes or so, as the falling temperature causes thermal distortion in the Joule-Thomson assembly resulting in violent pressure fluctuations.

14. When the machine stabilises and liquid He begins to collect

replenish N_2 , R.P. N_2 R.P. H_2 cans and He gasholders as required watching all pressure gauges and level indicators carefully for the duration of the run. It is particularly important to keep the H_2 and He can pressures constant as there are no safety valves to provide protection against over-pressures, and insufficient pressure and hence flow can result in freezing of the reduced pressure liquids. This makes for very inefficient heat transfer but it can also result in explosive boiling occurring when the solidified gas melts. Check making rate of liquefier keeping gas-holder stationary with gas brought from the medium pressure tank through a flowmeter, the flow rate giving a direct measure of liquefaction rate.

Filling He Cryostats at the Liquefier

Cryostats are brought to the liquefier room having been cooled to liquid N_2 temperature and filled to atmospheric pressure with clean dry He gas.

1. Clamp cryostat to vertical sliding stand by liquefier, remove plug from transfer tube inlet on cryostat cap, insert transfer tube and carefully raise. Seal transfer tube to cryostat cap with O-ring coupling provided.
2. Remove plug from return line connection on cryostat and connect to He return line using rubber tubing. (N.B. make sure this is rubber, plastic tends to crack by thermal shock when it is cooled

- by returning gas.) Pump out He gas to gasholder, close return line valve and check cryostat for gross leaks.
3. Open liquid transfer valve slowly allowing cryostat pressure to rise to atmospheric. Open return line and continue transferring keeping over-pressure in cryostat to 4 cm Hg, as level appears in bottom of dewar this pressure will suddenly drop and the transfer rate can be increased without going above 4 cm Hg.
 4. When cryostat is full close transfer valve and warm up rubber return line connection.
 5. Remove O-ring seal at transfer tube, remove rubber return line connection and simultaneously close return line valve. Lower cryostat clear of transfer tube and return it to the experimental laboratory.

Liquefier Shut Down Procedure

1. Close R.P. N_2 pump line valve, stop pump and allow air into line up to valve. (N.B. do not under any circumstances allow air from the atmosphere past the pump line valve as moisture from the air will block the exchanger and the resulting pressure rise may be uncontrollable.) Close R.P. H_2 pump line, stop pump and equalise pressure across inlet and exhaust side. Release pressure in liquid N_2 storage dewar.
2. Shut H.P. H_2 valve to liquefier and open liquefier bypass valve to maintain a pressure of 65 atmospheres. Plug radiation

shield vent on top plate and open transfer line to N_2 storage dewar.

3. Open He Joule-Thomson and liquefier bypass to obtain an H.P. of about 6 atmospheres and an L.P. of 1.75 atmospheres.

4. Connect heavy copper tube coil to He transfer tube using "Cryostat Filling" procedure except that return line and coil are filled with He from the gasholder before opening transfer tube valve.

5. Open transfer tube valve and allow any liquid remaining in the liquefier to evaporate in the coil and return to the gasholder. Use spare compressor capacity to store the He returning to the gasholder in the medium pressure tank, this is done by manipulating the liquefier bypass control to keep the flow through the liquefier constant, thus warming the whole liquefier.

5. When the liquefaction process is stopped the R.P. H_2 freezes due to there being no heat load. At this stage the gauge has to be watched carefully and the pumping valve opened when the pressure rises to atmospheric. All evaporated H_2 returns to the H_2 gas-holder and is stored in H.P. cylinders.

6. When all the liquid H_2 has gone from the liquefier and the R.P. N_2 can has reached a positive pressure the transfer valve between this can and the radiation shield is opened.

7. At this point all the gas from the liquefier will have returned

to the gasholders and they should be pumped as flat as possible before stopping both compressors.

8. Shut case evacuation valve, switch off diffusion pump, after ten minutes switch off backing pump.

2.14 Conclusion

It is obvious from the foregoing that while the liquefier had a remarkable record, in terms of cryostats filled, during its years of operation there were very definite drawbacks. Operation was a highly skilled task requiring constant attention and concentration both to make liquid and prevent dangerous overpressures building up for which no safety devices were provided. Skilled manpower was in short supply and should not in any case be used for providing the basic "tool" for use in a low temperature research laboratory. It was time to look again at the whole policy of producing refrigerants and decide how best to maintain supplies now that commercially produced liquefiers were available.

CHAPTER 3

3.1 Provision of Liquefied Gases in New Physics Building

In planning the new and very much larger physics (teaching and research) building we were faced with the problem of how best to cope with the increased demand for liquid gases.

The Philips PLN106, six litre/hour nitrogen liquefier, which had replaced our Philips air liquefier three years previously, was clearly inadequate in the long term. It could keep pace with estimated demand for a further three to four years provided liquid nitrogen was used only for cryostats and not for precooling a helium liquefier.

As far as liquid helium was concerned there was no hope of moving the old He-H₂ liquefier to the new building and getting it running again in a reasonable time. Demand had already far outstripped its 3.6 l/hr helium capacity regardless of any modification we might carry out to improve its performance.

From the above it might appear that the most satisfactory and economical way to provide liquids in the quantities required would have been to buy liquid helium in bulk, and buy a large storage tank for tanker delivery of nitrogen when the PLN106 became inadequate. There were however other points to consider.

The nearest source of commercially produced liquid helium was London, 460 miles from St. Andrews and under British Rail's

policy it seemed probable that the five mile single track line linking St. Andrews to the main London-Aberdeen line would be closed. Previous experience had shown that the fragile vacuum insulated containers would have to travel by passenger train rather than by road transport. As there would be no connecting line to St. Andrews the department would require to provide its own transport from the nearest main line station. While the containers would probably have survived a short journey by road, at the hands of British Rail large evaporation losses would have been the result of any delay in delivery. Though the transport problems were not insuperable they were very formidable. Also, the type of experiment envisaged and hence the quantities of liquid used had to be considered.

Prior to the move to the new building the main research groups were:

PROPERTIES OF SUPERFLUID HELIUM
SEMICONDUCTORS AND SEMIMETALS
NUCLEAR MAGNETIC RESONANCE
LASER PHYSICS
X-RAY DIFFRACTION

Of these only lasers and x-ray diffraction had no immediate need for liquid helium. All the users of liquid helium were going to increase their demands considerably and it was likely that work on superconductivity and possibly low temperature calorimetry

would begin.

Taking the liquid requirements of these existing groups it became apparent that considering only experiments for which plans were advanced and equipment scheduled, our total annual requirements for liquid helium would rise to about 2,500 litres by 1967. This quantity of liquid, if purchased from a commercial source, would have had to be transported in nitrogen shielded vessels of 15 litres capacity amounting to 167 vessels per annum, an average of more than three per week. Liquid helium could have been obtained in vessels very much larger than 15 litres, but with a full nitrogen shield a 15 litre vessel weighed 126 lb, as much as could be comfortably lifted by two men. While lifting equipment for larger vessels, say, of 50 litres, could easily have been provided in the laboratories, there was no chance of such equipment being provided at a country railway station.

To provide the service envisaged we would have required a total of at least six 15 litre vessels, an addition of four to our existing stock.

In the quantities of liquid helium under consideration a gas recovery rate of 95% reduced the liquid cost by nearly a third, but this would have meant returning 316 standard 200 ft³ cylinders per year to London, an average of just over six per week.

It was obvious that to contemplate this source of supply we

would require the use of departmental transport. While the capital cost of the vehicle could probably have been spread over the department as a whole it would be required for not less than two half days per week for cryogenic transport purposes. It was also likely that when the rail link to St. Andrews was closed the nearest station coping with our sort of traffic would be Cupar, ten miles away, so the van would have had to run 40-50 miles per week.

3.2 Bulk Purchase of Liquid Helium

Taking all these points into consideration and with a predicted annual consumption by 1967 of 2,500 litres the costs would be as follows.

1. 2,500 litres of liquid helium without gas recovery at £3/litre	£7,500
2. Liquid loss from 3 x 15 litre storage vessels at 2%/24 hrs 325 litres/annum	975
3. Transporting 166 vessels/annum at £7 return Cupar-London	1,160
4. Capital cost 4 x 15 litre helium storage vessels £640 amortized over 5 years ..	130
5. Maintenance of storage vessels, three overhauled/annum at £95	285
6. Quarter of one technician's time driving storage vessels to and from station topping up liquid nitrogen shields etc	250
7. Cost of running department vehicle	105
	<hr/>
<u>Cost per litre of liquid used</u>	£4-3

In spite of transport costs there was a considerable saving if the helium gas was recovered.

1. 2,500 litres of liquid helium with 95% gas recovered and returned 42/6d/litre	£5,310
2. Liquid loss from 3 x 15 litre storage vessels 2%/24 hrs 325 litres/annum	690
3. Total of items 3, 4 and 5 from above ..	1,575
4. Capital cost of helium recovery compressor fittings and valves amortized over ten years	50
5. Lubricants spares etc	30
6. Three quarters of one technician's time driving storage vessels and H.P. helium cylinders to and from station, running and maintaining recovery compressor etc	750
7. Power consumed recompressing helium gas 12 KW hrs/week at 1.1d/KW hr cost per annum	3
8. Cost of running department vehicle ..	105
9. Cost of transporting 326 H.P. helium cylinders/annum W.T. 123 lb/cylinder Cupar-London return at £40/ton goods	720
	<hr/>
	£9,233
<u>Cost per litre of liquid used</u>	£3-15

In spite of the extra labour and transport charges involved it was more economical to buy liquid and return the gas than let the gas escape to the atmosphere, though to maintain this advantage a high gas recovery rate was called for. In any case transport charges were very high, amounting to more than 10% of the

total cost in one case and more than 20% in the other.

The above cost of between £9,000 and £10,000 per annum to provide liquid helium by bulk liquid purchase after only two years in the new building meant that local liquefaction had to be considered.

3.3 Local Production of Liquid Helium

As already stated, the He/H₂ liquefier in operation in 1965 had been built in the laboratory, so, in principle at least, this could have been done again on a larger scale. We were, however, very seriously under-staffed and had no technicians to spare to tackle such a large project.

Also construction of a liquefier in the laboratory did not make economic sense. Considering the main costs we have:

1. Labour cost of eight man years ..	£10,000
2. Compressors vacuum pumps materials etc	5,000
3. Two year interim supply of liquid helium	14,000
	<hr/>
	£29,000
	<hr/>

As it was neither possible nor economic to build a liquefier we had to list our requirements in a laboratory liquefier and examine commercially available machines for suitability.

TABLE OF COMMERCIALY PRODUCED

HELIUM LIQUEFIERS

Commercially Produced Helium Liquefiers

Manufacturer	Cost	Liquefaction Rate L/Hr	L.P. H.P. Pressure Atmos. Absolute	Quantity Liquid Transferred 9 Hr Day Litres	Start Time Minutes	Shutdown Time Minutes	Liquid N Consumption L/Hr	Power Consumption K/Watts	Delivery Months	Remarks
<u>Linde Type</u>										
Elliot Bros. Britain	£25,447	20	1.5	119	60	60	40	90	12	Note 1
T.B.T. France	£18,700	8	1.5	47	60	60	25	28	10	Note 2
<u>Expansion Engine Type</u>										
A. D. Little U.S.A. Type 2	£18,400	4-8	1.3	59	120	30	12	26	4	Note 3
A. D. Little U.S.A. Type 200	£24,844	4-10	1.3	200 Litres/ 24 Hrs	105	-	10	26	4	Note 4

Note 1 - No machine to this design had been constructed by Elliot.

Note 2 - The pumped hydrogen was not recovered in the T.B.T. design and it used 5 M³/Hr presenting an explosion hazard. It would probably have been possible to recover this hydrogen.

Note 3 - Liquid hydrogen could be produced with this machine by circulating hydrogen gas at a low pressure through a separate coil in the heat exchanger and collecting it in what was normally the helium container. It would also operate at a reduced rate without liquid nitrogen precooling.

Note 4 - This liquefier was fitted with an external Joule-Thomson expansion valve and liquefied into a 250 litre gas shielded helium vessel. It was fully automatic and would look after itself after the initial cooldown, operate at reduced output without liquid nitrogen and shut itself down in the event of a fault in the system.

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3.4 Principal Requirements of a Laboratory Helium Liquefier

1. Production capacity capable of coping with all foreseeable liquid demands.
2. Automatic or semi-automatic operation making minimum demands on technical manpower.
3. The ability to produce liquid helium with or without liquid nitrogen precooling.
4. Simple construction making for reliability and enabling maintenance to be carried out rapidly and with normal laboratory facilities.
5. The complete system to be supplied by one manufacturer including all valves, pipework etc.

Referring to the table of commercially produced helium liquefiers the only liquefier built in Britain was the Elliot and at that time none had been completed. If we had purchased this very large machine our unit would probably have been the first, with all the attendant teething and development troubles.

The T.B.T. (French) machine had been in full production for sometime but had a number of disadvantages. The most important of these, the fact that the pumped H_2 was not recovered could probably have been overcome, but the machine required constant supervision which was wasteful of manpower.

Both the above machines required liquid nitrogen precooling in order to work at all and both had high pressure (200 kg/cm^2) hydrogen circuits. While liquid hydrogen can be very useful in the low temperature laboratory and has been used in large quantities

in some laboratories, we were anxious to avoid high pressure hydrogen with its attendant dangers.

Both the A. D. Little machines were developments of the well known Collins (1947) design, using expansion engines to provide cooling. They could be run without liquid nitrogen precooling though only at a reduced production rate of 4 litres/hour.

Only high pressure (17 kg/cm^2) helium was used in the working cycle. If necessary, liquid hydrogen could be produced in this machine simply by passing hydrogen gas at atmospheric pressure through a separate coil in the heat exchanger. The liquid hydrogen was collected in what was normally the helium can so there was a certain danger of contaminating the helium supply.

In spite of being more complicated than the Linde type machines, A. D. Little units had a remarkable record of reliability and the most advanced of these, the type 200, was unique in laboratory liquefiers. It differed from its predecessors in having the Joule-Thomson expansion valve outside the liquefier casing; the liquid being made directly into a 250 litre storage vessel. This innovation meant that the internal capacity of the machine did not restrict the run time between liquid transfers. The steady state conditions in the machine were therefore maintained for long periods making for high stability during a run.

Coupled to the advantages of the external Joule-Thomson valve

were changes in the gas and electrical circuitry making for fully automatic operation. This was as follows: as soon as the machine was stabilized after initial cooldown and was liquefying into the external vessel, a control on the console was placed in the AUTOMATIC position. With the control in this position any malfunction in any part of the system resulted in a safe automatic shutdown of the liquefier, with consequent collection and storage of the helium gas. This arrangement made the liquefier capable of unattended overnight operation.

The A. D. Little type 200 was obviously the most suitable machine from the point of view of laboratory operation but the cost per litre liquefied compared with the cost per litre purchased was the most important factor.

3.5 List of Component Parts and Total Cost of Complete A. D. Little Type 200 Helium Liquefier and Gas Recovery System

- A. A. D. Little Collins Helium Liquefier Model 200 including:-
1. Liquefier cabinet with external Joule-Thomson valve.
 2. One 250 litre gas shielded liquid helium storage vessel.
 3. One liquid helium transfer tube for above.
 4. Two 4 stage 17 kg/cm^2 helium compressors, with motors and starters.
 5. One low pressure helium gasholder of 700 litres capacity.
 6. One liquid nitrogen-cooled medium pressure (250 lb/in^2) helium trap with automatic nitrogen filling control.
 7. One 50 litre nitrogen vessel to supply trap.
 8. Two 200 litre nitrogen dewars to supply liquefier during overnight operation.

9. Low and medium pressure tubing, valves, fittings, safety valves, spares etc.
- B. A. D. Little Automatic Gas Recovery System
1. One medium pressure, 250 lb/in², 600 S.C.F. helium recovery tank.
 2. One control box.
 3. Solenoid valves, regulators, relief valves, spares etc.
- C. A. D. Little Helium Repurifier System
1. One high pressure, 2,000 lb/in², helium repurifier cabinet.
 2. One 4 stage high pressure helium compressor complete with motor and starter.
 3. One helium purity monitor control.
 4. One helium gas recovery bag 100 ft³.
 5. Eight six-station helium cylinder manifolds with valves and pressure regulators.
 6. One control cabinet.
 7. High pressure tubing, valves, fittings, safety valves, bursting discs, spares etc.
- D. Helium Cylinders and Helium Gas
1. Forty high pressure steel helium cylinders 200 ft³ at 2,000 lb/in².
 2. Forty fillings of helium gas 8,000 ft³.
- E. 1. Installation check and initial start-up by A. D. Little engineer.
2. Crating, freight and insurance charges.

Total Cost £24,844

Cost per Litre Liquid Produced

Total cost of installation amortized over ten years, cost per annum	£2,485
Predicted annual consumption of liquid helium by 1967, 2,500 litres and evaporation from 250 litre storage vessel 2% or 5 litres/24 hrs, at 1,825 litres/annum and evaporation from 2 x 15 litre storage vessels at 2%/24 hrs, at 216 litres/annum.	
Total liquid production required 4,541 litres at 4 litres/hr, 1,135 hrs at 26 KW, 29,510 KW hrs at 1.1d/KW hr ..	135
Spare parts, lubricants etc	200
Replacement helium gas assuming 90% recovery of 4,541 litres liquid/annum	670
One technician	1,000
	<u>£4,490</u>
<u>Cost per litre liquid used</u>	£1-16

In spite of the high initial capital cost of the A. D. Little helium liquefier we could produce liquid locally for less than half the cost of that supplied from a commercial source. As well as this we had the added convenience of additional capacity immediately available when needed without negotiating further contracts with an outside supplier. We therefore decided to purchase the A. D. Little type 200 liquefier and purifier system.

3.6 Liquid Nitrogen Requirements and Methods of Supply

Though the A. D. Little helium liquefiers would operate without liquid nitrogen precooling, the production rate was only four

litres per hour against ten litres per hour with precooling.

Using liquid nitrogen the helium liquefier could be run for much shorter periods with consequent savings on wear and maintenance.

As previously stated, our Philips PLN106 nitrogen liquefier, producing six litres per hour was going to be inadequate in a very short time. In June 1965, to cope with the demands of cryostat cooling as well as precooling the old hydrogen/helium liquefier, the machine was running 24 hours per day six days per week. The hydrogen/helium liquefier required about 120 litres/week from a total production of between 850 and 900 litres/week. Changing over to the A.D. Little helium liquefier would release 120 litres/week for other purposes but the use of larger capacity cryostats would soon take up this slack, and more.

Calculating future liquid nitrogen demand on the basis of experiments for which equipment was scheduled, consumption was expected to rise to 2,000 litres/week by December 1968. Future requirements were rather more difficult to estimate but assuming low temperature research was restricted to the space originally allocated and allowing for the expected number of research spaces, consumption would rise to 3,000 litres/week by December 1969. We therefore had to cost commercial bulk N_2 purchase against laboratory production.

3.7 Purchase of Liquid Nitrogen from a Commercial Supplier

Most low temperature research laboratories consuming liquid nitrogen in quantity employ a large powder-insulated storage tank with liquid N₂ delivered by road tanker from a commercial supplier.

With our consumption of 3,000 litres/week, a 9,000 litre storage tank would have been required to allow a reasonable interval between deliveries, to minimize transport charges and to give some spare capacity for supplier's holiday periods, transport and industrial disruption.

By contracting to purchase all our liquid nitrogen requirements from British Oxygen Company for a period of five years such a tank would have been supplied free of charge. The liquid cost was calculated on the price per 1,000 ft³ of gas evolved and in our case was 27/- per 1,000 ft³.

Cost per Litre Liquid Nitrogen using Bulk Storage

With a liquid consumption of 3,000 litre/week or 72,300 ft ³ of gas, cost per annum ..	£5,075 4
In addition to liquid consumed we had an evaporation loss from the 9,000 litre tank of 1%/24 hrs 90 litres = 2,169 ft ³ at 27/- per 1,000 ft ³ , cost per annum	1,062 2
Quarter of one technician's time transferring liquid into smaller vessels, checking liquid levels etc	250 0
	<hr/>
	£6,387 6
<u>Cost per litre at 3,000 litres per week ..</u>	9.81d

3.8 Local Liquid Nitrogen Production

There were several nitrogen liquefiers available which were suitable for use in the laboratory, of which the most attractive was that made by the Philips company. This machine used a Philips-Stirling cycle (Kohler and Jonkers 1954) in which ambient air was condensed on the outside of the cold cylinder head. The working gas, hydrogen, was contained within the machine which operated at an average pressure of 25 atmospheres. A fractionating column removed water vapour, carbon dioxide and oxygen and provided 99.9% pure liquid nitrogen.

Unlike the Joule-Thomson and Claude process there was no need to compress the incoming air to 150-200 atmospheres with all the attendant problems of mechanical strength and powerful compressors. It was also a more efficient process.

We had been running a Philips PLN106, six litres per hour, for some years with very little trouble and we were very favourably disposed towards the larger PLN430 with an output of thirty litres per hour and a more modest 2,500 litre nitrogen storage tank housed in the building, close to the liquefier.

To make more efficient use of the liquefier, it was decided to store the liquid in the tank under pressure so that liquid could be drawn off at any time. As the Philips liquefier makes liquid at atmospheric pressure, the liquid was collected in a 15 litre powder

insulated vessel containing a submersible liquid nitrogen pump. A float switch in the 15 litre vessel controlled the pump; liquid being forced through a 3/8" diameter foam plastic insulated pipe to the storage tank in 10 litre batches. The pump was capable of raising the pressure of the liquid to 70 lb/in² with a non-return valve to prevent liquid bleeding back from the tank, in which pressure was maintained at 40 lb/in².

Cost per Litre Liquid Nitrogen employing the Philips System as Described

Philips type PLN430 nitrogen liquefier complete with motor switchgear etc	£9,000
2,500 litre nitrogen storage tank	2,500
Philips type PW7213 submersible pump	500
	<hr/>
	£12,000
	<hr/>
Total cost amortized over ten years	£1,200
Running cost at 3,000 litres/week 100 hours at 30 litres/hour 3,600 KW hrs at 1.1d/KW hr £16 10/- per week	858
Half of one technician's time maintaining equipment and transferring liquid	500
Cost of spares running 100 hrs/week at £10 per 1,000 hours	52
	<hr/>
	£2,610
 <u>Cost per litre at 3,000 litres per week</u>	 4.01d

Evaporation loss from tank at 1%/24 hrs 175 litres/week plus 700 litres/week giving a total of 5.21d per litre.

An alternative method of obtaining the above equipment was on a contract leasing basis.

3.9 Cost per Litre with Contract Leasing the Philips PLN430,
2,500 Litre Tank and Pump

Rental cost per year for first five years PLN430	£2,030
Rental cost per year for first five years 2,500 litre tank	560
Maintenance contract	625
Running cost for 3,000 litres/week ..	858
Quarter of one technician's time running equipment and transferring liquid into smaller vessels	250
	<hr/>
	£4,323
<u>Cost per litre at 3,000 litres per week</u>	6.65d

Evaporation loss from tank at 1%/24 hrs 175 litres/week plus 700 litres/week from smaller dewars 875 litres/week giving a total of 8.59d per litre.

