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Design and construction of a liquid nitrogen cryostat for use in a swimming pool type reactor

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DESIGN AND CONSTRUCTION OF A LIQUID NITROGEN CRYOSTAT FOR USE IN A SWIMMING POOL TYPE REACTOR

BY

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THESIS

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ABSTRACT

The continued expansion of the nuclear reactor facility and the nuclear engineering program dictates the need that a liquid nitrogen cooled cryostat be available at the reactor. This unit would be available for use in graduate and undergraduate laboratories and for graduate research.

A liquid nitrogen cooled cryostat capable of maintaining a sample between 77°K to 80°K for an extended period of time in or adjacent to the core of the reactor was designed, constructed, and successfully operated.

Recommendations were made for possible modifications to expand the versatility of the system.

ACKNOWLEDGEMENTS

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I. INTRODUCTION

Many radiation effects investigations performed in nuclear reactors are greatly improved if they may be performed at cryogenic temperatures. Radiation induced defects in metals tend to anneal themselves at a rate proportional to the temperature of the sample. Irradiating at low temperatures tends to freeze (pin) these defects in the lattice structure.

Many experiments require the use of a constant low temperature environment to minimize the adverse effects of heat; this is especially true when electronic components are subjected to radiation. In addition, the combination of radiation and low temperature approximates the situation that will be present in a nuclear propelled space vehicle.

A. Design Goals

The problem therefore, was to design and construct a device capable of maintaining a constant low temperature environment adjacent to the core of the reactor. This device had to be safe, reliable in operation, contain provisions for cold sample removal, and be completely compatible with the reactor and its operation. An additional desired design feature included automatic operation backed up by audio and visual annunciation in the event of any serious abnormality in its operation.

This project was selected because of the need for such a device at this institution. The resulting cryostat with its supporting sub-systems and instrumentation has completely satisfied the initial design criteria and it is hoped that other institutions will adopt this system as an instrument to further expand low temperature irradiation investigations.

B. Review of Literature

The need for developing cryogenic apparatus for radiation damage research was generally realized in 1947 or before. A solution was approached from a number of directions. The first facility which was available for comprehensive work was that used in 1948 by the North American Aviation, Inc. group at the 60 inch cyclotron in the Crocker Laboratory of the University of California. In 1952 the University of Illinois installed cryogenic facilities for use with their cyclotron.(18)

With the advent of research reactors in the mid 1950's, researchers realized the advantages of irradiating samples in the core of a reactor at sub-normal temperatures. In 1956 the Oak Ridge National Laboratory constructed the first liquid nitrogen cooled cryostat which was installed in their graphite reactor. This device was an open-mouthed aluminum Dewar, 22 feet in length which was filled with liquid nitrogen. This cryostat exploded after three days

of operation in the reactor and was immediately followed by an intense odor of ozone in the vicinity.

In the same year, another group at the Oak Ridge National Laboratory constructed a liquid helium cooled cryostat for use in the same reactor. This device, called the Hole No. 12 Cryostat, exploded during its initial run after the vacuum line leading to the sample tube ruptured. Several hours elapsed before repair could be made, and air froze in the sample chamber. The cryostat exploded during warmup.

It is believed that in each incident a large fraction of the oxygen which condensed in the cryostat was converted to ozone by the ionizing radiation of the reactor. The exact nature of the explosion is not known, but some possibilities are the sudden decomposition of ozone, a nitrogen-ozone reaction, or the reaction of ozone with small quantities of foreign organic material.[4]

These are the only serious malfunctions that have occurred in cryostats being used in reactors and in neither case was any damage done to the reactor.