Rental cost per year for following ten years PLN430	30
Rental cost per year for following ten years 2,500 litre tank	9
Maintenance contract	625
Running cost for 3,000 litres/week ..	858
Quarter of one technician's time running equipment and transferring liquid .	250
	<hr/>
	£1,772
<u>Cost per litre at 3,000 litres per week</u>	2.72d

Evaporation losses 875 litres/week giving a total cost of 3.51d per litre, therefore the average cost per litre is 6.05d.

The difference in cost per litre between buying the liquefier and contract leasing only amounted to 0.84d per litre but using 3,000 litres/week, buying the machine resulted in an average saving of £546 per annum.

3.10 Use of Liquid Nitrogen Precooling for the Helium Liquefier

Use of liquid nitrogen for precooling the A. D. Little helium liquefier increased the production rate from 4 litres/hr to 10 litres/hr. As a consequence, for a total production of 4,541 litres per annum the running time was reduced from 1,135 hours per annum to 454 hours per annum. The cost of power consumed thus fell from £135 5s. 1d. to £54 2s. 4d.

Running at 10 litres/hr the liquefier consumed 1 litre of liquid nitrogen for every litre of liquid helium produced. With locally produced liquid nitrogen costing 5.21d per litre this amounted to £98 11s. 6d. increase in running costs to be set against £81 2s. 9d. saving in power costs, a total increase of £17 8s. 9d. or 1.67d per litre consumed.

However the reduction in running time by some 60% made the use of liquid nitrogen precooling well worth while because of the saving of wear on the machine and reduction of demands on the technician in charge.

3.11

SUMMARY OF COSTS

LIQUID HELIUM

With an annual consumption of 2,500 litres:-

Purchasing liquid	(Cost per litre without	
from a commercial	(gas recovery	£4 3 0
supplier	(Cost per litre with gas	
	(recovery	3 15 0
Cost per litre using A. D. Little type 200		
helium liquefier with precooling		1 16 0

LIQUID NITROGEN

With a weekly consumption of 3,000 litres:-

Cost per litre purchasing liquid from B.O.C. .	9.81d.
Cost per litre renting the Philips PLN430	
system	6.05d.
Cost per litre purchasing the Philips PLN430	
system	5.21d.

Since we have had a good deal of experience in the liquefaction of gases, and since it was much more economical to liquefy locally, we decided to purchase both the A. D. Little 200 helium liquefying system and the Philips PLN430 nitrogen liquefying system.

CHAPTER 4

4.1 Design of Liquefaction Area

Having primarily for reason of economy but also for convenience decided that we should produce our own liquid helium and nitrogen, the next task was to house and arrange them in such a way that safe, efficient and economical liquid production could be ensured.

Since we were designing a new building on an open site for the sole purpose of teaching and research in physics, it was possible to plan the building from the ground up with no real limitation of space or restrictions other than the estimated size of the research effort in low temperature physics, which was aimed at about twenty users of liquid helium. We were thus in a singularly fortunate position, limited only by our own abilities and those of the architects and builders, and of course the overall cost of the building.

A requirement for the liquefaction of hydrogen is always a possibility in the development of a low temperature laboratory, leading to the accidental release of dangerous quantities of hydrogen gas. In view of this it was important to ensure that the area was as safe as possible and that the risk of explosion was minimised. Also, in the event of an explosion, the building design should be such that damage would be kept to a minimum and confined to the liquefaction

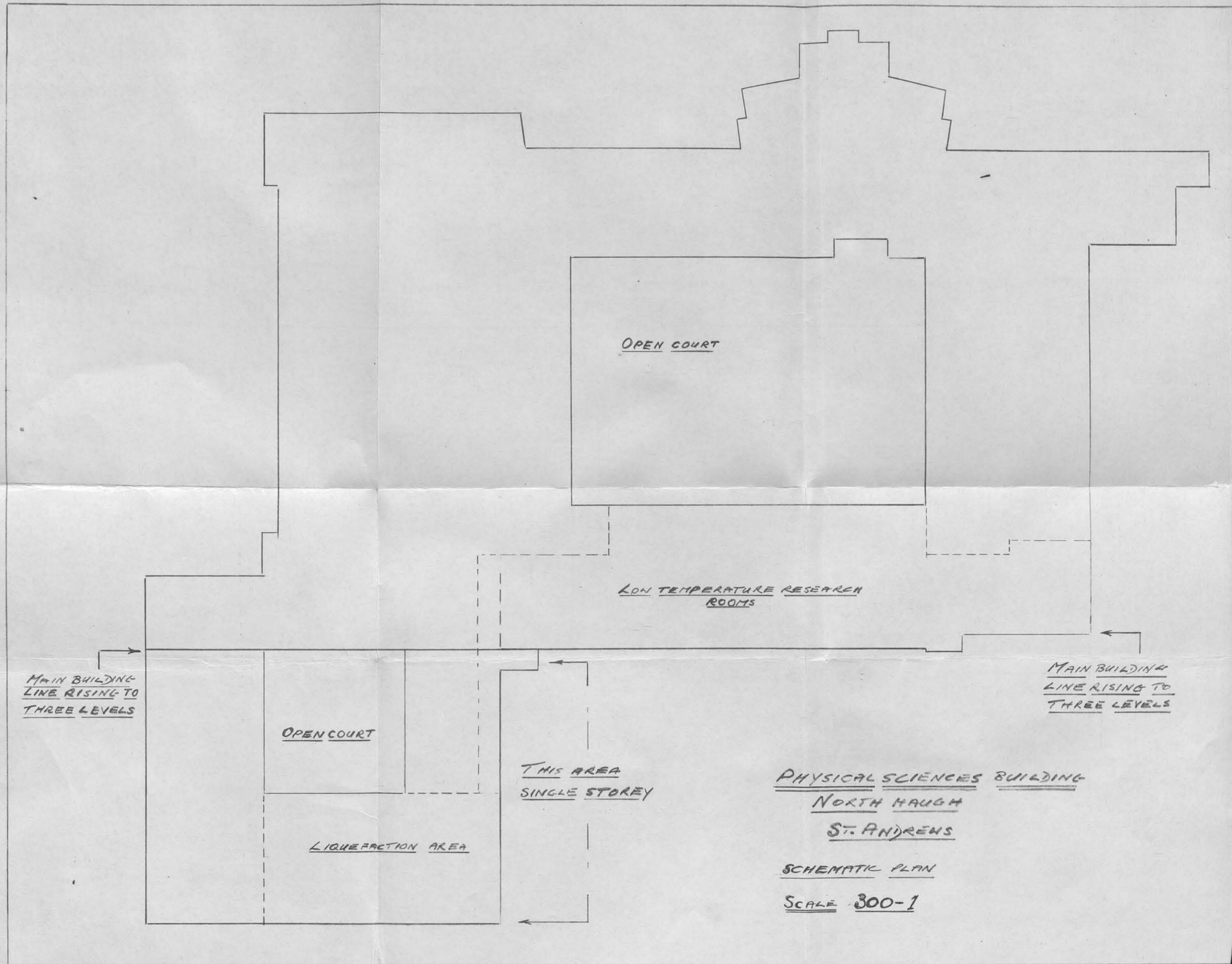
area.

4.2 General Layout

As will be seen from Figs. 1 and 2 the liquefaction area forms part of a hollow rectangle built as an extension of the main structure. Since the site was on a slope it was possible, without excavation, to plan the liquefaction area on two levels. The lower level, with a necessarily low ceiling, was designed to take heavy and largely unattended equipment, i.e., liquid nitrogen storage tank, helium gas cylinders, helium gas recovery bags, water pumps etc. Being on ground level, with direct access to the outside of the building, the handling of full and empty helium cylinders was thus made an easy task. A five ton travelling chain hoist was fitted above a wide double height entrance to permit off-loading of heavy equipment directly into the area on both levels. This entrance is wide enough to allow a lorry to reverse into the building under the hoist.

The upper floor forms the main cryogenic working area. This floor is at the same level as the whole of the research, workshop and stores area, so that all apparatus and equipment can move easily everywhere. Referring again to Fig. 2 it will be seen that this level houses the nitrogen liquefier together with the helium liquefier, 250 litre helium dewar, helium gas distribution panel and repurifier. The nitrogen liquefier and helium compressors are

Fig.4.1



OPEN COURT

LOW TEMPERATURE RESEARCH ROOMS

MAIN BUILDING LINE RISING TO THREE LEVELS

OPEN COURT

MAIN BUILDING LINE RISING TO THREE LEVELS

THIS AREA SINGLE STOREY

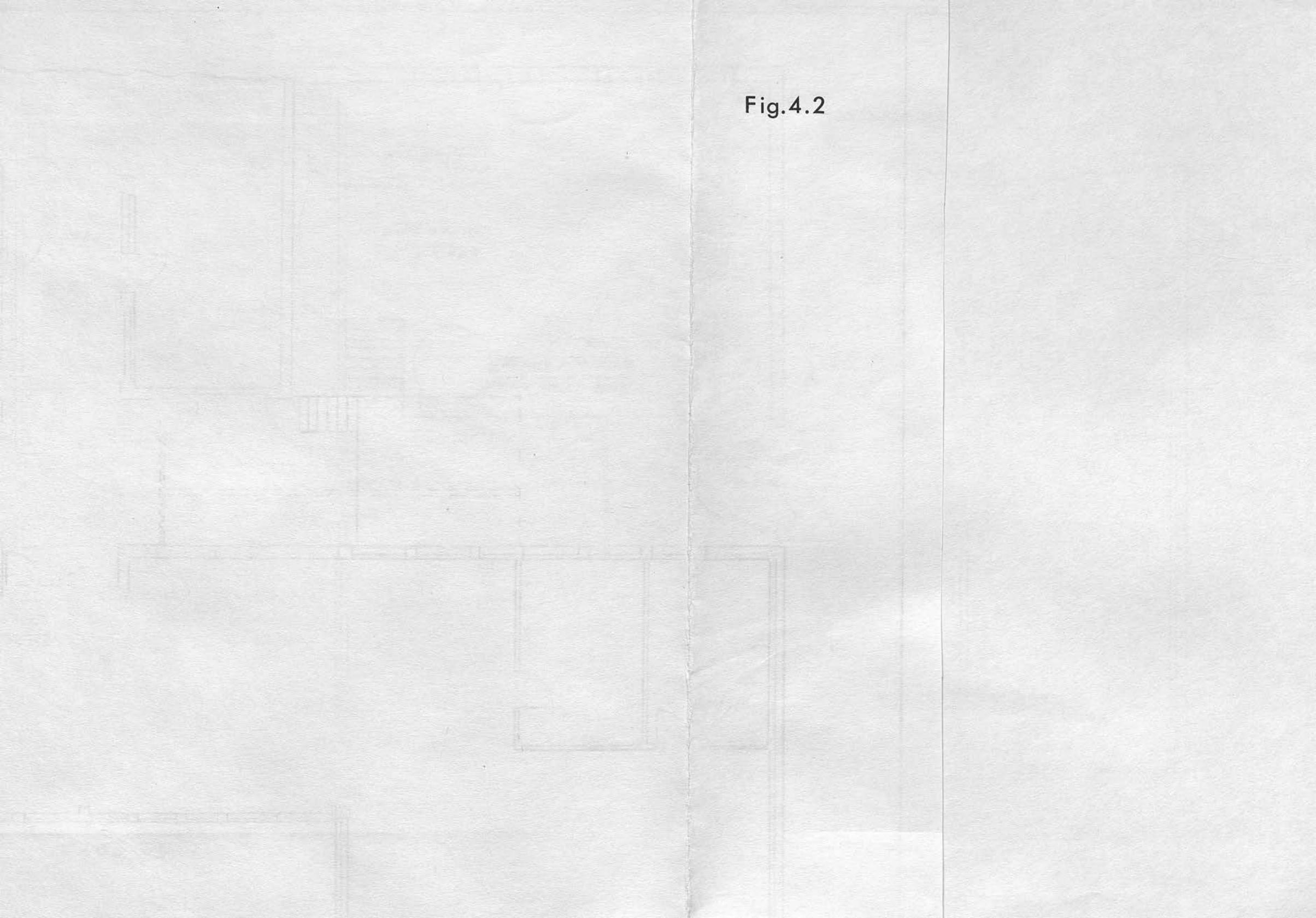
PHYSICAL SCIENCES BUILDING
NORTH HAUGH
ST. ANDREWS

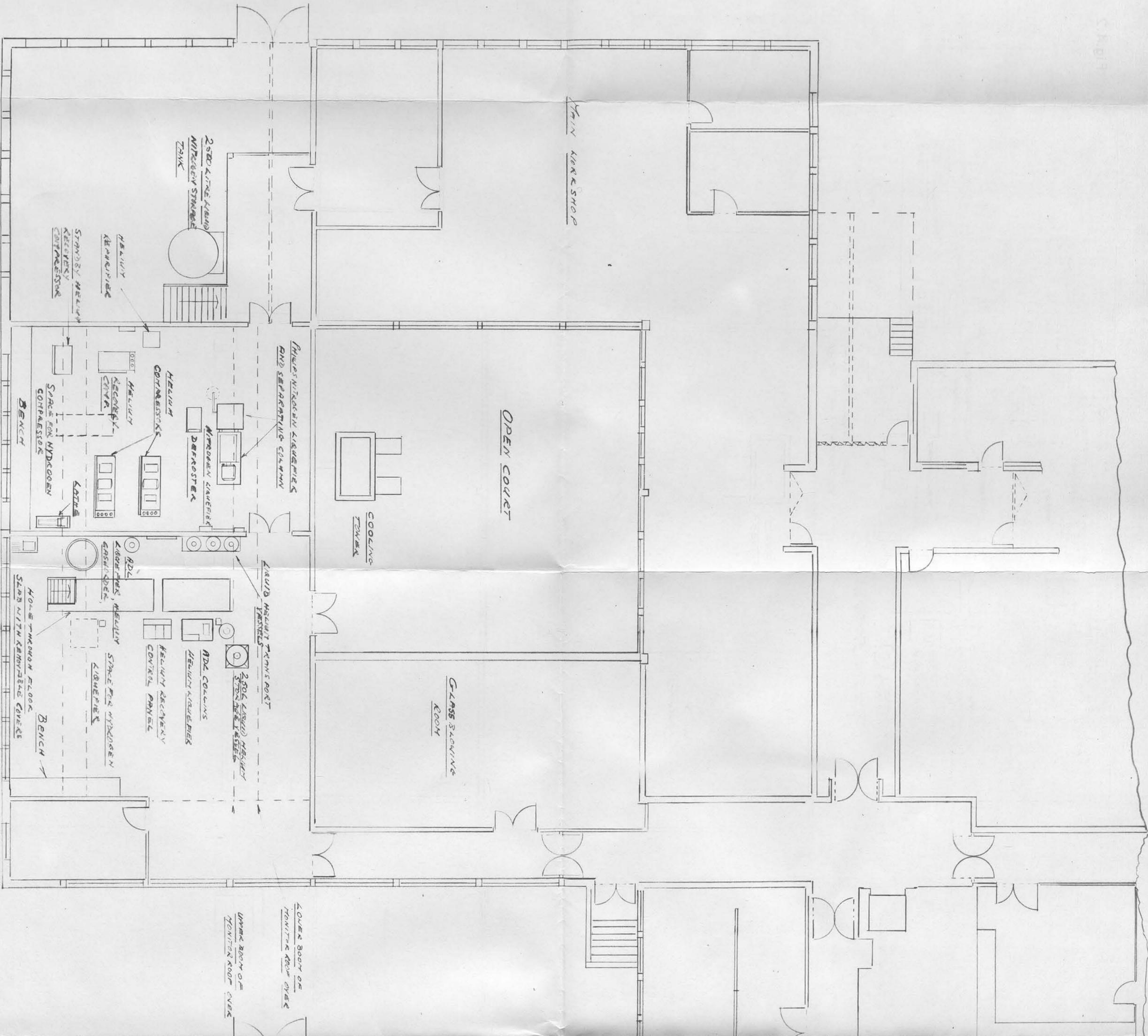
LIQUEFACTION AREA

SCHEMATIC PLAN

SCALE 300-1

Fig.4.2





C.M.D. 13

MAIN WORKSHOP

OPEN COURT

COOLING TOWER

GLASS SLOWING ROOM

2 STD LINE LUBR MTRAGEN STRAINER TANK

HELIUM RECOVERY

STANDBY HELIUM RECOVERY COMPRESSOR

HELIUM COMPRESSORS

SECURITY CHAMBER

SPACE FOR HYDROGEN COMPRESSOR

BENCH

APPROXIMATELY DEFROSTER

HELIUM COMPRESSORS

SECURITY CHAMBER

KOTLE

LIQUID HELIUM TANKS

200L LIQUID HELIUM STORAGE TANKS

ADL COLLINS HELIUM LIQUEFIER

HELIUM RECOVERY CONTROL PANEL

SPACE FOR HYDROGEN LIQUEFIER

LABORATORY GASLINES

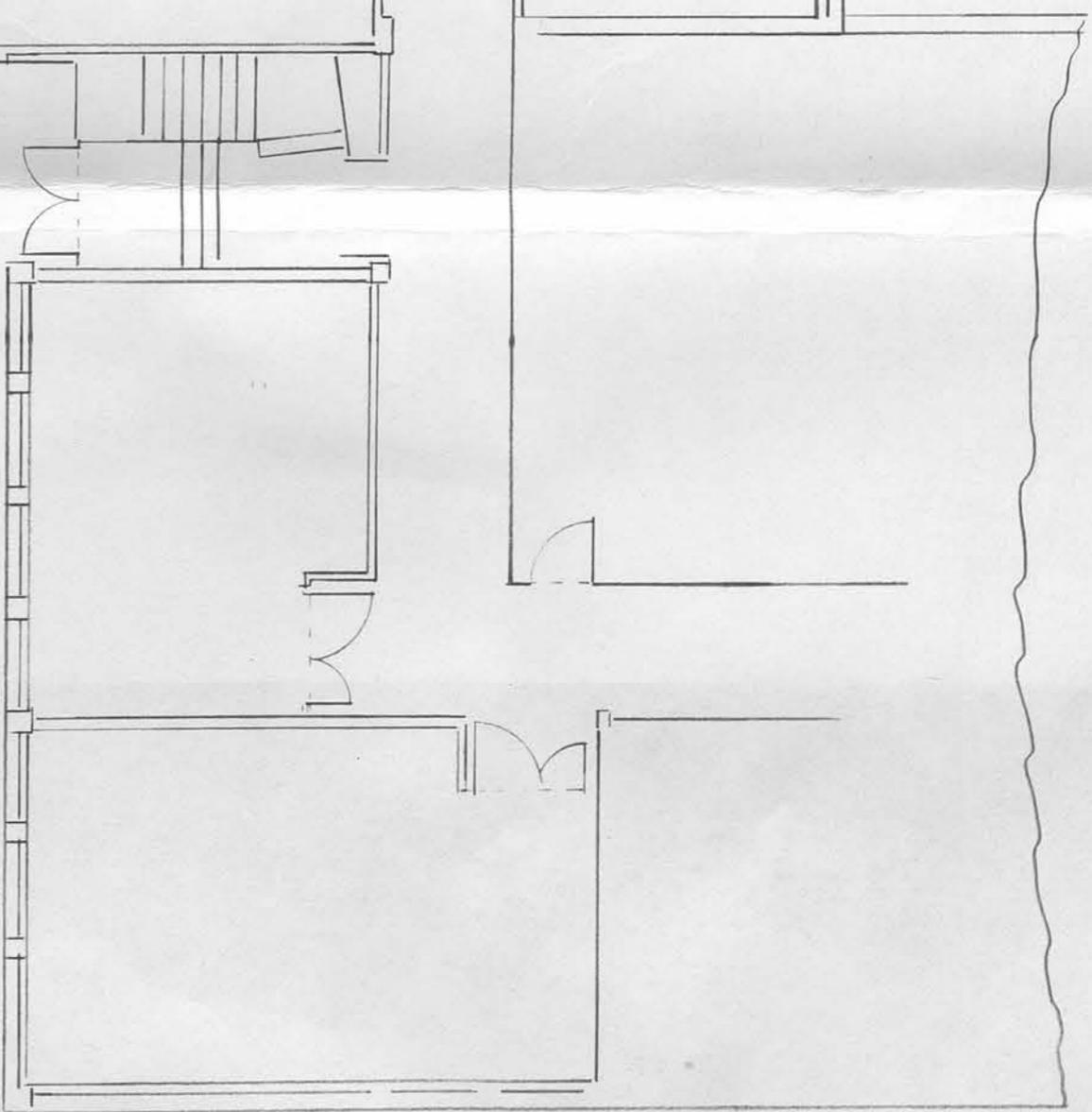
RDL

HOLE THROUGH FLOOR STAIR WITH REMOVABLE COVERS

BENCH

LOWER BOOM OF MONITOR ROOM OVER

UPPER ROOM OF MONITOR ROOM OVER



SPACE FOR HYDROGEN

CHIMNEYS WITH (PICKET)

SCREEN WALL



Photo 4.1 The liquefaction area showing part of the monitor roof, and to the left of the photograph the heavy equipment entrance

separated from the rest of the apparatus by a partition with a double glazed screen. This serves to reduce the noise level in the manned area and yet the liquefier operator can visually check the compressors.

Areas were left free on both sides of the partition to allow for possible future inclusion of a hydrogen liquefier and compressors. A further area was left clear on the liquefier side of the screen to house any experiment requiring very large quantities of liquid helium and therefore requiring to be filled directly from the 250 litre storage vessel. Furthermore, the A.D.L.-Collins machine can be used as a refrigerator for precooling large heavy items of apparatus prior to filling with liquid.

4.3 Building Construction (Photo 1)

As previously mentioned, the liquefaction area is extended from the main building. Thus, in the event of an explosion the damage would be confined to that area. This extension also houses part of the main workshop and glass blowing shop. An open area in the centre of the rectangle is utilised for the closed circuit water cooling tower which serves both helium and nitrogen liquefiers. (Photo 2)

The building construction took the form of a conventional steel framed building with concrete cast in situ around rolled steel joists. The suspended floor is a very strong structure of twelve inch thick concrete with high tensile steel reinforcing bars giving a maximum



Photo 4.2 The open area in the centre of the liquefaction extension showing the cooling tower

safe loading of 100 lbs/ft². All holes required for water cooling pipes, gas and compressed air lines, electrical cables, heating pipes etc., had to be marked on the original drawings and cast in position during construction. Drilling holes as afterthoughts in a floor of this type is a major undertaking. To allow for greater flexibility and unavoidable modifications to the liquefier pipework a rectangular hole 24' x 4' was left on the liquefier side of the partition. Wooden covers are provided and openings for pipes are easily cut where required.

As shown in Fig. 2 a monitor roof was provided over most of the liquefaction area which raised the major part of the ceiling to a height of 14' 3" and leaving a ceiling height of 10' around the sides. The sides and top of this monitor roof are very lightly constructed and would fly outwards in the event of an explosion, thus safeguarding the bulk of the liquefaction area as well as the main building. To obtain as much natural light as possible, two of the outer walls are glazed floor to ceiling and there are clerestorey lights around much of the monitor roof.

Vinyl tiles were used for the floor covering since they give an easily cleaned dust-free finish which is impervious to oil, and individual tiles are easily replaced in the event of damage. Acoustic tiles which cover the entire area of the ceiling help to reduce the general noise level.

4.4 Power Supply and Main Switch Panel for Liquefier Area

The power requirements of the new physics building are so large that we require our own substation where the incoming 11KV supply is reduced to 440V. Two transformers were installed, one for 'dirty' use for machine tools, compressors, pumps, and the boiler house, etc., one for 'clean' use in the teaching and research laboratories where a stable supply is essential for accurate measurements with delicate instruments. When it is remembered that the on load starting currents of heavy machines can rise to 90 amps per phase it will be realized that the reactions of the affected research worker may well be unpredictable.

Power is taken from the 'dirty' transformer to the main fuse board and the isolating switches for the liquefier area. This panel is located on the wall of the room adjacent to the liquefier room safe from any possible accumulation of hydrogen. It can also be reached in an emergency without entering the liquefier area.

4.5 Electrical Services

As previously stated there is always a strong possibility of liquid hydrogen being required in a low temperature laboratory and since the working gas in the Philips nitrogen liquefier is hydrogen there is always a chance of leakage from that source. We therefore decided to make the building as safe as possible for use with hydrogen gas.

The avoidance of electric sparks is of course essential and normally "flame proof" fittings are specified for use in atmospheres containing flammable gas. The principle in designing flame proof fittings is to produce an enclosure which is strong enough to withstand an internal explosion and at the same time prevent transmission of flame to the surrounding atmosphere. Normally such fittings are manufactured from heavy castings with broad flanges and narrow gaps. By this technique any products of combustion are cooled below the ignition temperature of the explosive mixture surrounding the fitting.

The clearances vary with the flammability of the gas in question and for the purpose of specifying clearances the gases have been divided into four groups. These groups, which are listed in British Standard Specification 229, are numbered one to four in order of increasing ease of ignition. A few gases including hydrogen are placed in group four and it has not so far been possible to establish clearances which will render flame proof enclosures safe for use with these gases.

As fittings are available for group three gases, which includes methane, these were considered for use in our building. While their use would have diminished the hazard it would not have eliminated it. It has also to be remembered that flame proof fittings are very expensive; taking a typical example, a standard direct on-line starter for a 2 H.P. 3 phase motor costs £3 15s., while the same item in a flame

proof pattern costs £21. It was therefore decided to approach the problem from a different viewpoint.

In any laboratory or industrial concern electrical equipment falls into two categories as far as operation is concerned;

- (a) items of electrical equipment whose operation is more of a reflex action than a deliberate movement,
- (b) those items where the action of the operator is premeditated and usually involves operation of other controls.

The only items which really come under the first category are lighting and small power circuits. By making the items in the first category completely safe and by careful location of the rest of the equipment the most efficient and economical solution can be provided. While it has already been stated that flame proof enclosures are not certified safe for use with group four gases there is a way of rendering safe these items in category (a).

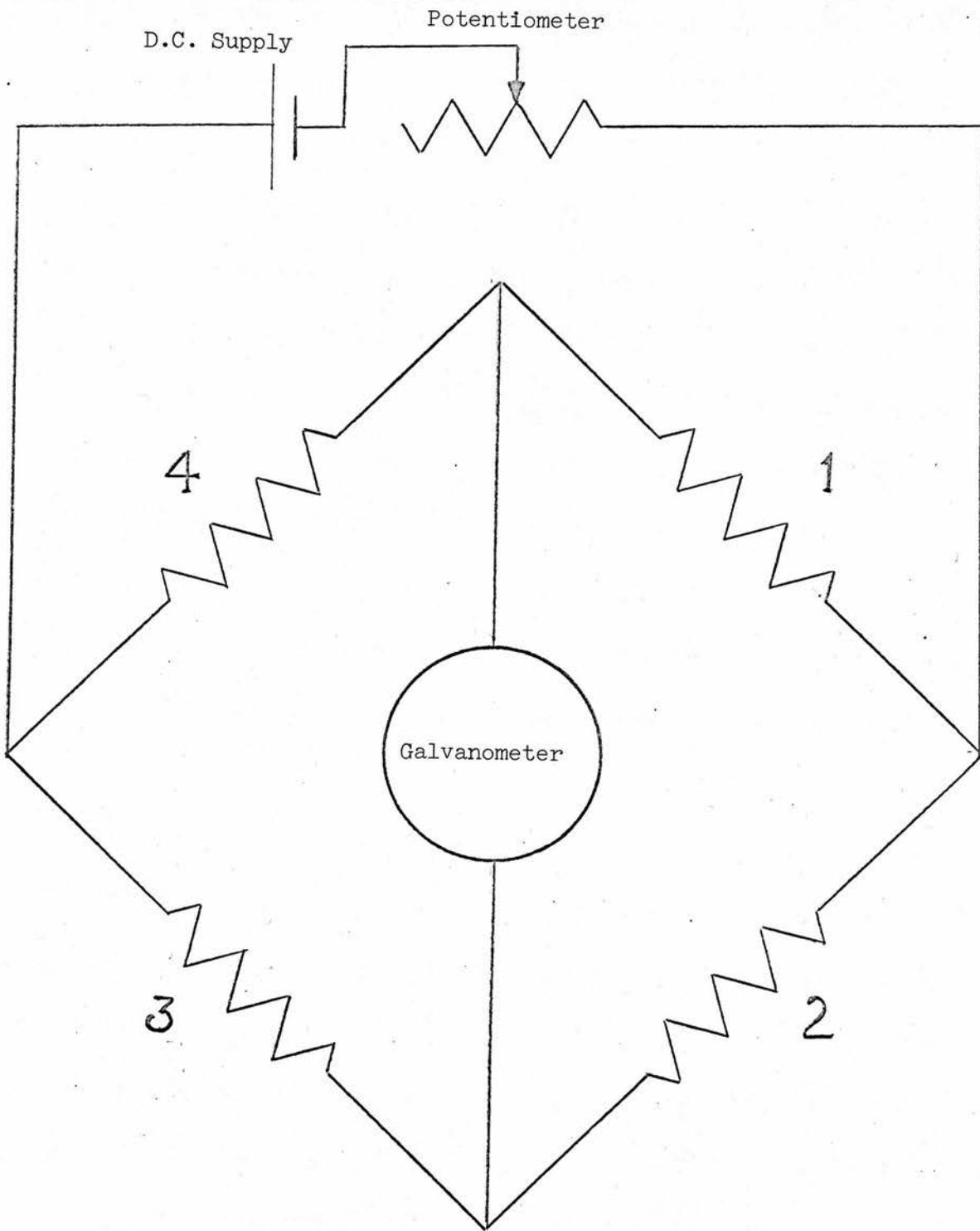
In the normal flame proof fitting it is assumed that the surrounding atmosphere has limited access to the interior of the fitting through the small gaps between the flanges. Since hermetic sealing of the fittings would have been very costly, not only in installation but maintenance, we decided to adopt another method. By sealing as well as possible the entire conduit system, light fittings and switches, by providing small overpressure of air in the conduits, we could ensure that the only possible leakage would be air escaping, and no hydrogen

could enter. The compressed air for this system is drawn from the main laboratory supply, and by a suitable series of valves reduced in pressure from 80 lbs/in² to $\frac{1}{2}$ lb/in². The lighting conduit circuit is divided up into separate segments each fitted with a sensitive pressure switch; these are placed in a safe area away from liquefiers. Should a circuit develop a serious drop in air pressure, the switch opens and automatically cuts the power from that circuit.

A pressurised conduit system is satisfactory for lighting, but socket outlets are obviously impossible to treat in this way and a different technique had to be devised to render them safe.

The method adopted was to wire throughout in mineral insulated cable, which is safe for use in any atmosphere, and fit sealed mercury switches in the socket outlet boxes. A locking pin actuated by the switch movement makes it impossible to insert or withdraw a plug with the power on.

The equipment in category (b) could be dealt with much more simply. As hydrogen is a low density gas it is most likely to collect in the higher part of a room. By keeping the main motor contactors and isolators at floor level, accidental ignition of hydrogen accumulation is greatly reduced. Remote control of the contactors is accomplished by sparkless switches and mineral insulated cables. In addition, starting or stopping compressors or pumps are not operations



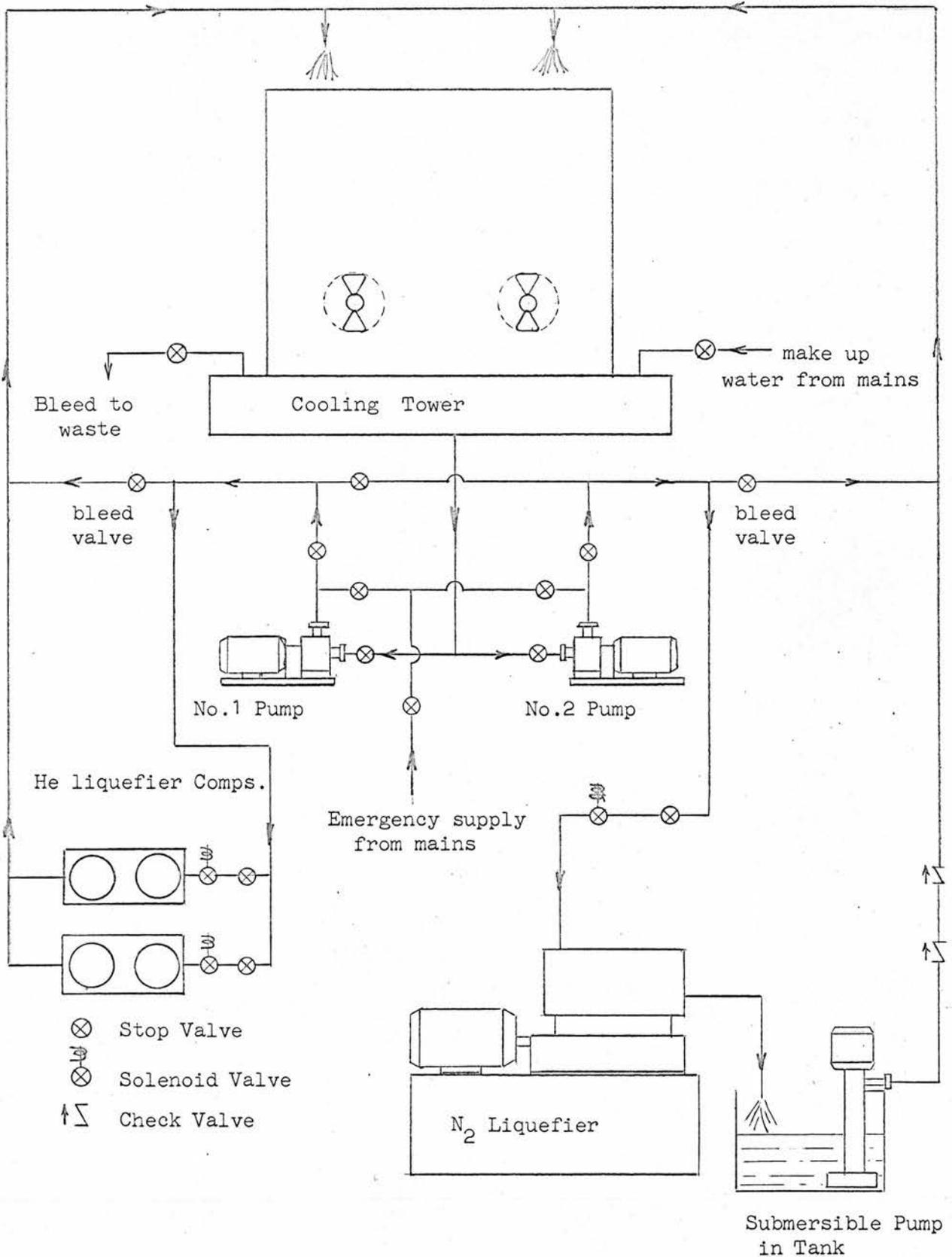
Katharometer

Fig. 4.3

which are carried out without some thought to the consequences and this in itself provides an additional safeguard.

4.6 Atmospheric Analysis and Extraction Control Equipment

We have seen in the previous section how it is not possible to render all electrical equipment safe for use in an atmosphere containing hydrogen. Sparking, however, is not confined to electrical equipment and static charges sufficiently large to cause sparking may be built up in clothing made from nylon and in a dry atmosphere merely combing the hair. Obviously the best way to minimise risk is to prevent hydrogen in the atmosphere reaching explosive concentrations, i.e. around 4%. Towards this end, we have installed a system of gas analysers which continuously monitor the air in the rooms likely to be used with hydrogen. Whenever the hydrogen content of the monitored air reaches a predetermined unacceptable level, extractor fans are automatically started. The detector units are manufactured by the Cambridge Instrument Company. These are in the form of katharometers, Fig. 3 which contain four platinum wires having the same electrical resistance and temperature coefficient. They are enclosed in separate cells within a metal block, each wire forming one arm of a Wheatstone bridge. A mains unit supplies a constant preset d.c. The Joule heating of the elements is balanced by heat loss to the gas in the cells, so that the temperature and thus the resistance of the element depends on the thermal conductivity



COOLING WATER CIRCUIT SCHEMATIC DIAGRAM

Fig. 4.4

concentration to an unacceptable level can easily be checked. Any sudden increase can be related to a particular incident and a repetition thus avoided. The recorder controller and the switchgear for the extract fan are situated in safe areas.

4.7 Liquefier Cooling Water

To help reduce the cost of liquid helium and nitrogen production, the compressors are cooled by recirculating the water through a cooling tower installed by Film Cooling Towers Ltd., Fig. 4 . Cold water is drawn from the base of the tower and driven through the compressor cooling stages by two pumps. The returning warm water is led to the top of the tower where it is distributed over an assembly of bars and baffles. These break the water up into films and drops which fall into the reservoir tank at the base. The tower is a twin cell unit capable of cooling 1480 gallons per hour from 30°C to 20°C when operating at a wet bulb temperature of 17°C and a dry bulb temperature of 20°C. Two 50 cm diameter fans provide forced draught to assist natural convection cooling. Since the tower is absorbing nearly 80 kW when fully loaded, ten gallons of water are evaporated from the reservoir every hour. Make up water is supplied from the mains through a ball cock valve, but to keep the total alkalinity of the tower at a reasonably low figure rather more than ten gallons of water per hour are bled from the tank direct to waste. A thermostatically controlled immersion heater in the reser-

voir and trace heaters on the pipelines remove any danger of frost damage in winter.

The tower is located in the courtyard but the two Holden and Brooks centrifugal circulating water pumps are in the lower level of the liquefier area. Being indoors they are protected from the weather and maintenance can be carried out in greater comfort. The pump capacity is such that one pump is capable of cooling either the helium liquefier or nitrogen liquefier but not both together. By having two small pumps rather than one of twice the capacity it was possible to arrange the pipework so that the machines can be cooled by either. In this way a certain standby capacity is available, since the present demand does not require both liquefiers to be run at the same time. When it becomes necessary to run both machines continuously, a further standby pump will be required.

4.8 Cooling of Philips Nitrogen Liquefier

The design of the water cooling system of the Philips liquefier requires that the exhaust water be allowed to flow freely from the heat exchangers without being subjected to any back pressure. This means that it is not possible to use a closed circuit cooling system directly. The problem was overcome by placing a sump tank in the lower level immediately below the machine into which the spent water is allowed to drop freely. A submersible scavenge pump, controlled by a float switch, returns the water to the top of the cooling tower. To

reduce the danger of the sump tank overflowing, power is fed to the pump immediately the main isolator for the liquefier is closed. As an additional safeguard, a further float switch is fitted at the top of the sump tank controlling a solenoid valve on the water inlet to the liquefier. Should the tank over-fill, due to failure of the scavenge pump system, the power to the solenoid valve is interrupted, the valve closes and the liquefier is automatically shut down.

A Rotatherm contacting thermometer is also fitted to the incoming water line so that in the event of an excessive rise in inlet water temperature the liquefier is also automatically shut down.

4.9 Cooling of A.D.L.-Collins Helium Liquefier

Since the compressors for the helium liquefier will accept a small back pressure in the cooling water, there was no difficulty involved in running them on a closed circuit. While the water is returned to the top of the cooling tower without difficulty we were obliged to fit flow indicators on the exhaust water lines to each compressor. These indicators are of the rotary type and give a quick visual check on the equal division of water flow. In addition, adjustable calibrated thermal trips are fitted to the third stage intercoolers which automatically shut the compressors down in the event of an excessive rise in temperature.

A periodic check on the efficiency of the cooling system is also carried out by means of these trips. By running the compressors for a

predetermined period and then reducing the tripping temperature until the compressor stops, a cut-out temperature is established. From past records it can easily be established whether or not the intercoolers are maintaining their performance.



Photo 5.1 The helium gas recovery and distribution panel

CHAPTER 5

System Monitoring and Control

5.1 Liquid Helium Production Costs

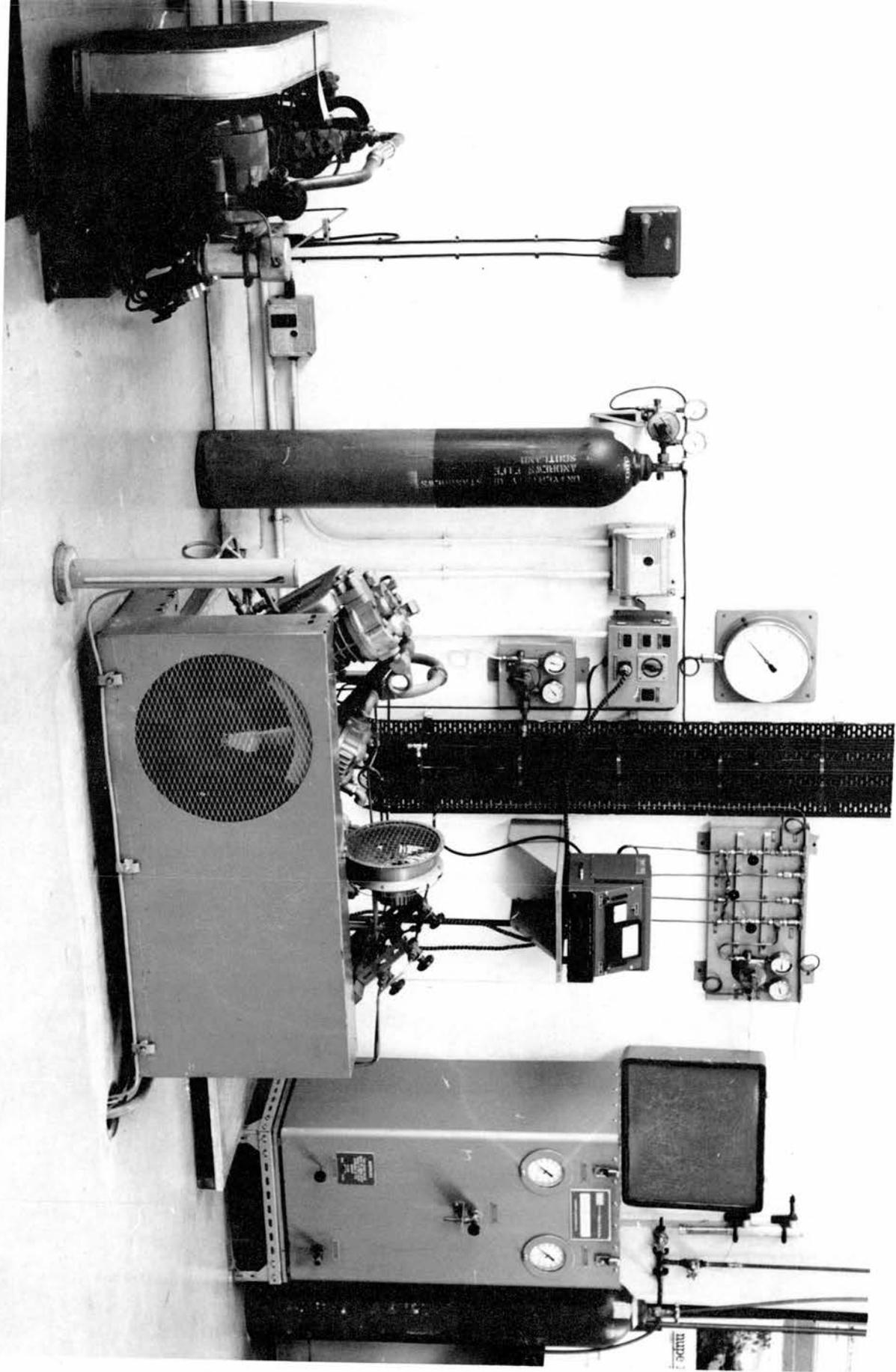
Looking again at our liquid helium production costs as outlined in Chapter 3 we see that for an annual consumption of 2,500 litres per annum the cost per litre liquid used is £1 16s.

Increasing the annual consumption of liquid to 5,000 litres the total cost per litre of liquid would fall to £1 0s. 2d., provided the helium gas recovery and work force could be kept the same.

Referring to Chapter 3, page 53, it will be noted that half of one technician's time is allotted to running the nitrogen liquefier but by training two technicians in the operation of both liquefiers we provide back-up in the event of illness, holidays, etc., as well as ensuring a more complete supervision of helium gas recovery. Adding the extra one half of one technician's time to the cost of liquid helium raises it only 2/- per litre to £1 2s. 2d. on the 5,000 l/year basis.

To ensure that the technicians in charge have time to carry out routine maintenance and repairs it is essential that the gas monitoring processes should be made automatic and that as far as possible faults should not only be indicated but located as well. Furthermore as many as possible of the systems should be monitored and controlled from one central panel. Photo 1.

Photo 5.2 Helium recovery compressor with repurification cabinet. The standby compressor is on the left of the photograph



5.2 Helium Gas Recovery - Photo 2

The helium gas from all experiments is fed via return lines to an atmospheric pressure gasholder. When the latter is full it automatically starts a compressor which puts the gas into high pressure storage cylinders, from whence it goes to a repurifier before being reliquefied.