The Brookhaven National Laboratory was the next institution to provide for irradiating samples in liquid nitrogen. The device consisted of a small Dewar flask, similar in size and construction to a common vacuum bottle which was laid on its side in one of the horizontal access holes to their graphite moderated reactor. The device was

filled periodically with commercial liquid nitrogen through a tube which led outside the reactor. The size of the sample to be irradiated was somewhat limited since the depth of liquid nitrogen in the device was about 1 inch.(16)

In 1957 a somewhat complex cryostat was constructed at the Oak Ridge National Laboratory. High purity nitrogen gas was condensed in a heat exchanger and pumped to the sample chamber in the core of the reactor. The coils of the heat exchanger of this closed loop were submerged in a commercial liquid nitrogen bath which was open to the atmosphere. The nitrogen in the closed loop was maintained at 15 psig and its boiling point was therefore 85°K, while the commercial liquid nitrogen at 0 psig was at 78°K. Additional gaseous nitrogen was supplied to the closed loop and condensed until the loop was completely filled with liquid nitrogen.

The vacuum insulated irradiation flask of this system was constructed of 2S (1100) aluminum. To accomplish cold sample removal the reactor was shut down and the induced activity of the flask was allowed to decay. The sample was maintained at 80°K during this period.(18)

In 1958 the researchers at the BEPO reactor at Harwell, Berkshire, England reported on the construction of a liquid nitrogen cryostat. This device was a vertical aluminum Dewar, 24 feet in length. As opposed to the device con-

structed at Oak Ridge, this unit was not open to the atmosphere. Commercial liquid nitrogen was used as the coolant in the sample chamber. A high speed suction pump from a milking machine was connected to the sample chamber and operated periodically to draw liquid nitrogen into the cryostat from a supply vessel. Cold sample removal was easily accomplished by removing a specimen can containing the sample and liquid nitrogen from the vertical sample chamber. The only complication encountered with this device was that under irradiation, oxides of nitrogen were formed as a sludge in the bottom of the cryostat. This sludge made it difficult to remove the specimen can, and on warming up the cryostat, the nitrogen oxides present attacked the aluminum vacuum jacket. This problem was alleviated by purging the cryostat with dry nitrogen or by starting the cryostat again immediately after warming up.(20)

In 1959 a similar cryostat was constructed at the BR I reactor at Mol, Belgium.(7)

In 1960 a complex liquid nitrogen cryostat was constructed by Bochirol, Doulot, and Weil at the Centre d'Etudes Nucleaires de Grenoble, Grenoble, France. This device employed pure nitrogen gas in the sample chamber adjacent to the core. A tube above the sample chamber contained commercial liquid nitrogen at atmospheric pressure. The nitrogen in the sample chamber was maintained at 80 mm Hg pressure to assure liquification on the con-

denser tube. The pure liquified nitrogen then dripped back into the sample chamber thus maintaining a relatively constant level of liquid nitrogen in the sample chamber. Cold sample removal was a complicated performance which was accomplished only after the induced activity of the cryostat had been allowed to decay for two days.

The cryostat to be described in this paper is identical in concept to the French device. The cryostat itself however, is considerably simpler in construction and operation.