When a helium storage vessel or experimental cryostat is to be filled with liquid helium it is first cooled to about 80°K by liquid nitrogen, and must then be cooled from there to 4.2°K by liquid helium before the latter begins to collect in the vessel. This final cooling is thus performed at the expense of the latent heat of the liquid and the heat capacity of the cold helium gas. Since the latent heat of vaporisation of liquid helium is very low, 0.6 cal/cc, a large volume of gas is evolved during this initial cooling process. An illustration of this very low value of latent heat is given by the fact that it would require 33 ccs of liquid helium to cool 1 gm of copper from 77°K to 4.2°K if the latent heat of vaporisation only were used. The volume ratio of room temperature gas to 4°K liquid for helium is 740:1. Therefore in the above example, 24.4 litres of gas at N.T.P. would be evolved. By careful design of the cryostat and transfer system, the heat capacity of the gas can be utilised to assist in the cooling process, thereby reducing by as much as a factor of ten the amount of liquid used up

siderable journey to the gas storage area to see the state of the gas holders before releasing gas in quantity back to them, indicators in each research room show not only the level of gas in the holders, but also whether they are filling or emptying.

In Fig. 1, it will be seen that the gas is taken from these gas holders by means of a rotary pump and delivered to the recovery and repurifying compressor. The rotary pump was chosen to have a throughput somewhat smaller than that of the compressor so that there is no danger of the repurifying gas holder being overfilled. Micro switches on the gas holders start the pump as soon as there is 0.8 m^3 of gas present; a volume about equal to that of the entire helium pumping line. It is necessary to have 0.8 m^3 of gas always available because at the completion of a helium run the evacuated pumping line must be refilled to atmospheric pressure with helium from the gas holder. If it were filled to any lower pressure, air might be sucked into the line, which would be undesirable. By arranging the micro switches as described, there is always the maximum usable volume available in the gas holder should there be an emergency and gas returned rapidly from a cryostat without warning.

5.3 Gas handling control panel

The two gas holders are painted blue and white, while the recovery compressor bag to which the gas is pumped for storage is

Fig. 5.1

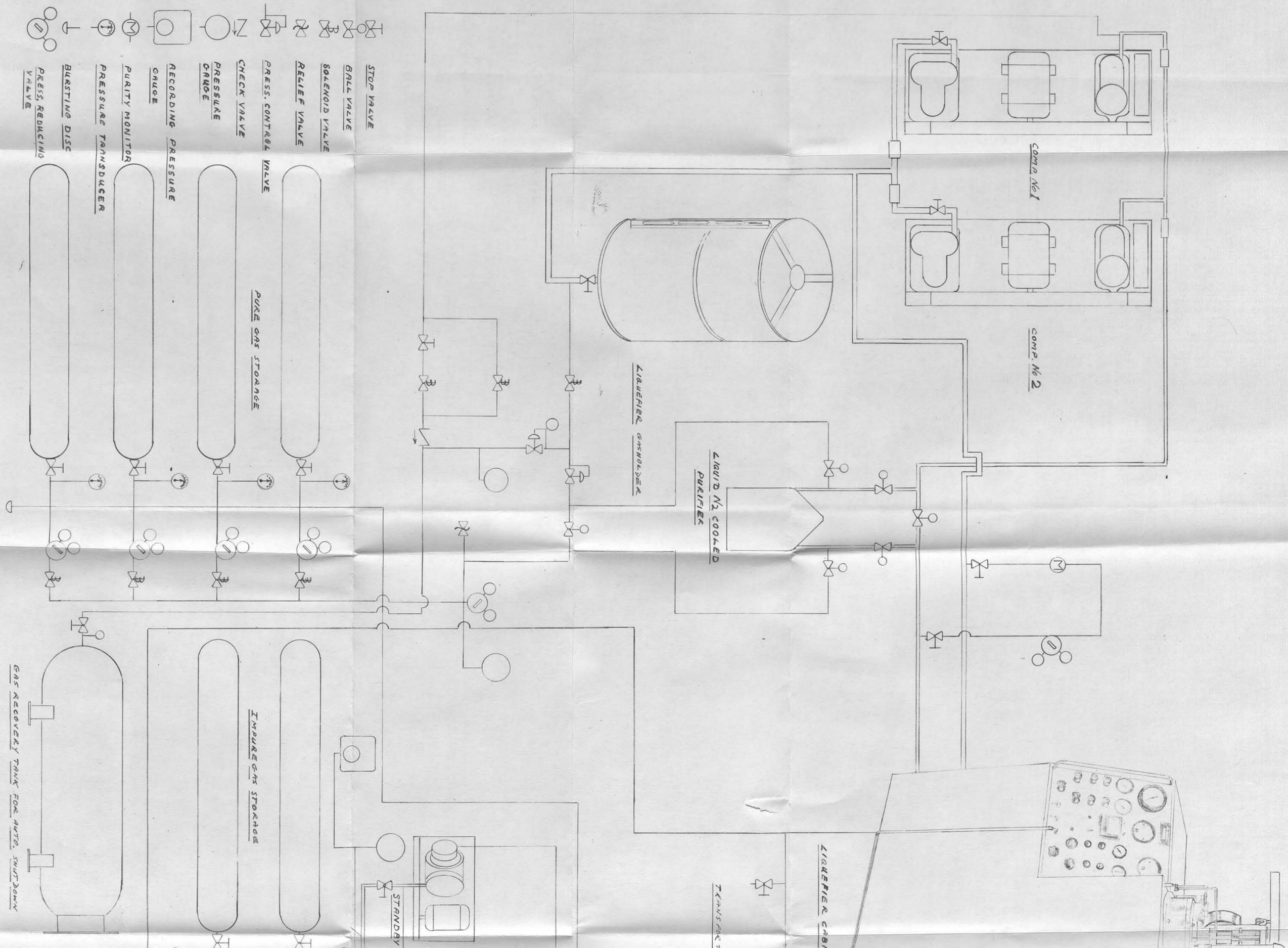
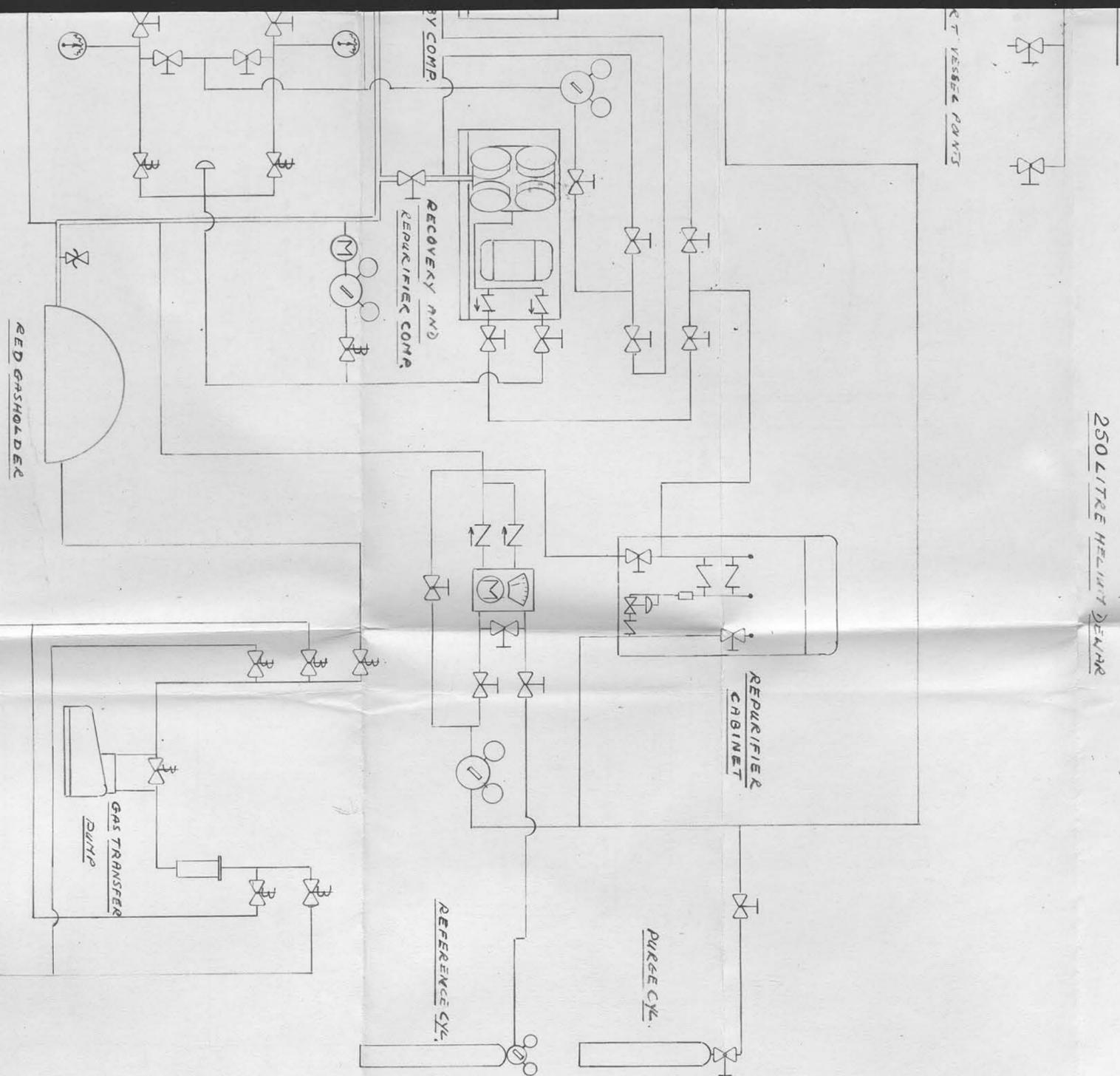
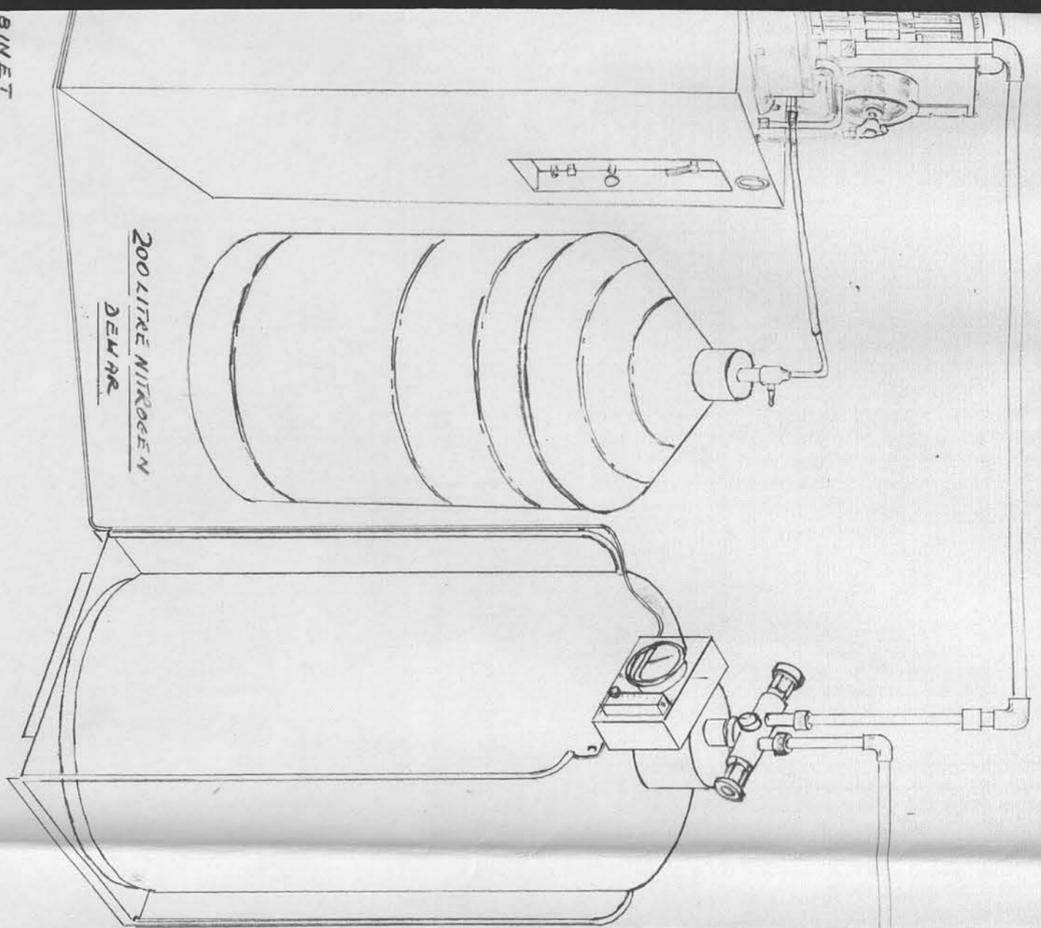


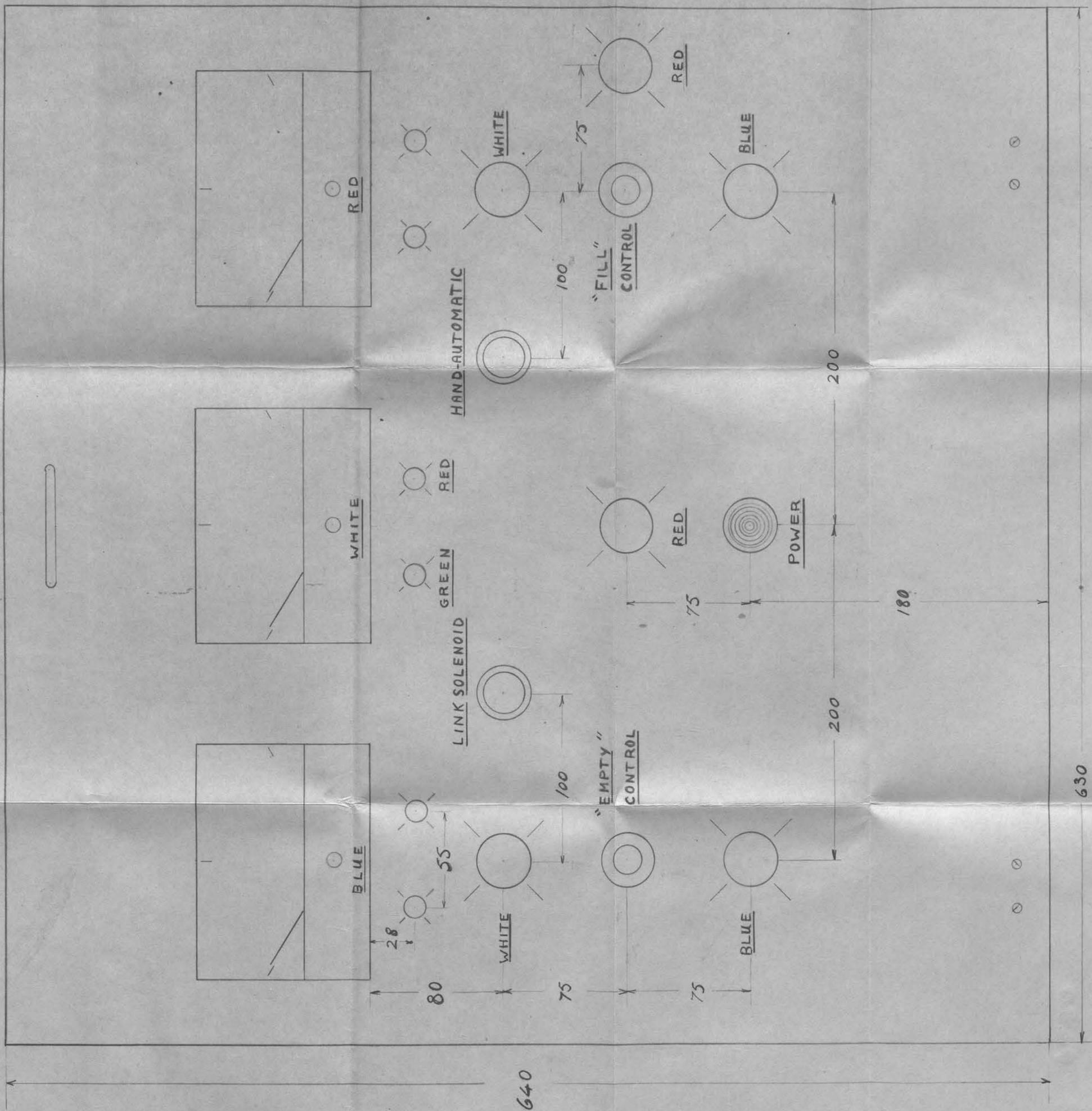
Fig. 5.2



HELIUM LIQUEFACTION,
RECOVERY AND
REPURIFICATION

SCHEMATIC PIPING CIRCUIT

RETURN LINE FROM
 TEACHING APPS.
 RETURN & LINE
 CRYOSTAT
 RETURN LINE
 FROM PANTS



HELIUM RECOVERY
CONTROL PANEL

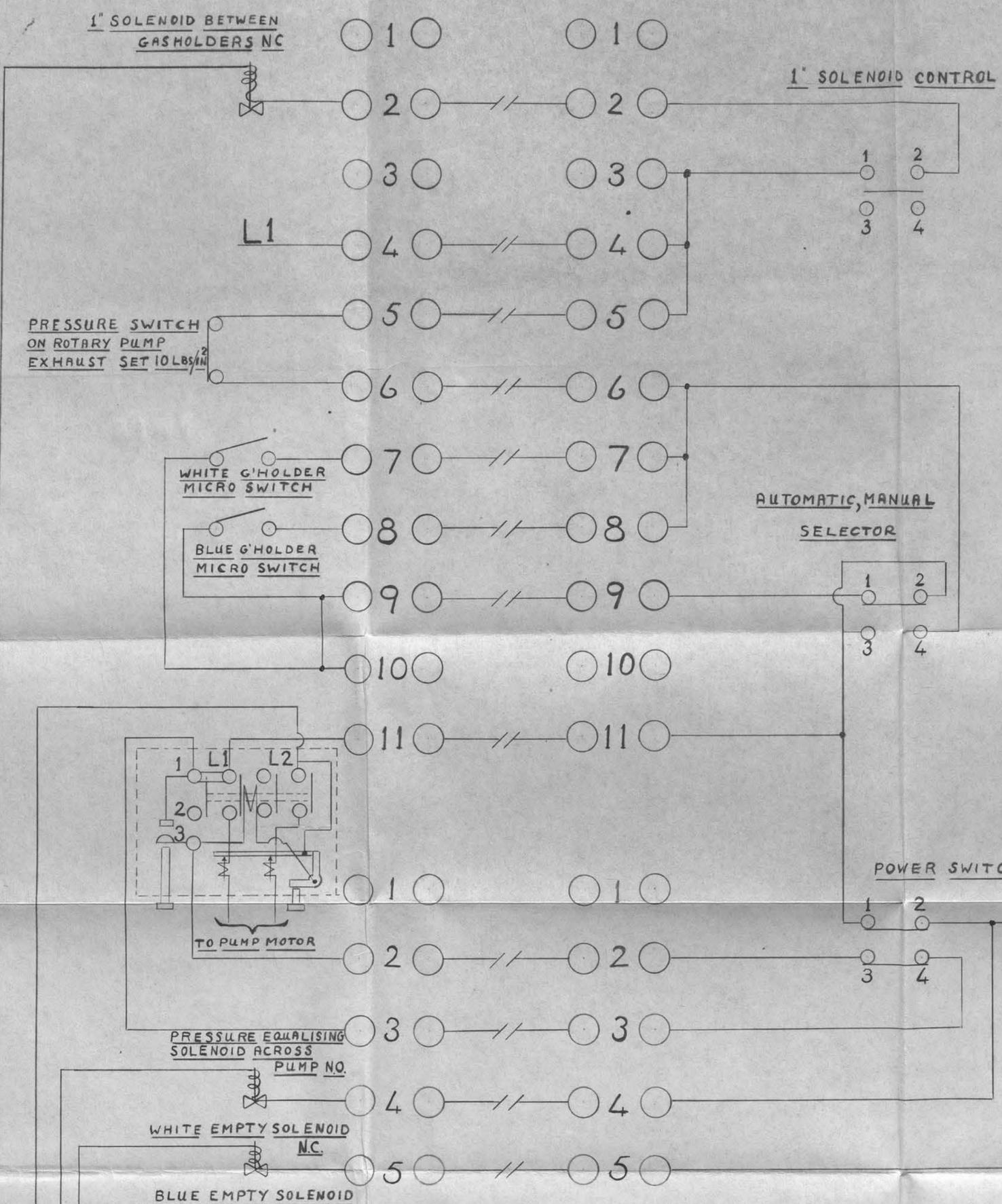
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ALL DIMENSIONS IN MILLIMETRES

painted red. Figure 2 shows the control panel layout, and Figure 3 shows the electrical circuit. It will be seen that operation can be either manual or automatic and the joystick selector switches can be set up first before the circuit is energised, thus avoiding gas being inadvertently sent to the wrong gas holder.

5.4 Purity and Pressure Monitoring

When an experimenter takes a helium storage vessel away to fill his cryostat there is usually no way of checking that all of the gas from the liquid which is transferred is recovered. This means that an unknown quantity of gas may be lost due to poor transfer tube insertion technique or leaks in the cryostat. Since a litre of gas at 4°K amounts to about 100 litres at N.T.P. even a small leak of very cold gas is undesirable and costly. To check losses of this kind, there is a recording pressure gauge attached to the recovery cylinder. The experimenter initials the chart on this recorder before taking the storage vessel to his research room. Since the amount of liquid he requires for his apparatus is known, the amount of gas expected back is also known. If the recovery pressure is unduly low, or the gas return too small, then a fault must have occurred and must be traced to its source. It is, of course, likely that there will be several persons running at one time but then it is a simple matter to arrange for a possible offender to run separately for test purposes.

Fig. 5.3



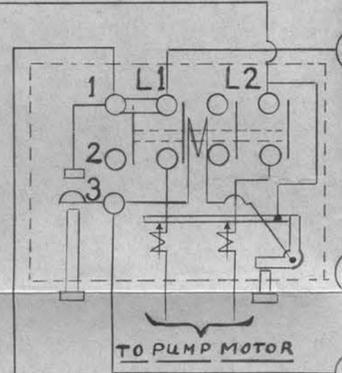
LEVER CONTROLS "CEMA" SERIES 99
TYPE TS 1234 R

PILOT LIGHTS "CEMA" SERIES 99

PUSH BUTTON SWITCHES "NAMCO" SERIES N

PRESSURE SWITCH MECHANISMS LTD TYPE M2015

SOLENOID VALVES DEWRANGE - MSCO TYPE 8210A94



BLUE EMPTY SOLENOID

N.C.

WHITE FILL SOLENOID

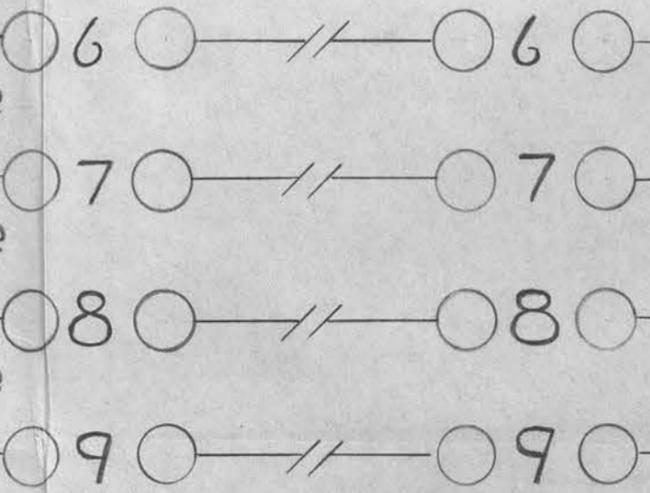
N.C.

BLUE FILL SOLENOID

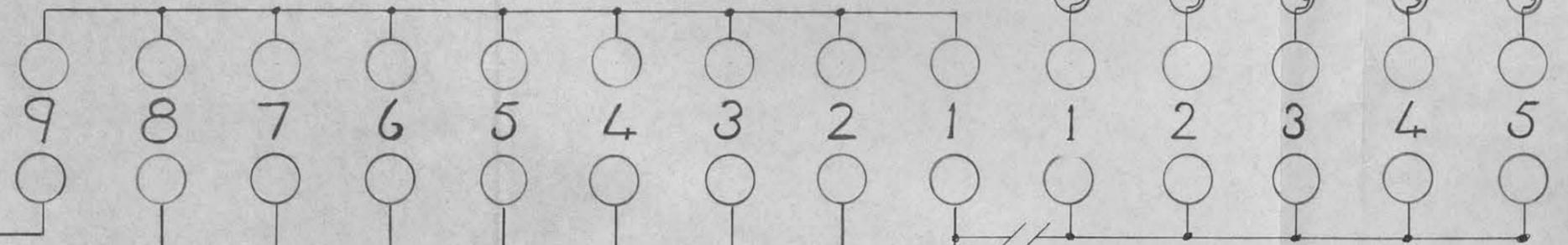
N.C.

RED FILL SOLENOID

N.C.



LBL 220 LB 220 LBL 220 LB 220 LR 220



L2

HELIUM RECOVERY
CONTROL AND SOLENOID
PANEL

SCHEMATIC ELECTRICAL CIRCUIT

Should an experimenter operate a leaking cryostat at a pressure below atmospheric, then, since the pump exhaust line is connected to the recovery system and if the leak is a bad one, one will get more gas back than was taken away in the form of liquid, the additional gas of course being air. Whether or not the leak is a large one, the gas will be contaminated. By means of monitoring and recording the purity of the returning gas, using a similar system of chart initialling as for pressure recording, any offending cryostat or pump can be located.

If a large quantity of air should happen to be drawn into the main helium storage system, the repurifier, which is based on N_2 -cooled charcoal, would be unable to cope since it will accept only about 0.6 m^3 of impurity before ceasing to function. However, by having a further purity monitor on the high pressure side of the storage compressor together with a switching indicator, very dirty gas can be directed into a separate dirty storage cylinder. When the repurifier is next used gas from this dirty cylinder can be directed through the purifier at the end of the normal purifying run, thus ensuring that no time is wasted due to the repurifier blocking. Of course if the gas is very badly contaminated it will be cheaper to discard it rather than attempt heavy purification. This procedure would not be possible if all gas were stored in a single system.

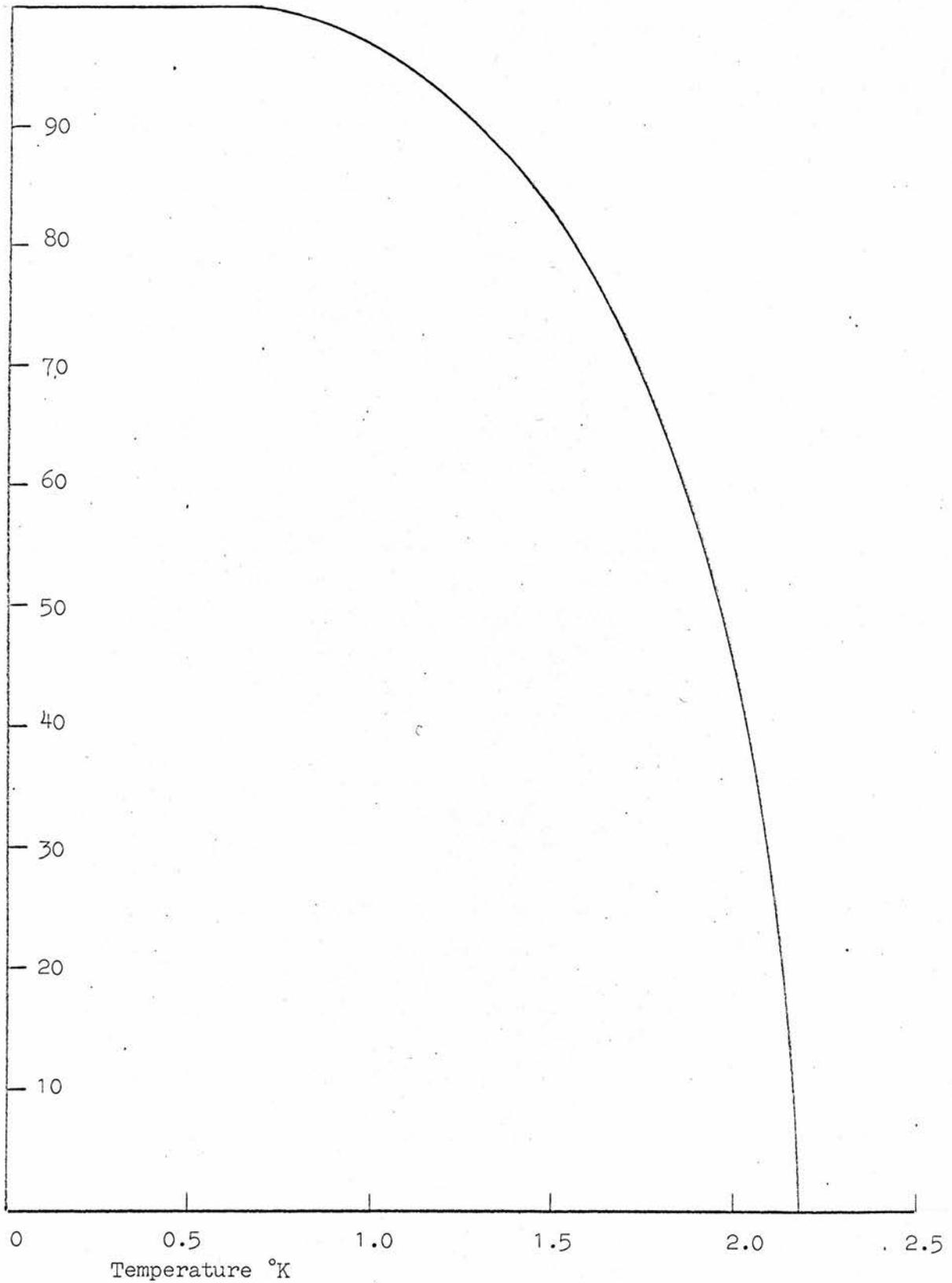
The helium storage cylinders for recovered gas have a total capacity of 112 m^3 at a pressure of $2,000 \text{ lbs/in}^2$. The pressure in these cylinders is indicated on the main control panel using pressure transducers on the cylinders and 0-1 mA meters on the panel, reading $0-2,000 \text{ lbs/in}^2$. Electrical transducers on the cylinders enable the pressure to be read remotely and also avoid the use of vulnerable small diameter high pressure gas lines leading to gauges.

5.5 Pure Helium Gas Storage

A standard commercial helium cylinder contains 6 m^3 of gas equivalent to 8 litres of liquid. Since one normally makes about 220 litres at one time, this would require a battery of 28 cylinders, and a corresponding number of couplings and potential leak sources. Instead, we store the pure gas in four large high pressure cylinders each with a capacity of 60 m^3 . By fitting the cylinders with reducing valves and solenoid valves, gas can be drawn as required from high pressure storage by simple manipulation at the central control position.

Four pressure transducers, one on each cylinder, are used to provide a remote indication of pressure, again connected to 0-1 mA meters scaled $0-2,000 \text{ lbs/in}^2$.

%
100



Percentage Superfluid as a function of Temperature

Fig. 6.1

CHAPTER 6

6.1 Cryostat Pumping and Helium Recovery

6.1.1 Relevant properties of liquid helium

The boiling point of liquid helium under atmospheric pressure is 4.22°K , the liquid range being from its critical temperature of 5.2°K at a pressure of 2.26 atmospheres down to the absolute zero, there is no triple point and to solidify the liquid it must be subjected to a pressure in excess of 25 atmospheres.

As the vapour pressure is reduced below 40 mm Hg, at 2.19°K , the liquid undergoes a change, becoming known as helium II. This transition temperature is called the lambda point because of the resemblance of the shape of the specific heat curve to the Greek letter lambda.

Helium II displays many unique properties and the two fluid theory of London, Landau and Tisza has been very successful in providing a description of these properties. In this theory liquid helium II is considered to be a mixture of a superfluid component with zero entropy and a normal component possessing all of the thermal energy and entropy of the fluid. The superfluid concentration is a function of temperature, being 100% at the absolute zero and zero at the λ point, (Fig. 1).

The expression superfluid coined by Kapitza in 1939, refers to the ability of liquid helium II to flow so rapidly through very

fine channels that it appears to have almost zero viscosity (Allen and Misener, 1938). Another unique property of liquid helium II is that for all practical purposes it is a perfect conductor of heat, which means that no temperature difference can persist in the liquid bath.

Since reducing the vapour pressure reduces the temperature of the liquid in the bath, the lower the pressure which can be attained over the liquid the better. There are however practical limits to the temperature which can be obtained by any particular pumping means. The vapour pressure of liquid helium at 1.26°K is 1 mm Hg at 1°K 0.12 mm Hg but at 0.3°K the vapour pressure is only $3 \cdot 10^{-10}$ mm Hg.

6.2 Pump Types and Characteristics

The capacity of pump required to attain lower temperatures is obviously determined by the unavoidable evaporation losses from the cryostat due to heat leaks of various sorts, mainly conduction and radiation. In practice it is difficult to keep the heat leak into a cryostat below 0.03 watts, so that suitable pressures and temperatures cannot be obtained with a pump having a capacity of less than 500 litres/minute. Due to its comparatively large size, high noise level and mechanical vibration, it is unlikely that such a pump would be tolerated in the research room itself. We must therefore consider pumps of large capacity and pipelines suitably dimensioned which will

- X MECHANICAL BOOSTER PUMP
- ROTARY PISTON PUMP
- GAS EVOLVED FROM CRYOSTAT BEFORE COMPRESSION

Speed
Litres/Minute

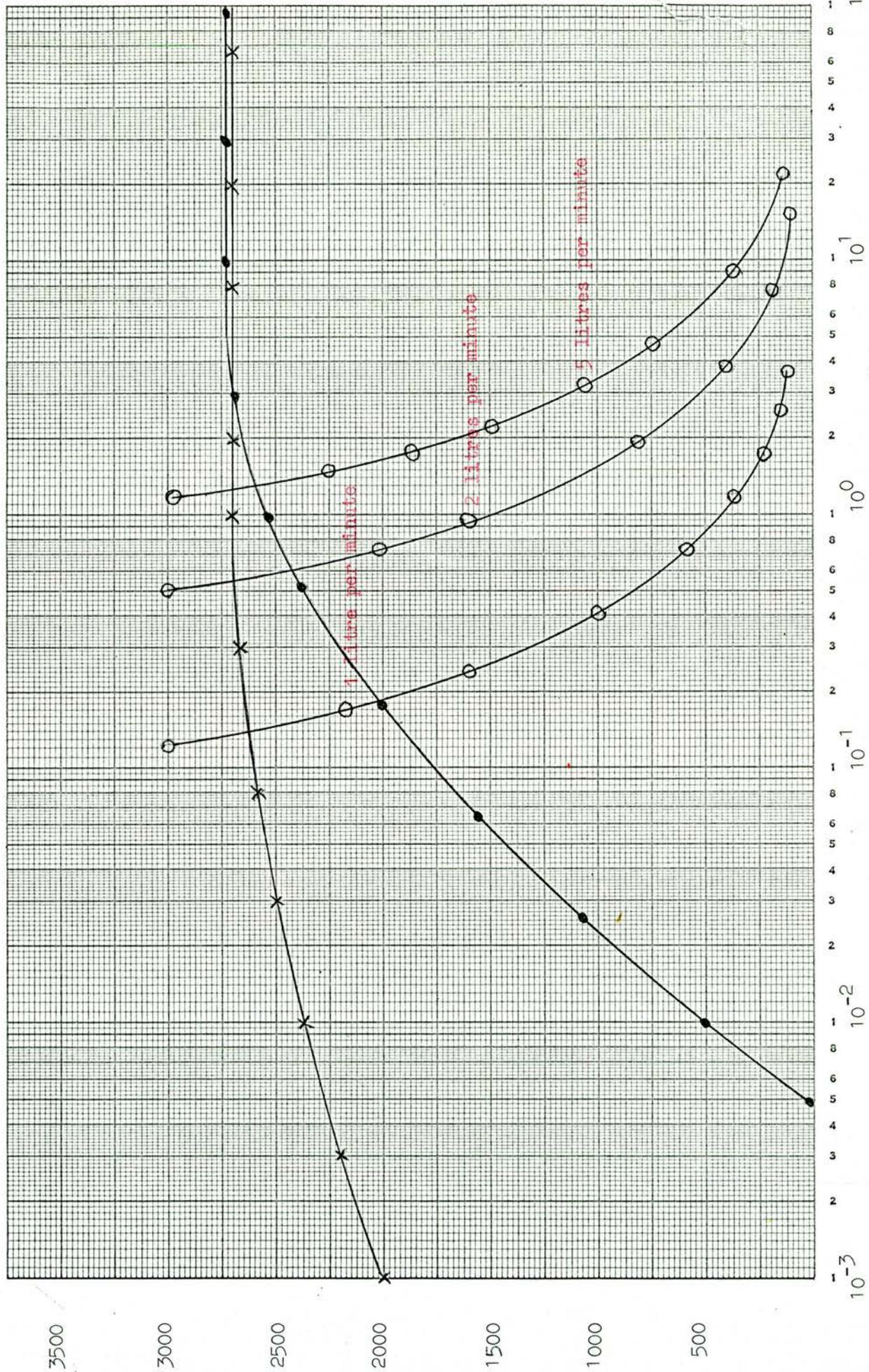
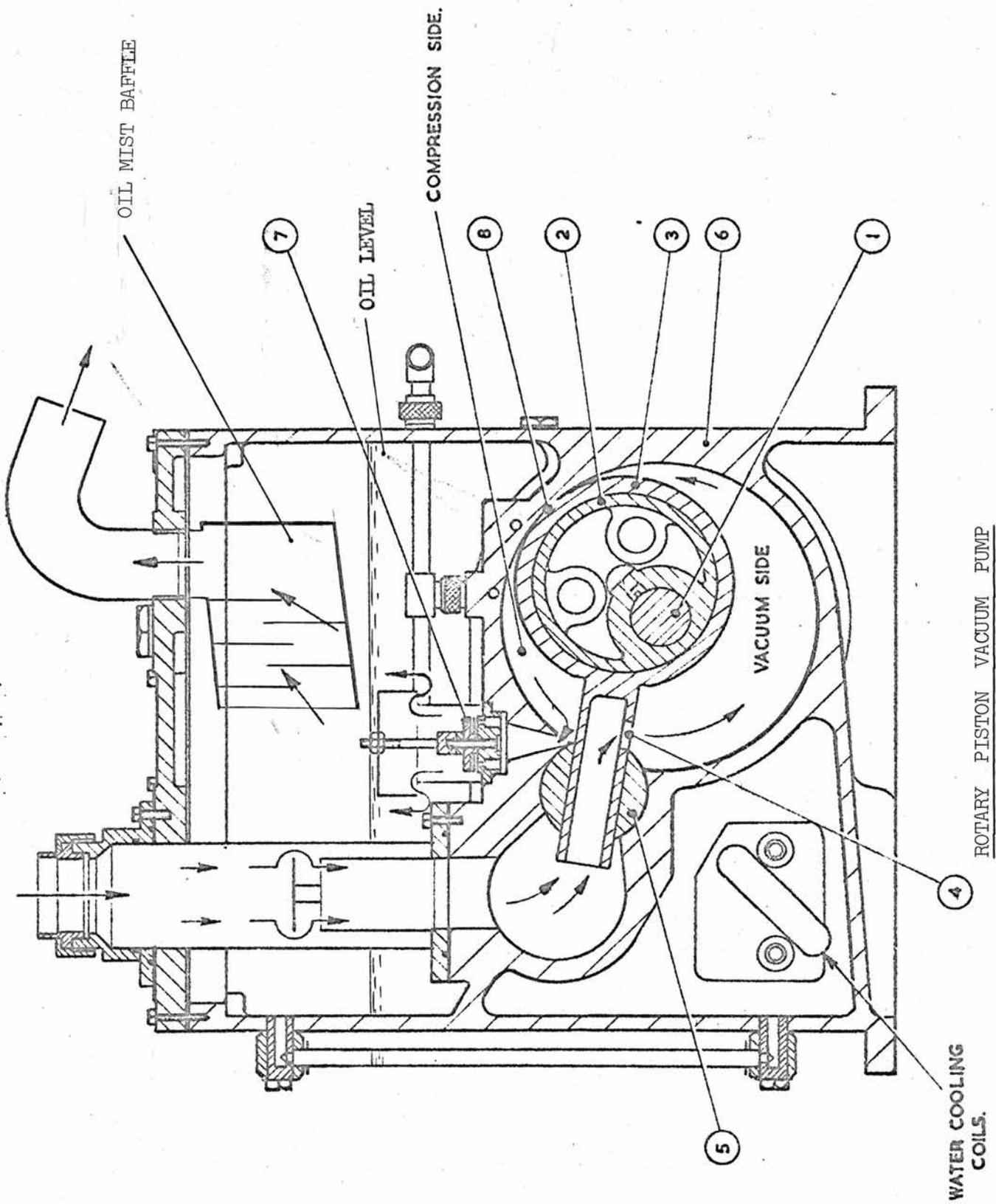


Fig. 6.2

Pressure mm Hg



ROTARY PISTON VACUUM PUMP

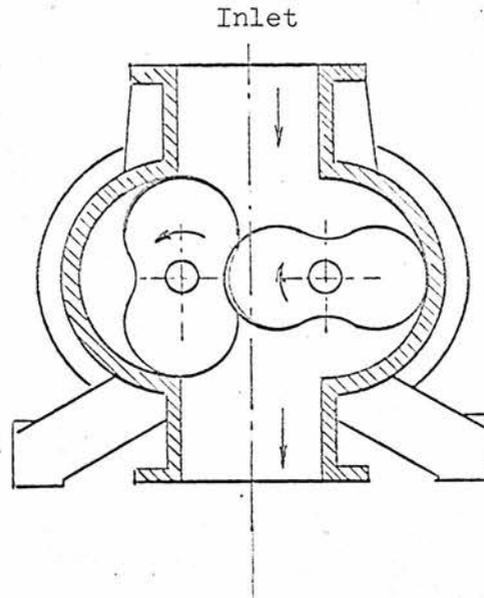
Section Showing Working Principle. FIG. 6.3

permit a single pumping station to serve an area in which there may be a dozen or more cryostats attached at various inlets to a ring main vacuum line, although it is unlikely that more than two will be operating at any one time.

Normally the evaporation rates of cryostats range between those of small glass dewars, about 30 litres of gas at N.T.P./hour and large metal dewars about 300 litres of gas at N.T.P./hour. As will be seen from Fig. 2, a rotary pump with a nominal capacity of about 3000 litres/minute is sufficient for most purposes provided the pipeline between pump and cryostat does not involve too large a pressure drop. Fig. 2 indicates that a cryostat with a low heat input connected close to the pump without any intervening valves or pipelines reaches a temperature of about 0.9°K while a cryostat with a high heat input under the same conditions reaches a temperature of about 1.3°K .

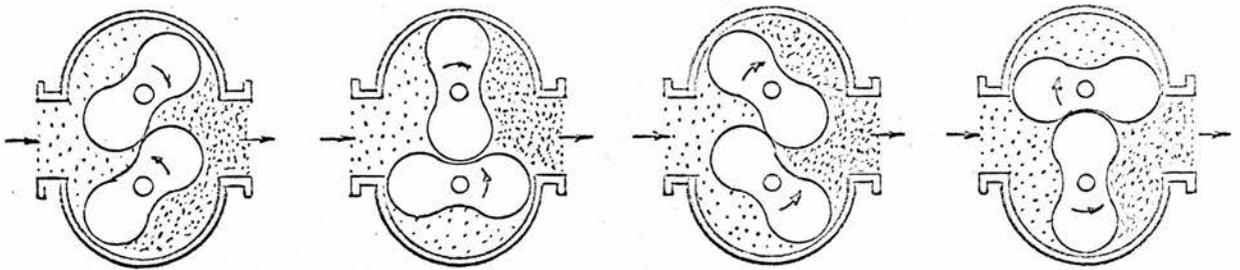
In a mechanical vacuum pump a volume of low pressure gas is compressed to atmospheric pressure with each revolution of the pump. Fig. 3 illustrates the principle of a rotary piston pump of the type used for pumping helium.