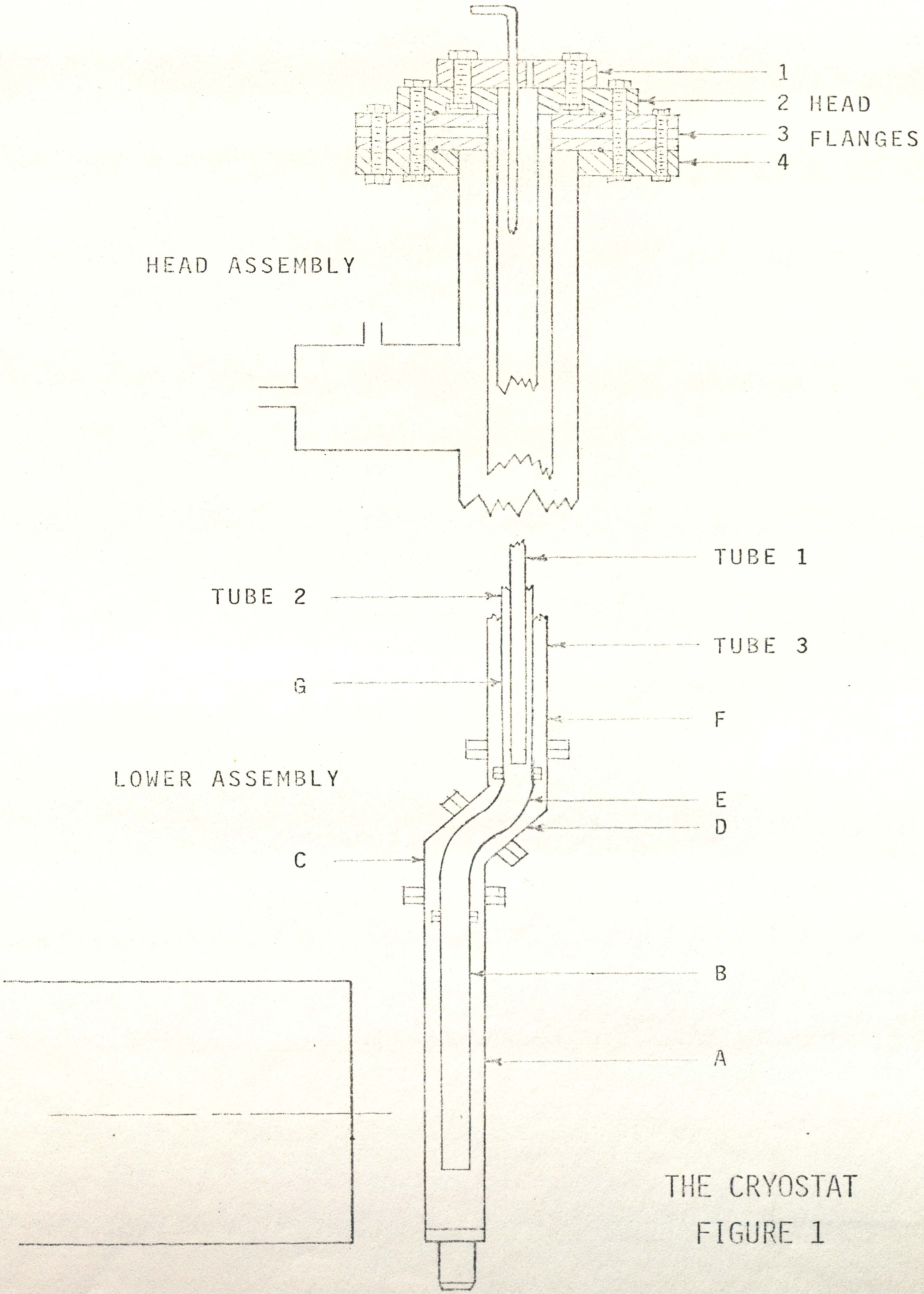
II. CRYOSTAT DESIGN

A few workable designs for liquid nitrogen cryostats have been available for several years. The basic drawback to these designs was their inherent complexity and high cost or their potentially hazardous method of operation. A greatly simplified design was needed to bring a safe and reliable device of this nature to within financial reach of almost any institution of higher learning.

Several unsuccessful approaches to a workable design were attempted before the basic design of this cryostat emerged. The design was then refined many times before the final plans were formalized. Even then some minor modifications were required when difficulties arose in the actual construction of the device.

The principle upon which the cryostat operates is an employment of the very basic concept of a heat exchanger and the gas laws. Figure 1 shows the basic configuration of the cryostat.