An eccentric cylindrical member 2 is keyed to the drive shaft 1 which is located centrally in a cylindrical housing. The eccentric causes the cylindrical member 3 to sweep the volume of the stationary housing 6, sealing at point 8 being accomplished by a



To Backing Pump

Section Through Rotary Booster or Roots Pump



Schematic Diagram of Pumping Cycle

Fig. 6.4

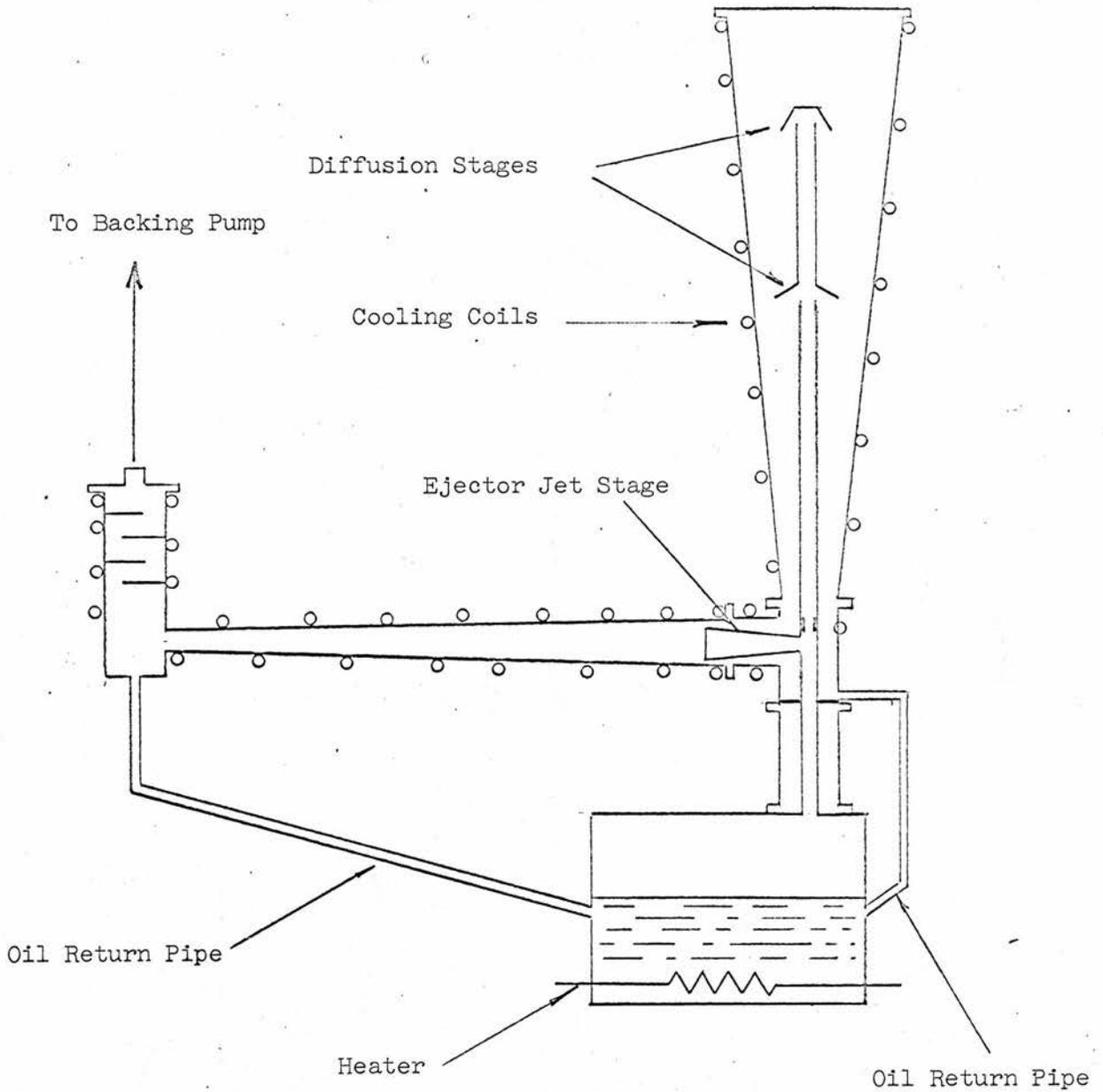
film of oil. A hollow sliding bar 4, located by the wrist pin 5 allows gas into the interior of the pump. The sealing of wrist pin and bar again depends on a film of oil. The compressed gas is forced out through the exhaust valve 7 into the oil and out of the pump.

In the design shown, rotational speeds have to be kept low due to the problem of dynamic imbalance. A much better design has three stages in parallel, the outer two stages being half the size of the inner and 180 degrees out of phase. In this way both mechanical and gas loads are balanced out and vibration free running up to 1200 r.p.m. is quite normal.

It will be noted from Fig. 2 that the pumping speed falls off very rapidly at pressures below about 1 mm Hg and reaches a limit of $5 \cdot 10^{-3}$ mm Hg. There are a number of factors which influence this reduction in throughput; among them are:-

- (a) the vapour pressure of the pump oil,
- (b) the vapour pressure of possible contaminants,
- (c) the solubility in the oil of the gas being pumped,
- (d) leaks across the end plates and shaft seals, and
- (e) the unavoidable dead space at the exhaust point.

A method of further lowering the pressure over the helium bath advocated by some experimenters is the mechanical booster pump, Fig. 4. This type of pump or Roots blower contains two counter-rotating lobes



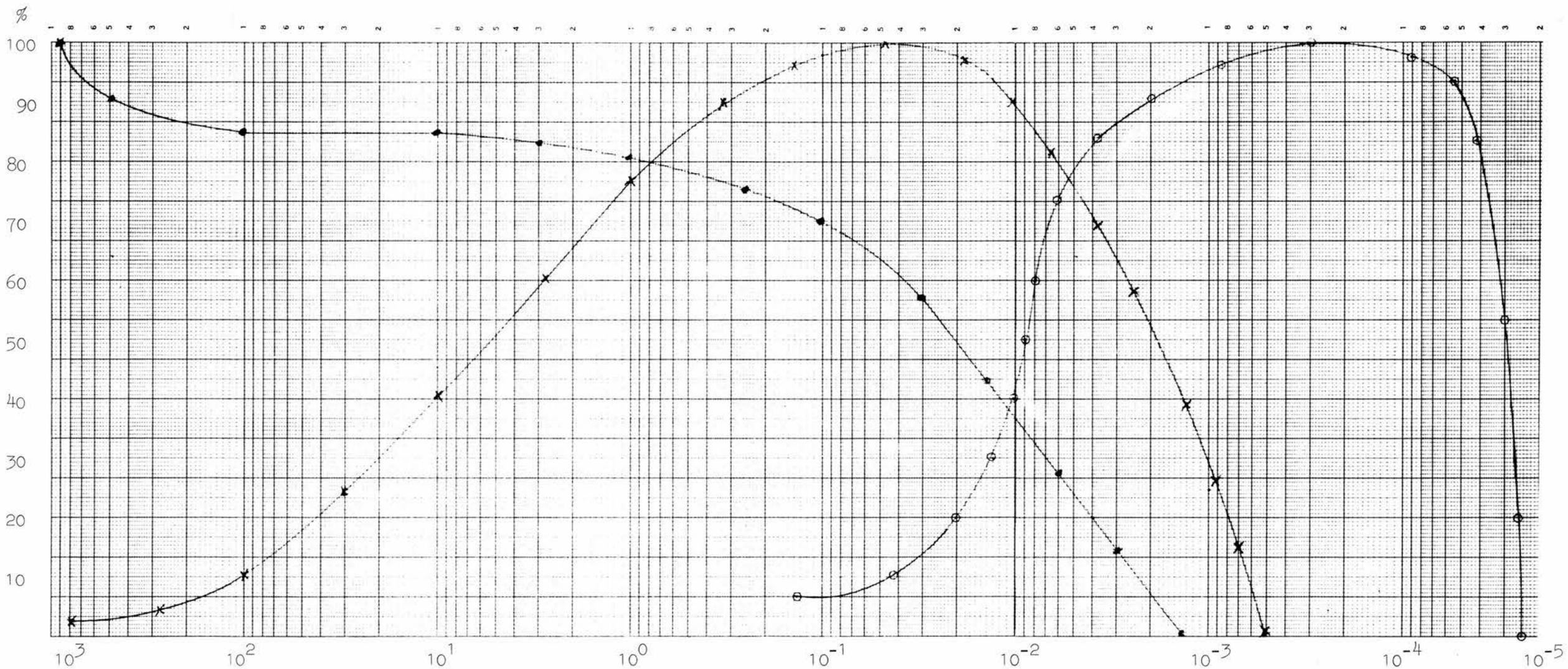
Schematic Diagram of Vapour Booster Pump

Fig. 6.5

each with a figure eight cross section. These lobes do not touch either each other or the pump casing. A clearance between lobes and between lobes and casing of 0.15-0.2 mm is commonly used. Normally a single shaft drives the machine with the second rotor synchronised and driven through a set of timing gears. It requires to be backed by a rotary piston pump as it cannot by itself compress gas up to atmospheric pressure, but running together they can maintain high pumping speeds down to about 5×10^{-3} mm Hg when the pumping speed falls off quickly to the ultimate of about $5 \cdot 10^{-4}$ mm Hg.

It would appear at first sight that the Roots pump combination with its high throughput maintained to well below that of an ordinary rotary pump would be ideal for pumping cryostats. However, if we plot a curve of pumping speeds from a Roots/rotary combination and an ordinary oil sealed rotary pump (Fig. 2), it will be noted that the reduction in temperature is only about 0.009°K for the cryostat with the lower heat input.

A much more satisfactory and economical way to obtain lower temperatures is by using a vapour booster pump. This is a particular kind of oil diffusion pump with an ejection jet stage as shown in Fig. 5. In an oil diffusion pump the operating fluid is vaporised in a boiler and by means of umbrella shaped jets ejected at high velocity away from the pump inlet. The gas molecules diffuse into



Pressure mm Hg

PUMPING SPEED AS A PERCENTAGE
OF MAXIMUM PUMPING SPEED FOR:-

- ROTARY PISTON PUMP
- x MECHANICAL BOOSTER PUMP
- VAPOUR BOOSTER PUMP

Fig. 6.6

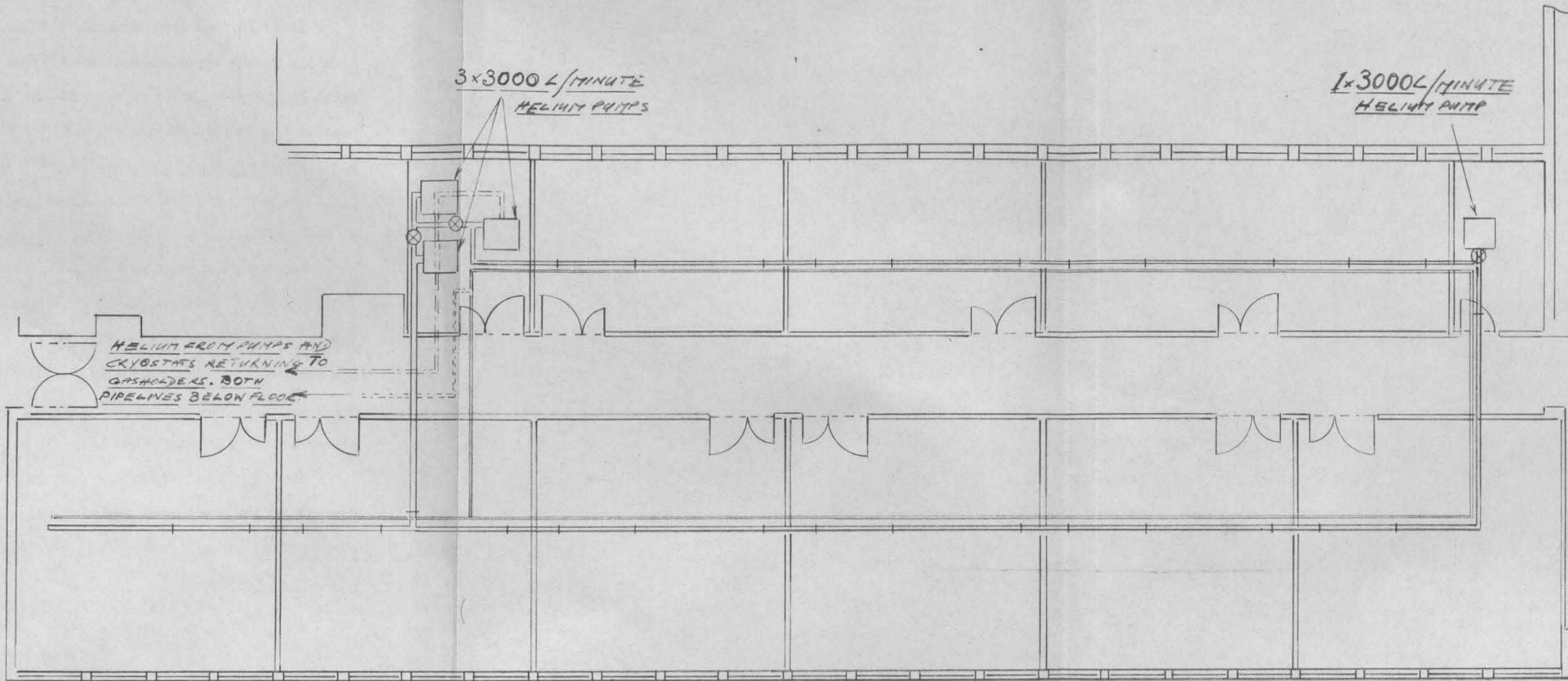
the vapour jet and are swept downstream. The jet is then condensed on the water or air cooled pump wall. The gas atoms or molecules are thus swept towards the discharge end of the pump and removed by a rotary vacuum pump.

In a vapour booster, some of the vaporised fluid is conducted through a nozzle or ejector stage towards the backing pumpline. Booster pumps, in common with ordinary oil diffusion pumps require to be backed by mechanical pumps but possess the advantage that they will operate effectively with a backing pressure of 10 mm Hg compared with 0.1 mm Hg required for an oil diffusion pump.

This high backing pressure tolerance together with high pumping speeds at around and below 10^{-2} mm Hg down to their ultimate of about $5 \cdot 10^{-5}$ make them very useful for reducing the pressure in cryostats. They have certain disadvantages such as fluctuations in pressure and therefore temperature, due to surging in the ejector jet stage. These fluctuations can be much reduced by careful control of the amount of fluid in the boiler and the power supplied to the boiler heater. With a carefully designed low heat loss cryostat, stable temperatures of 0.8°K can be attained.

Fig. 6 shows a comparison between a rotary piston pump, a Roots pump and a vapour booster pump with pumping speed as a per-

Fig.6.7



3 x 3000 L/MINUTE
HELIUM PUMPS

1 x 3000 L/MINUTE
HELIUM PUMP

HELIUM FROM PUMPS AND
CRYOSTATS RETURNING TO
GAS HOLDERS. BOTH
PIPELINES BELOW FLOOR

- 
 5 CM RADIUS PUMPING
LINE SHOWING POSITION OF
VALVE/CONNECTORS
- 
 3.8 CM RADIUS RETURN
LINE FROM PUMPS
- 
 1.25 CM RADIUS RETURN
LINE FROM CRYOSTATS
- 
 DIAPHRAGM VALVE

PLAN OF LOW TEMPERATURE RESEARCH AREA

SHOWING HELIUM PUMPING AND RETURN LINES

NB TAP OFF POINTS TO CRYOSTATS NOT SHOWN

SCALE :- 96-1

centage of maximum pumping speed plotted against intake pressure.

It is obvious that a vapour booster has to be coupled directly to the cryostat with the minimum of pipework. As it is quiet in operation and can be backed by the main vacuum line the booster can and indeed must be situated in the research room.

6.3 Vacuum Pumping Line and Pump Matching

The boiling point of liquid helium under atmospheric pressure is 4.22°K , but for many experiments as large a temperature range as possible is required. By reducing the pressure over the boiling liquid the temperature can readily be lowered to about 1.4°K , a pressure of 2.2 mm Hg. Lowest temperatures of about 1.4°K are adequate for most purposes and we have seen that such temperatures can most readily and economically be obtained by centrally situated rotary piston pumps.

The nine low temperature research rooms in this laboratory are all on the same level and situated along both sides of a single corridor as shown in Fig. 7. Using this layout a ring main type of pumping line can most usefully be employed. This means that the maximum distance any cryostat can be from a pump is 20 metres, assuming the pumps are located as shown.

Taking these points into consideration we have to determine what diameter of pipeline will be suitable for the pressures and pumping speeds involved together with the practical problems of

installation.

If we consider a cryostat which has a total heat input such that 250 ccs of liquid are evaporated per hour this will result in 180 litres per hour of gas at N.T.P. being evolved from the cryostat.

The pressure drop along a pipe can be determined from the Poiseuille relation

$$PV = \frac{\pi a^4 (P_1^2 - P_2^2)}{16\mu L}$$

where P_1 and P_2 are the pressures at either end of a pipe of length L and radius a for a gas of viscosity μ . We need to know the minimum cryostat pressure at the farthest point from the pump and we accordingly need to determine the pressure drop along a line of maximum length 20 metres, for the expected evaporation rate V of 180 l/hr of helium gas at N.T.P. Referring again to Fig. 2 we see that the lowest pressure at the pump end of the line P_2 is 0.9 mm Hg.

We can rearrange the equation,

$$P_1^2 = \frac{16\mu LPV}{\pi a^4} + P_2^2$$

Putting in the values, $\mu_{\text{He}} = 2.10^{-4}$ C.G.S.U., $L = 20$ m,
 $P_2^2 = 1.37 \cdot 10^6$ dyne² cm⁻⁴, and $V = 50$ cm sec⁻¹, and if we choose
a pipe of radius $a = 2.5$ cm, we find a value of $P_1 = 1.54$ mm Hg
at the farthest cryostat, which is undesirably large. It does
not match the capacity of the pump, and the condition will worsen
if we want to use a larger cryostat with a greater evaporation
rate. We therefore consider larger pipe sizes which are easily
obtainable on the market and obtain the following table.

Pipe diameter cm	Pressure at Cryostat 20 Metres from Pump mm Hg	Cryostat Temp °K	Superfluid Concentration %
5.0	1.54	1.339	94
7.6	1.05	1.276	95.9
10.0	0.95	1.261	96.1
12.6	0.92	1.256	96.6
15.2	0.91	1.253	97

Thus at the end of 20 metres of pipe of 7.6 cm radius we would obtain a pressure of 0.91 mm Hg at the cryostat or a temperature of 1.253°K. Similarly using 5.0 cm diameter pipe a pressure of 0.95 mm Hg a temperature of 1.261°K. There is therefore no significant advantage in using the two largest diameter pipes since we gain only 0.008°K improvement in temperature over the 10 cm pipe, which corresponds to a gain of only 0.9% in superfluid concentration.

There are the further disadvantages in the two largest sizes of pipe in installation and particularly in manufacturing the valve, flange and coupling assemblies.

In the end, it was decided to employ 10 cm (4") diameter tubing for the ring main pumping line and to couple this to two rotary piston vacuum pumps each with a capacity of 3000 l min⁻¹, valved in such a way that either each can handle one half of the ring main or they can work in parallel to give extra pumping capacity to a single cryostat using both halves of the main.

6.4 Pipeline, Valves and Couplings

Let us now examine suitable pipeline material.

Welded stainless steel would have been the most satisfactory

from the point of view of lightness, strength and durability; it is, however, difficult to fabricate and also expensive. Mild steel is less expensive than either stainless steel or copper but is very difficult to obtain as thin walled tubing in large diameters. Welded and brazed joints are also subject to crevice corrosion which produces volatile reaction products and possible sources of leaks.

Plastic tubing is inexpensive but is subject to creep at joints when O-rings are used in compression; it also suffers from crazing at cemented joints. Furthermore, inexpensive plastics such as polyvinyl-chloride have a high vapour pressure due to trapped solvents and water vapour.

Light gauge copper domestic water pipe B.S. 659, is less expensive than stainless steel and does not have the disadvantages of mild steel or plastic. It is readily obtainable and strong reliable joints can be made with soft solder. A large variety of pipe fittings are available suitable for soft soldered joints, so that pipework can be quickly assembled. For these reasons copper pipe was chosen for all gas recovery and vacuum lines.

As can be seen from the plan of the low temperature research area Fig. 7, the rooms lie on both sides of a central corridor. A 10 cm ring main pumping line serves all of these rooms with a

short branch line to serve one additional room. The height from floor level to pipe centre is 2.7 metres, the pipe being suspended by hangers from the ceiling at 2 metre intervals. The research rooms are also provided with Unistrut frames suspended from the ceiling at a height of 2.6 metres above the floor. These frames are parallel to the pipeline and serve as cryostat frame supports and carry other gas lines, water and electrical services to cryostats and local pumps.

To permit easy changes in room layout, the 10 cm diameter pipework is assembled from standard lengths clipped together with special couplings of our own design. Three standard lengths are used. A four foot straight length for spanning the partition walls between rooms, a six foot length with one tap-off point two feet from one end, and a ten foot length with two tap-off points four feet from one end and two feet from the other. These tap-off tees to which a cryostat can be connected are 30 cms long and the same diameter as the main pumping line. Since the rooms are nearly all twenty feet in length we can thus arrange to have three tap-off points in each room. Flexibility of arrangement is assured since the six foot and ten foot sections can either be interchanged or reversed.

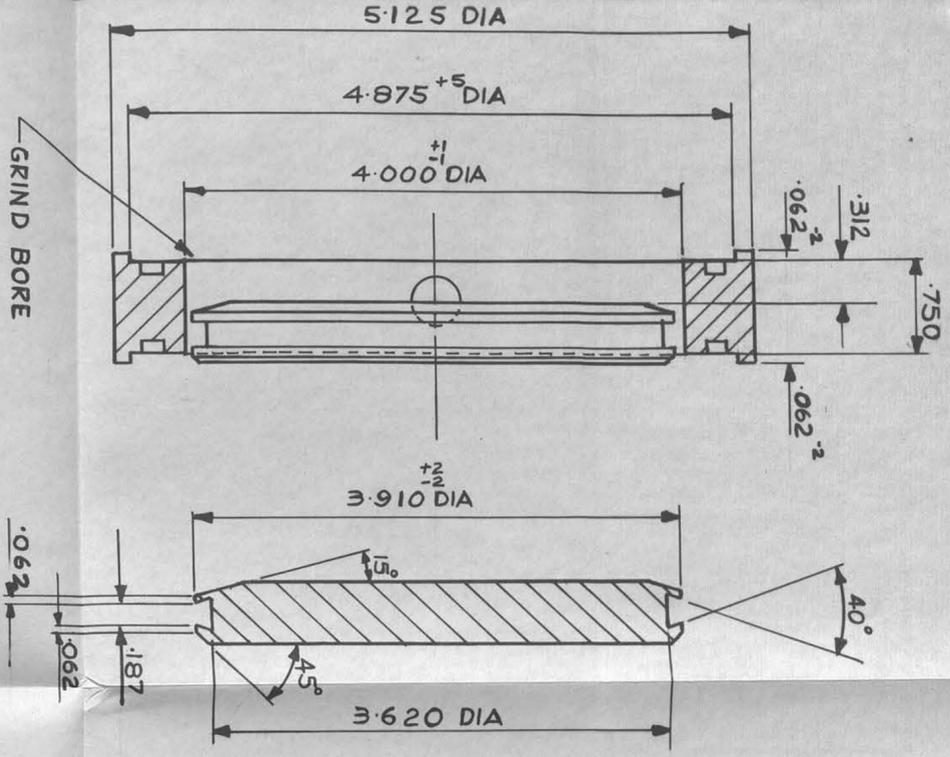
It is highly desirable to be able to separate cryostats from each other to prevent pressure changes in one cryostat from being

communicated to another, which might upset critical experiments. It may also be necessary to have more than one pump operating on one cryostat in order to achieve minimum temperatures. Referring again to Fig. 7 it will be seen that cryostat separation is achieved by inserting a valve at each coupling point in the line. Since we wished to have simple couplings and valve couplings of the same dimensions and interchangeable, and since there were none suitable on the market, we were forced to design and make our own. The design of these fittings is therefore described in detail.

Apart from being leak tight, an efficient vacuum coupling should also have positive locking action in which the O-rings are given a predetermined compression. Furthermore, insertion of the coupling between pipe segments should be easy to do. The valve should have a positive action while the position of the handle should indicate whether the valve is open or closed. In addition, component parts of individual couplings should be interchangeable. From Fig. 8 it will be seen how these requirements have been satisfied with simplicity of design.

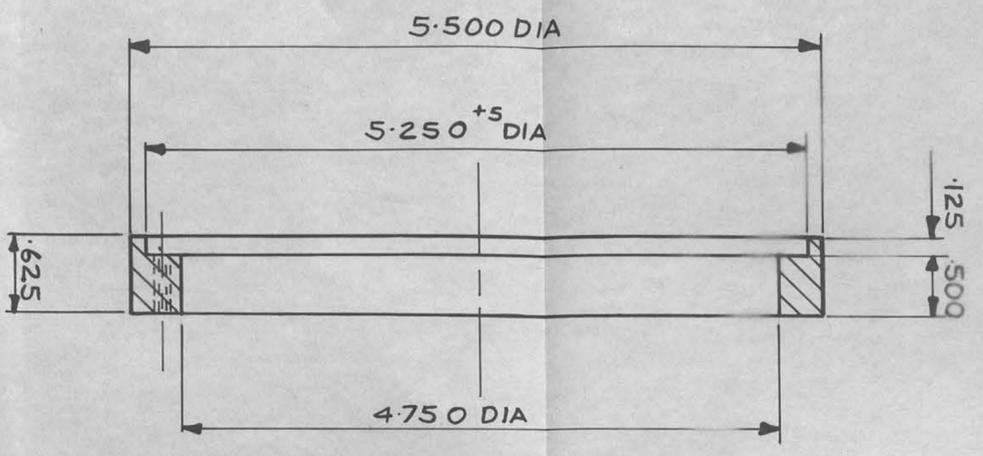
The valve bodies, which were made from thick wall tubing, were first bored in a lathe. The locating rim and O-ring grooves were then machined before they were ground internally. The bodies were then held in the three jaw chuck of a milling machine dividing head for drilling and reaming of the valve spindle holes. The valve gate was turned from bar stock and the spindle attachment holes drilled

Fig.6.8

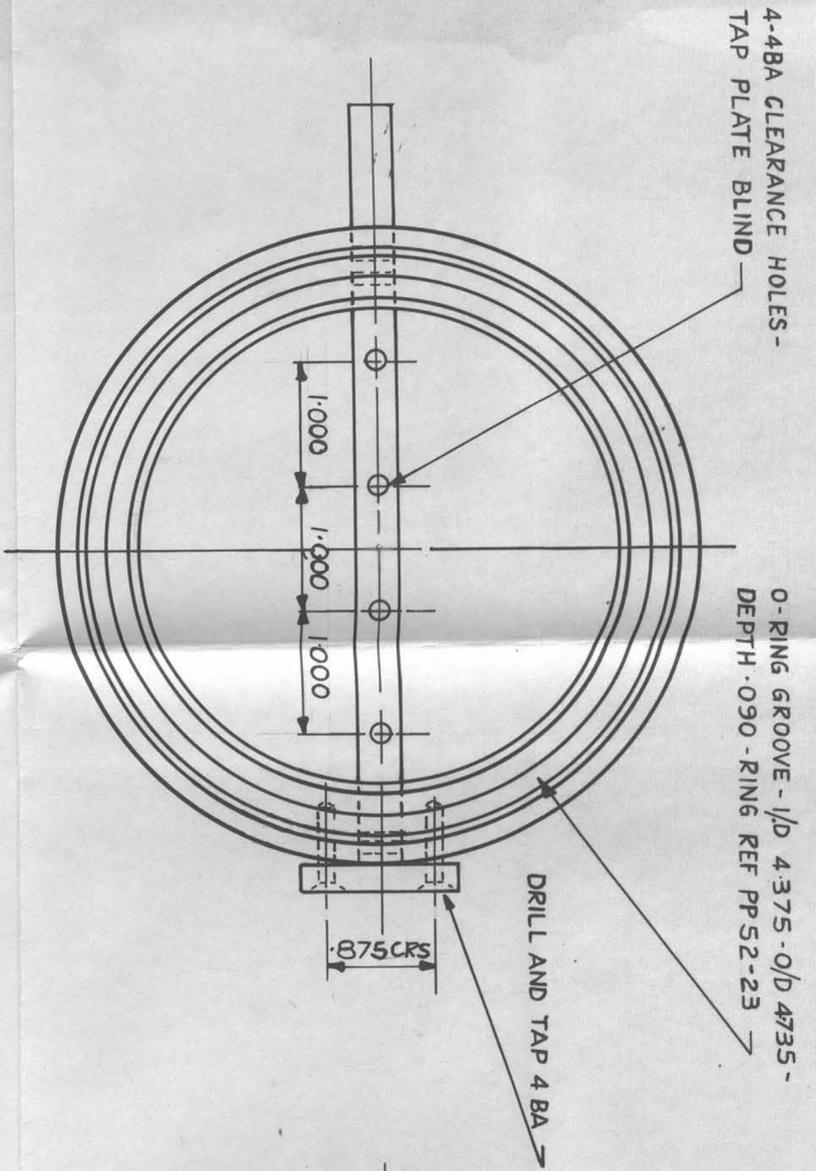


VALVE BODY
 MATERIAL-MILD STEEL
 BOTH TO BE CHROMIUM PLATED AFTER
 FINAL MACHINING

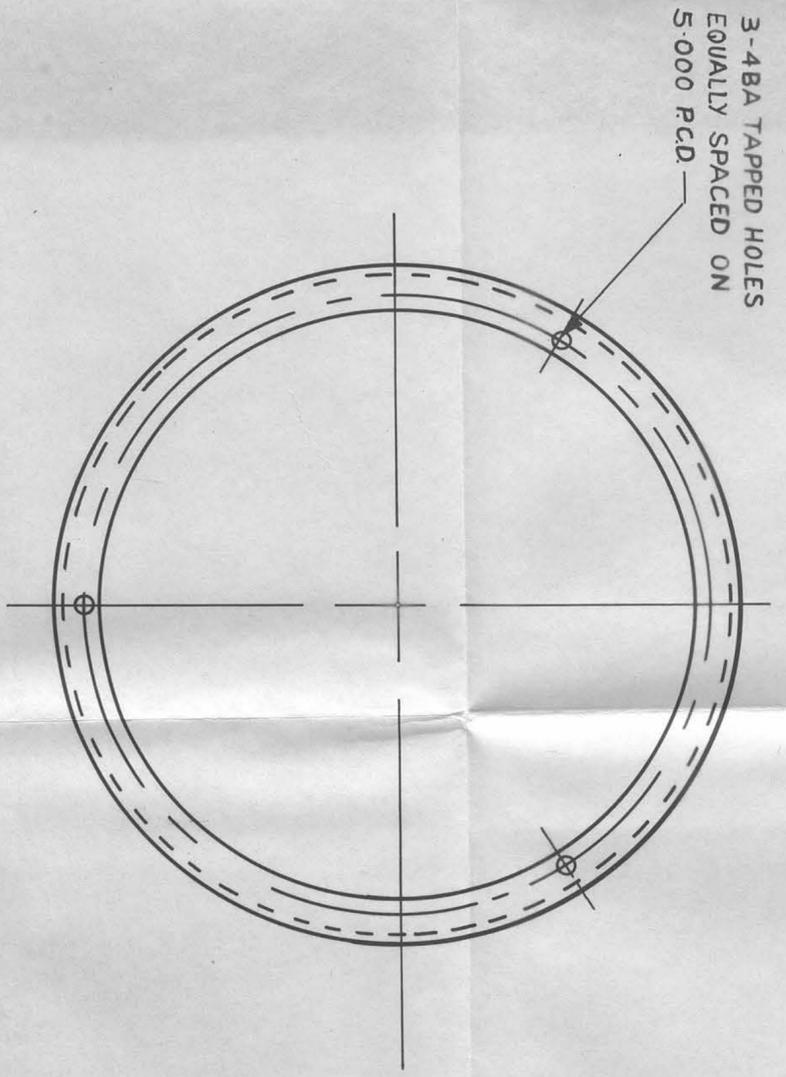
GATE
 MATERIAL-MILD STEEL



CLAMPING RING
 MATERIAL-MILD STEEL

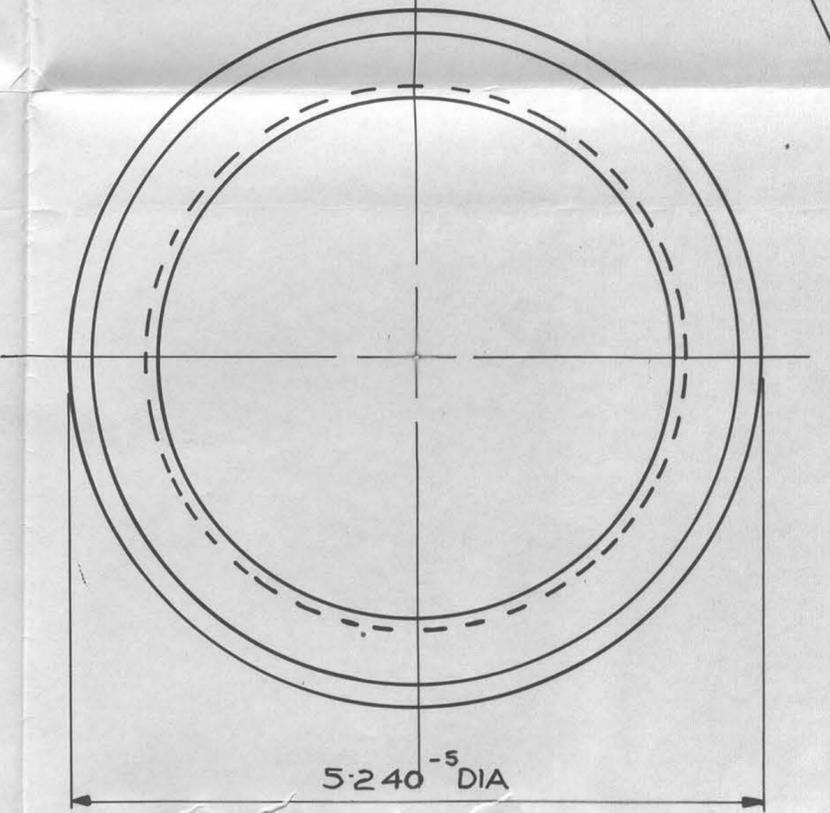
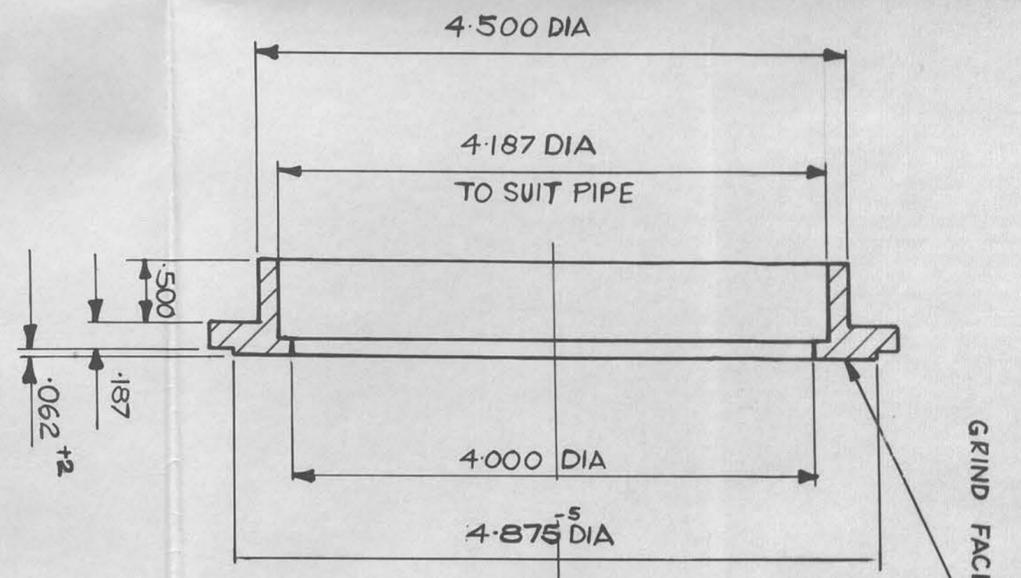
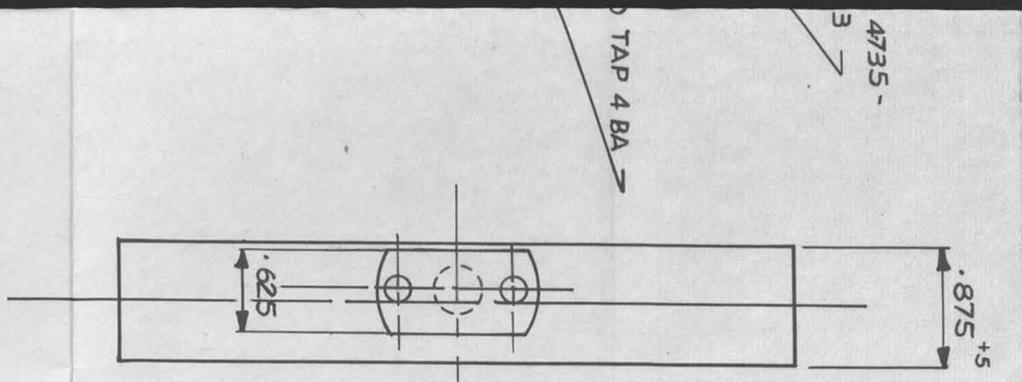


SPINDLE
 MATERIAL-STAINLESS STEEL

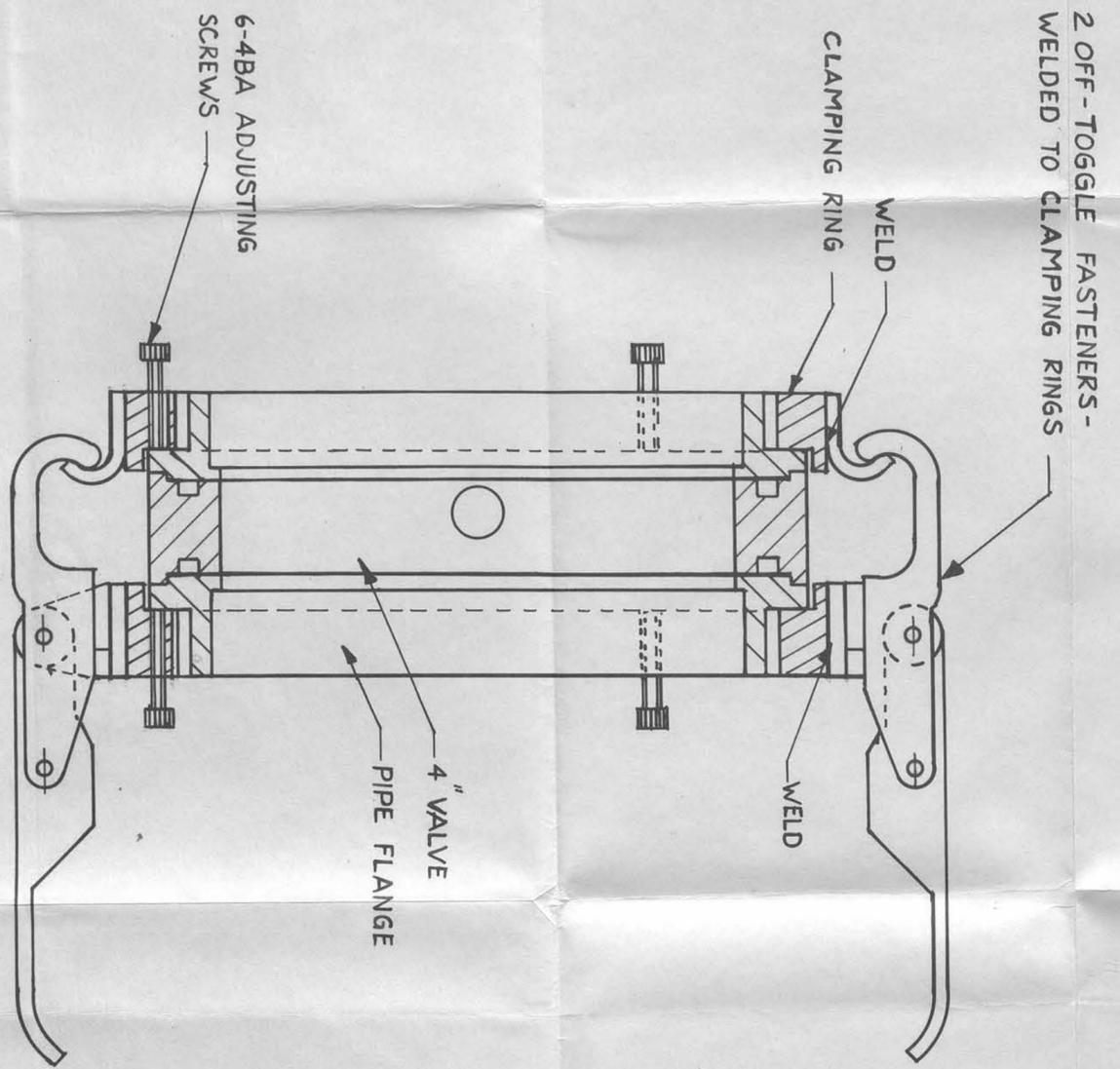


BLANK MATERIAL

4" VACUUM GATE VALVE
 SCALE - FULL SIZE



BLANKING PLUG
MATERIAL - BRASS



PIPE FLANGE
MATERIAL - MILD STEEL

and tapped blind.

A simple multi vee clamping jig permitted the locating flats to be milled on six spindles at a single pass.

The pipe flanges were turned from the same thick wall tube as the valve bodies, and their sealing faces were ground by a tool post grinder before they were removed from the lathe. The toggle clamps were standard heavy duty box fasteners welded to adjustable clamping rings to give a simple positive locking arrangement. Adjusting screws in the clamping ring allow fairly loose tolerances in flange and body thickness. This also had the advantage of permitting remachining of the joint faces and O-ring grooves in the event of damage, thus minimising waste.

Mild steel was used for the entire valve and coupling apart from the valve spindle which was stainless steel ground stock and the spindle blanking plug which was brass. The valve bodies and gates were hard chrome plated to reduce wear on the O-rings and also to prevent corrosion. All thirty-five valves and couplings in the line were tested on a "20th Century" mass spectrometer leak detector and in only three cases were leaks found; these were in spindle seals which were easily rectified. No leakage past the valve plate was detected in any valve on first assembly.

While this standard of tightness was not strictly necessary, the mass spectrometer proved to be a very convenient, quick and re-

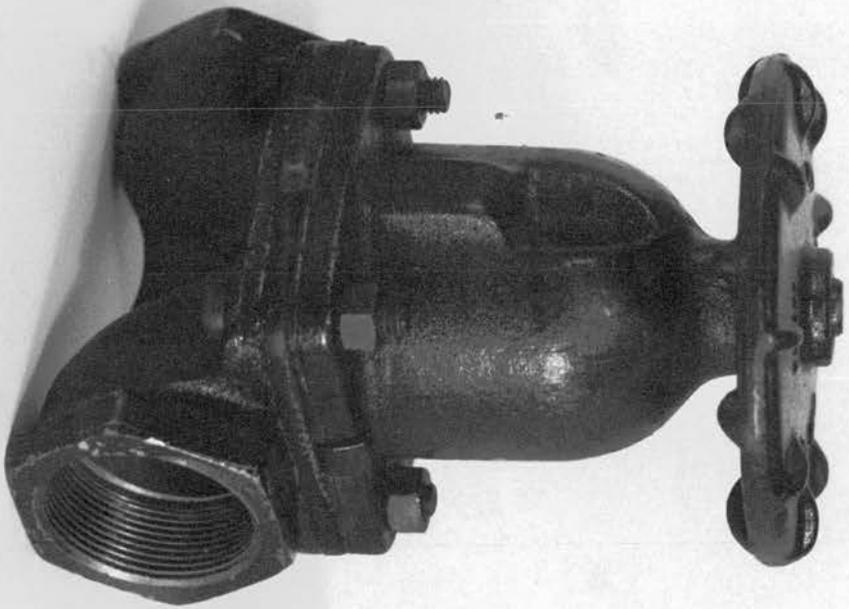
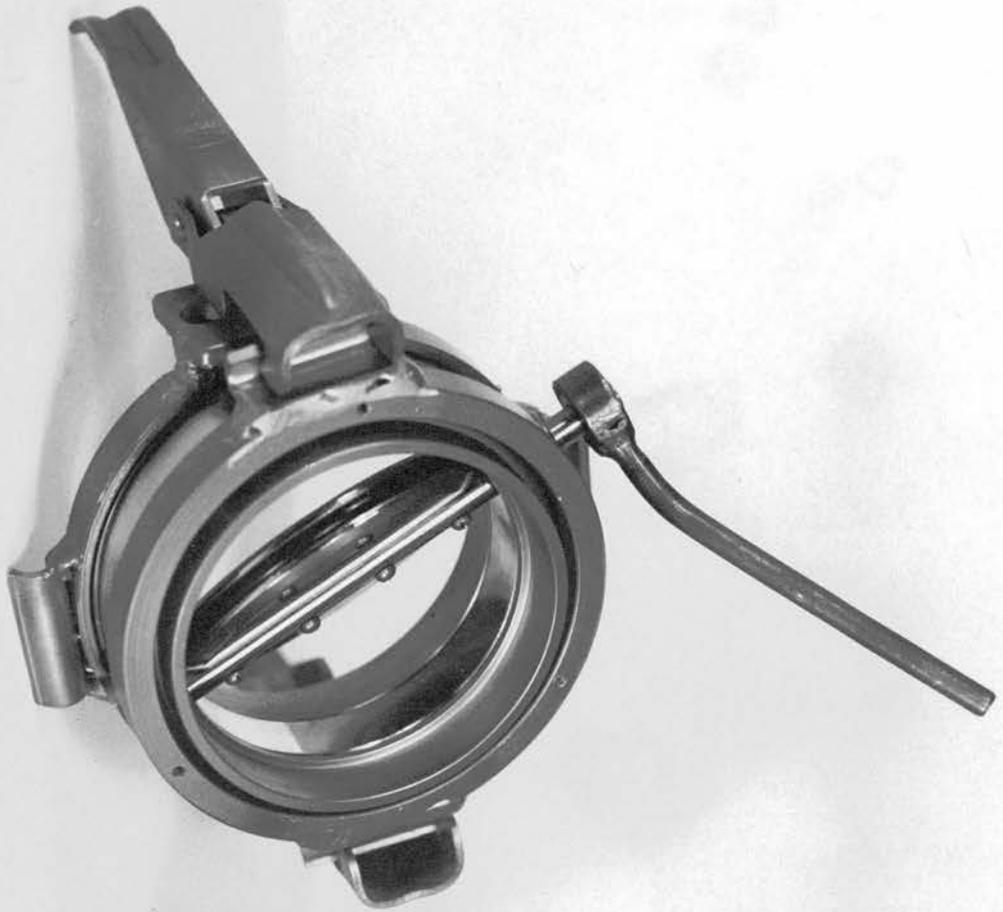


Photo 6.2 The assembled 10 cm valve and coupling compared with a 5 cm bore diaphragm valve.
A 10 cm bore diaphragm valve is some 70% larger than that shown