When in operation the volume between tubes 2 and 3 is evacuated to establish a vacuum insulation between the sample chamber and the warm pool water. Tube 2 contains high purity gaseous nitrogen and tube 1 contains commercial liquid nitrogen. The gaseous nitrogen (GN_2) in tube 2 is maintained slightly above atmospheric pressure. The liquid nitrogen (LN_2) in tube 1 is at atmospheric pressure and is



THE CRYOSTAT
FIGURE 1

vented to the atmosphere through the small (1/4 inch) hole in head flange 1. Thus, the heat in tube 2 is conducted to tube 1 where the heat is picked up by the LN_2. The heat of vaporization of the LN_2 then removes the heat from the system. The GN_2 in tube 2, being at the same temperature as tube 1 but at a slightly higher pressure, will condense on the outside of tube 1 and drip into the sample chamber (item B on Fig. 1).

Before the reactor is brought to criticality the sample chamber is purged several times to remove oxygen and back filled with high purity nitrogen gas. The liquid nitrogen in tube 1 contains 1-2% oxygen. Fractionization will cause this figure to increase proportionally with the length of a given run. No danger is present however, since this concentration is sufficiently removed from the core to prevent ionization of the oxygen by the radiation from the reactor. Calculations show the approximate amount of liquid nitrogen that would be required to operate the cryostat in the core of the reactor for 8 hours with the reactor at 200 KW is 25 liters.

A. Material Selection

1. Metals

A variety of metals were considered as possible construction materials. A high rate of heat transfer was desired for tube 1 and silver or copper would be the best choices from this standpoint. However, cost would make

the first prohibitive and the large cross section of the second would eliminate the possibility of its use. Aluminum is inexpensive, has a low cross section, has a high thermal conductivity, and therefore was chosen.

For tube 2 stainless steel was considered. This material would have allowed maximum effective use of the somewhat limited available volume since the portion of the cryostat in the core was to occupy a volume no greater than that of a fuel element. The shiny surfaces of the polished stainless steel would also have greatly reduced the thermal radiation heat loss, as opposed to those metals that oxidize rapidly. However, after irradiation, this metal would have been much too radioactive to be safely handled. Aluminum would require a greater thickness for the same mechanical properties and would have a higher thermal radiation heat loss, but would be compatible with the radiation; therefore, aluminum was chosen.

Since tube 3 was to be in contact with the pool water, a non-corrosive metal was required. For this reason and for those listed above, stainless steel was considered. It was again rejected from a health physics standpoint. Aluminum is essentially non-corrosive and as before is compatible with radiation and therefore was chosen.

For all of the above uses, iron or steel was given brief consideration because of its low cost. This material

would also have become sufficiently radioactive to prevent safe handling after irradiation. In addition, steel becomes very brittle at temperatures below -20°F.

Aluminum was chosen for all the tubes and flanges of the cryostat. The use of aluminum also eliminated the possibility of an electrolytic effect in the core of the reactor since most of the materials in the core are made of aluminum. In addition, some of the physical properties of aluminum improve at lower temperatures.(2) The particular alloys chosen were selected more for a low cross section and short half-life than for their mechanical properties. The order of preference of the useable alloys was 1100, 6063, and 5052. It was found that tubes are not made of 1100 Al except on special order. It was therefore decided to use 6063 aluminum with a heat treatment of T6 for tubes 2 and 3. Tube 1 was selected to be of 5052 aluminum. This selection was based upon the availability in the desired size. The flanges and the tail piece were made of 1100.

The alloying elements and their maximum percentages in these three types of aluminum are listed below.(1)

alloy	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti
1100	1.0	Si+Fe	0.20	0.05	-	-	0.10	-
5052	0.44	Si+Fe	0.10	0.10	2.8	0.35	0.10	-
6063	0.6	0.35	0.10	0.10	0.9	0.10	0.10	0.10

Aluminum and normal impurities constitute the remainder.

2. Insulation

During normal reactor operation the temperature of the water in the pool is about 105°F. The temperature inside the sample chamber was planned to be about -320°F. If tube 3 should become extremely cold, several problems could arise. Ice forming on the outside and thus in contact with a fuel element would set up a large temperature gradient in that fuel element which could cause some warping. This cold temperature around the outside of the cryostat would also produce a positive reactivity since the reactor has an inherent negative temperature coefficient.