Photo 6.1 The component parts of the 10 cm bore helium pumping line valve and quick release coupling



Photo 6.3 Section of helium pumping line with coupling valves in position. Two cryostat tap off points are shown (Photos inverted)



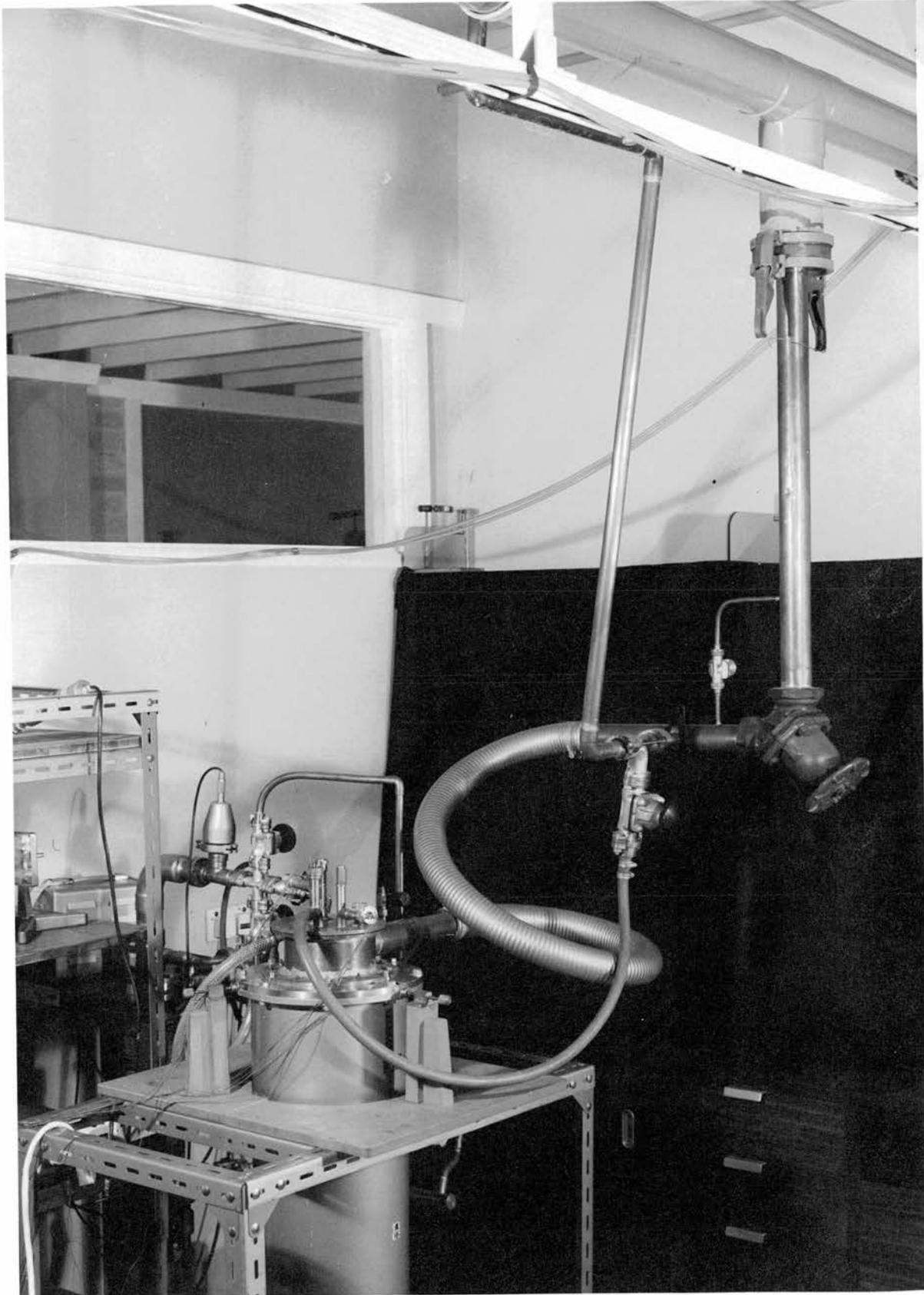


Photo 6.4 A typical cryostat connected to the line using flexible plastic tubing

liable instrument. We were indeed pleased to be able to produce so many of these very simple inexpensive and compact but sophisticated valves with such precision and interchangeability. Plates 1 and 2 show the component parts and the compactness of the complete unit compared with a 2" diaphragm valve, and plate 3 shows a section of pipeline with coupling valves in position.

6.5 Connecting Cryostats to Tap Off Points

The toggle clamp system is used to connect cryostats to tap off tee junctions, a typical installation being shown in plate 4. When not in use these points are closed with standard blanks.

Prevention of vibration either building-borne or pipeline-borne is a serious problem in very low temperature physics. At 10°K or less one is often concerned with energy transfer of a few ergs per minute so that even minute amplitudes at a few hertz coming into the cryostat must be avoided.

Until recently various arrangements of metal bellows on flexible metal tubing have been used to isolate the cryostat from the pumping line. However, large diameter metal bellows are too elastic, and both bellows and metal tubing are very expensive.

A new development is the use of large diameter, up to 4", corrugated P.V.C. tubing reinforced with a semi-rigid P.V.C. spiral inseparably fused to the pliable hose wall. By using a single complete loop of this tubing all vibration from the pipeline can be

eliminated. In the smaller sizes, 1" to 2", end connections may be made by turning ridges about 2 mm high, 3 mm wide and 15 mm apart on short lengths of metal tubing. These short lengths are made a good close fit to the inside of the flexible P.V.C. hose, and metal straps applied round the outside establish a good vacuum seal. With the larger sizes of P.V.C. tubing it is necessary to use cement as well since the inside surface finish is not sufficiently good for it to seal by compression alone. Since only short lengths of such plastic are used, and since the lower limit of required pressure is 10^{-2} mm Hg, the vapour pressure of the plastic is not important.

6.6 Returning Helium Gas from Research Rooms to Liquefaction Area

Since helium gas is expensive, great care is taken to reduce gas losses. When liquid helium is transferred from a storage vessel to a cryostat, a substantial quantity of liquid is evaporated in cooling the apparatus to 4°K, even if it has been precooled to liquid N₂ temperature. From each litre of liquid evaporated 740 litres of gas are evolved and this gas must be recovered. For this purpose a permanent 2.5 cm diameter recovery line was looped around the research rooms and led back to the recovery gas holders in the liquefaction area.

To obtain the most efficient transfer of liquid helium it is important that it should not be transferred too quickly. This is

because the density of the evaporating gas is nearly the same as that of the liquid and a substantial fraction of the incoming liquid can be carried up into the return line if the exhaust stream is moving too fast. Since it is necessary to keep the transfer rate down, a 1" diameter return line is adequate even though the most distant cryostat is 60 metres from the gas holder. Once again thin walled copper domestic water pipe to B.S. 659, was used for economy and ready availability of pipe and fittings. Tap off points are provided each fitted with 2.5 cm commercial diaphragm valves which are closed and wire locked when not connected to a cryostat. Such diaphragm valves, which can easily be seen in plate 3, are convenient for use with helium gas since they avoid the use of troublesome packing glands.

The helium gas returning from the 10 cm diameter helium pumping line poses a different problem, since the pumps can flush a considerable initial quantity of gas in exhausting the full 60 metres of pumping line or when first connected to a nearly filled cryostat with helium boiling at atmospheric pressure. At the worst the pumps would be pushing gas into the 30 metre long exhaust line at their maximum rate of 3000 litres/minute. The maximum acceptable pressure rise in the pump casing is 0.3 atmospheres, otherwise the seals begin to give trouble. Applying a similar calculation to that given in section 6.3 it was found that a 5 cm diameter

pipe was required since this would give a maximum pressure in the pump casing of from 0.1 to 0.15 atmospheres under a flow rate of 6000 litres per minute.

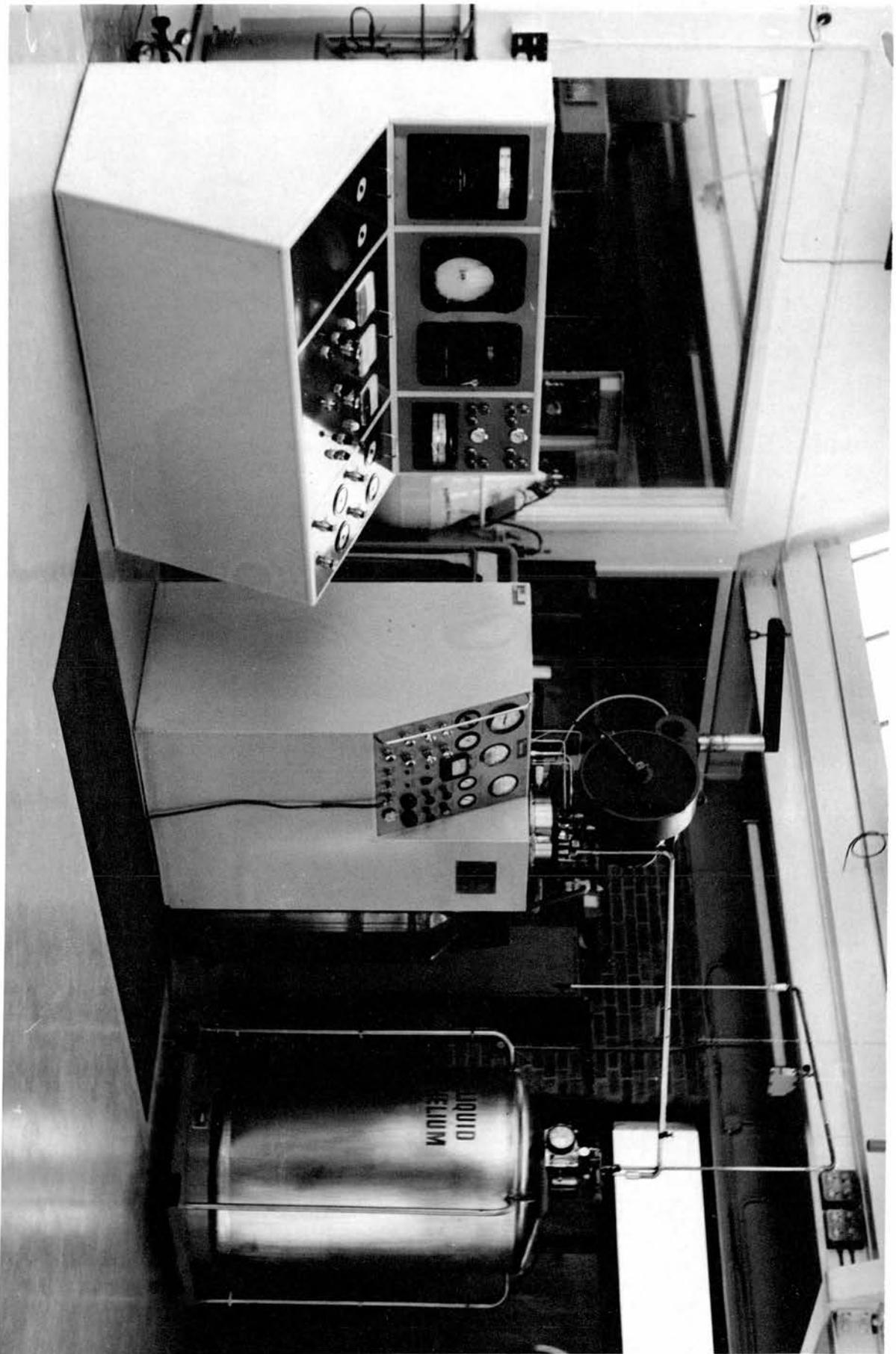


Photo 7.1 The central control panel, the Collins-A.D. Little helium liquefier and the 250 litre liquid helium storage dewar

CHAPTER 7

Conclusion

7.1 The Collins-A. D. Little Helium Liquefier

The Collins-A. D. Little helium liquefier was air freighted from Boston Massachusetts to Prestwick Scotland on the 16th July 1965, and liquefied helium for the first time on 17th August 1965. From that date until August 1968 it ran for a total of 2,050 hours without any serious trouble.

Apart from general failures of incoming power supplies there was only one unplanned shutdown during that time. This was caused by a broken valve rod on No. 2 expansion engine, which was probably due to faulty material since there was no evidence of valve seizure. The only serious criticism of the machine was the very high noise level of the engine crosshead assembly; this made working in the same room with the machine running very tiring and unpleasant.

Early in 1968 A. D. Little developed a new slow speed engine crosshead and engine assembly which we acquired on very favourable terms in return for allowing A.D.L. to use the crosshead on our liquefier for demonstration purposes during the L.T. 11 Conference.

Apart from virtually silent operation there are a number of other advantages in the new design. There is a reduction in speed from 600 r.p.m. to 150 r.p.m. The pistons of the new engine are made of a laminated synthetic resin-bonded plastic instead of

nitrided steel. The running clearance between piston and cylinder is larger than before, with consequent greater tolerance of gas impurities. The earlier small diameter connecting rod in tension is replaced by, effectively, a very long piston of uniform diameter from the cylinder head up to the room temperature seals just below the crankshaft.

Although the valves are still operated by small diameter rods in tension, the general change in design enables the complete valve to be easily and quickly withdrawn for maintenance. This operation can now be performed in five minutes, whereas it took a whole working day with the earlier model.

In the earlier design a hydraulic device absorbed the mechanical work produced by the heat engines, but in the new design this is replaced by an electromagnetic brake with a single variac speed control.

The slow speed engine crosshead assembly has now been in operation for sixteen months and has run for 1,400 hours. We were troubled initially with low production due to the valve leathers becoming loose when the engines were cooled down. After this was cured, the liquefier ran, and continues to run, satisfactorily.

7.2 Helium Compressors

The two helium compressors have now run for more than 3,600 hours and have absorbed more maintenance and repair work than the

liquefier itself. This has included replacement of valves and piston rings. Also, one needle roller bearing from a small end broke up during running, fortunately without damaging any other part of the compressor. No. 1 compressor runs more hours than does No. 2 since it is used for the automatic shut down cycle. It is not possible to use No. 2 compressor for this purpose without a major alteration to the control circuit wiring. The running hours are, however, partially equalised by using No. 2 compressor for circulating helium through the system before a run.

7.3 250 Litre Liquid Helium Dewar

The evaporation rate from the large 250 l helium storage dewar has always been very close to the manufacturer's estimate. We had some difficulty during the first few months with blockages of the capillary tube to the liquid level indicator but after replacing this by a double-walled evacuated dip tube no further difficulty has been encountered.

There is an occasional transient rise in pressure in the vessel above the normal of 6.7 lbs/in² to 10-12 lbs/in². This transient sometimes lasts from 24-48 hours after which the pressure returns to normal. The excess does not appear to be amenable to control; it is annoying but not damaging.

The manufacturers of the vessel have so far been unable to offer any explanation for this transient effect. There is no evidence

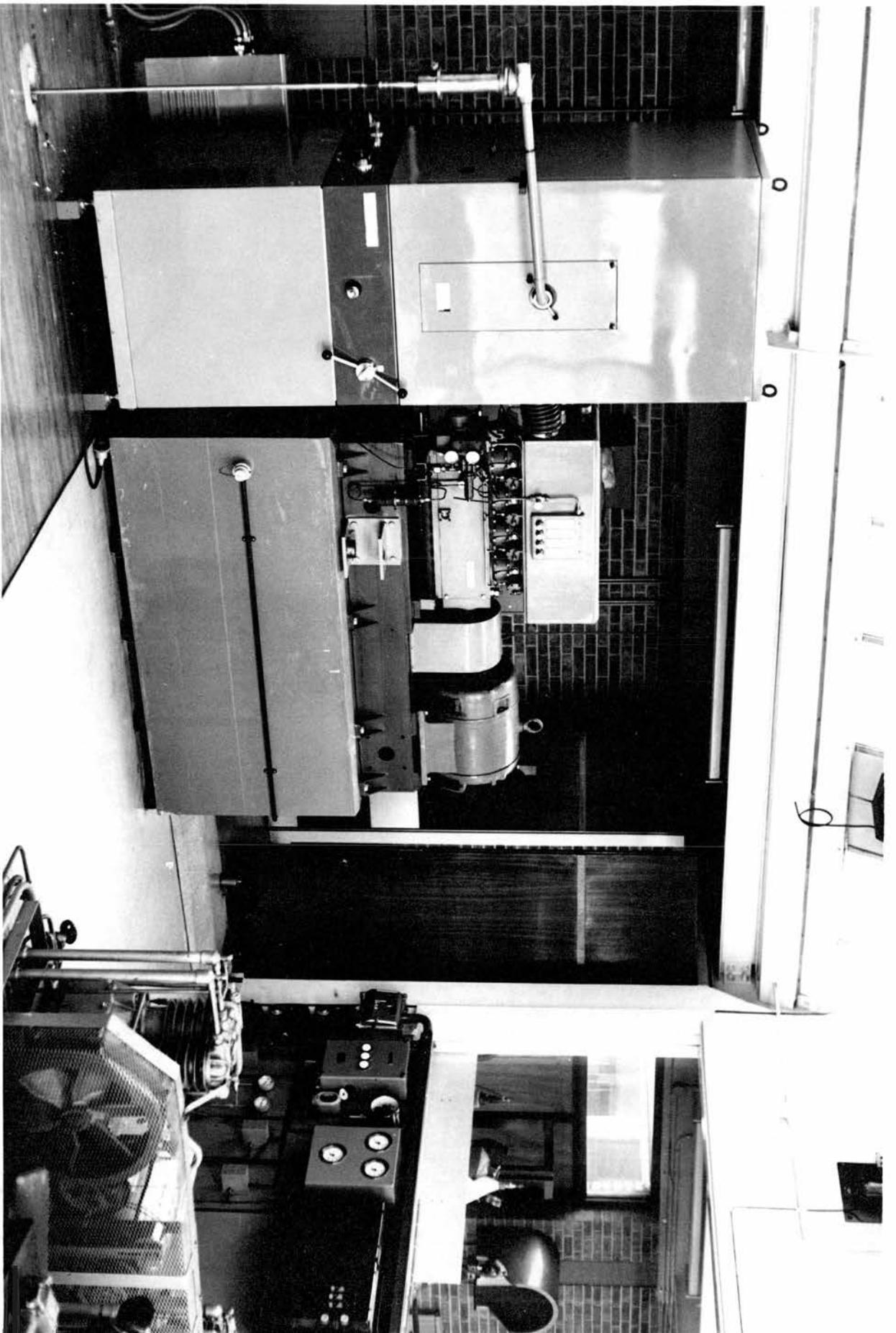
of there being any kind of thermal oscillation in the tubes leading into the dewar. The pressure rise seems to appear only when the liquid has been left undisturbed for a long time so that it may possibly be caused by a stratification effect similar to that which frequently occurs in liquid nitrogen vessels. Since the liquid is a poor conductor of heat, the lower part of the dewar may become superheated while the surface liquid remains at saturation temperature. This is an unstable condition, with the superheated liquid liable to rise through the surface layer at any time and boil more or less violently with consequent pressure rise. Such sudden ebullitions with nitrogen or oxygen are common in low temperature laboratories, but can be quite frightening to the uninitiated.

We hope someday to investigate this pressure surge and to satisfy ourselves as to its cause. Meanwhile, there are always a sufficiently large number of jobs of higher priority to cause us to postpone it.

7.4 Actual Consumption of Helium Liquid and Gas Compared with Prediction

During the twelve months from November 1968 to November 1969 the liquefier ran for a total of 1,377 hours at an average making rate of 5.2 litres per hour. This low average comes about because the machine is normally run through the night without liquid nitrogen precooling. Thus the total production of liquid helium for the

Photo 7.2 The Philips PLN430 nitrogen liquefier showing the transfer tube connection passing through the floor to the liquid nitrogen pump



period was 6,800 litres. This was in spite of a slower expansion in research student places than had been anticipated in 1963-65 when the laboratory was being planned.

During the same production period, 396 m³ of helium gas had to be bought to provide make-up gas due to losses. On the basis of our original estimated loss figure of 10% of all gas liquefied, we would have had to make up 481 m³. Although our recovery rate is gratifying, and indeed is superior to most cryogenic laboratories, we are still seeking ways to improve it because of money; 10 m³ of helium gas costs £27.

7.5 The Philips PLN430 Nitrogen Liquefier

The Philips PLN430 nitrogen liquefier was started up for the first time in September 1967. Between then and December 1969 it ran for a total of 5,188 hours making 130,000 l in all, of liquid nitrogen. It has suffered one major failure.

During the first twelve months of operation the production rate was not up to the manufacturer's specification. In September 1968, Philips service engineers, who were investigating the low rate, applied a vacuum to the base of the separating column which caused it to collapse. A new base had to be supplied from Holland with a long delay. While we were awaiting the replacement, Philips loaned us a CO₂/ice separator which allowed us to continue production, but of liquid air only.

When the column was repaired and replaced, the production rate was brought up to specification, since when only routine maintenance has been necessary.

7.6 Actual Consumption of Liquid Nitrogen Compared with Prediction

The consumption of liquid nitrogen at 1,900 litre per week is a good deal less than the 3,000 l/week which had been estimated originally. This is gratifying; it may be partly due to the increasing use of metal cryostats which have lower nitrogen loss rates than have the old glass cryostats. It has also been helped by the improved evaporation figures from the 25 l transport dewars.

7.7 2,500 Litre Liquid Nitrogen Storage Dewar

The evaporation loss rate from the 2,500 l liquid nitrogen storage dewar is rather less than the manufacturer's estimate, and the vessel itself always behaves in a very docile manner. A simple flow meter was constructed to give an indication of the rate at which gas was being evolved. By this means an early warning is given of any deviation from normal.

The liquid transfer lines into the dewar from the pump and into the pump from the liquefier are insulated with expanded plastic. These are not very efficient and it is our intention to replace them with vacuum insulated lines, when opportunity serves.

7.8 Helium Pumping and Return Lines

We have had no trouble with the helium lines apart from a tendency toward cracking of soft soldered joints in the 100 mm diameter pumping line. This was partly due to poor aligning of the steel flanges in the initial assembly, and also to the line not being uniformly supported along its length, which caused occasional heavy bending loads to be imposed on the flanges.

7.9 Summary and Conclusion

With the exception of the helium gas central monitoring and control panel on which some wiring and plumbing remains to be done, the cryogenic installation is complete. Naturally, improvements have been made and will continue to be made, but basically the installation is as it was planned on paper before bricks and mortar were laid, and it has proved to be economical, smooth in operation and easily workable by two technicians supervised, when necessary, by the author.

It would certainly have been daunting if we had not had, prior to this large plant, a decade and a half of experience of putting together hand-made nitrogen, hydrogen and helium liquefiers, gas handling systems and cryostats, and making them work. That experience was sometimes painful, infrequently hazardous but quite rewarding. It gave us complete confidence in being able to put together a sophisticated system ten times as large and thirty times as expensive,

and make it work in a docile and economical fashion.

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