In actuality, the cooling capacity of the cryostat is considerably less than that required to produce a coat of ice around it while operating in the warm pool.

It was obvious that some type of insulation was required to greatly reduce the thermal conductivity between tubes 2 and 3 in order to improve liquid nitrogen economy, to minimize temperature fluctuations in the sample chamber and to greatly reduce the possibility of any damage to the core of the reactor. Nearly every type of thermal insulation was given some consideration. Materials such as cork, asbestos, and glass wool were quickly eliminated because of their high thermal conductivity.

Solid foams such as polystyrene (Styrofoam) were attractive since they could be easily shaped and had a lower coefficient of thermal conductivity. The major

drawback to using a solid foam was that its insulating properties were inferior to those of ordinary permeable materials such as powder.

The combination of vacuum and powders such as perlite or "Sil-O-Cel" (Johns Manville) for thermal insulation is very attractive. Evacuating these powders to 10^{-2}mm Hg is equivalent to an evacuated space at 10^{-4}mm Hg. Outgassing such a system however, can be rather time consuming if the pumping speed of the system is low. Perlite has quite good shielding properties. This can be favorable or unfavorable depending upon the particular situation. Packing the space between tubes 2 and 3 with either of these powders would have been difficult and the operation would have to be performed everytime the cryostat was disassembled.

Every cryostat constructed thus far has used only a vacuum for insulating purposes. Leak detection and elimination is usually a long and tedious affair especially when many aluminum welds have been made. After all the leaks have been stopped, a vacuum provides the best insulation with the least amount of difficulty and at a reasonable cost.

The selection of appropriate flange seals was complicated by the combination of the three hostile environments; i.e., a hard vacuum, very cold temperature, and

irradiation by all types and energies of particles. A great variety of seals were available, ranging in cost from 20 cents each for a simple rubber "O" ring to $150 each for sophisticated silver plated seals of a complex design. Since a seal either works or does not work, the term "best" as applied to function has no meaning. The best seal then is that device which gives the most use per dollar.

After much consideration, it was concluded that simple "O" rings made of either Teflon or Buna-N would give the best service. Teflon is far superior for all the uses in the cryostat. Buna-N however, is a suitable temporary substitute and is more readily available.

The three sets of flanges on the lower portion of tube 3 use two concentric Teflon "O" rings to eliminate the possibility of water leaking into the evacuated area between tubes 2 and 3. This was done since even the smallest of water leaks would be enough to destroy the vacuum. In addition, the water vapor that would be absorbed in the vacuum pump oil would considerably reduce the ability of that pump to establish a vacuum. The water vapor destroys the lubricating properties of the oil and the sealing vanes in the pump would be severely damaged.

Eight stainless steel bolts are used to fasten each set of flanges. This allows a great deal of freedom in the desired orientation between the various parts of the cryostat and also allows a large force to be applied to the

"O" rings. Stainless steel bolts were employed because of their great strength and non-corrosive properties. Ordinary steel bolts would rust and thus contaminate the pool. Aluminum bolts are not strong enough and are prone to swelling and galling.

The flanges of tube 2 employ only one Teflon "O" ring between each set of flanges. Eight stainless steel bolts are used to fasten the flanges together. This allows the same freedom of orientation as tube 3 and permits a high loading force on the "O" ring. A leak between tubes 2 and 3 is serious only if it hampers the establishment of a suitable insulating vacuum. The nitrogen vapors, warm or cold, will have no serious effect upon the operation of the vacuum or diffusion pumps.

B. Construction

The actual machining, welding, and construction of the cryostat produced some difficulties. The flanges in particular, being made of 1100 aluminum, were extremely soft and very difficult to machine. The lightest touch by the cutting tool or by the shavings from the flange itself was sufficient to mar the sealing surface. Great care was exercised to minimize this damage.

A Heli-arc system was used to weld the various aluminum pieces together. Tube 1 and part F of tube 3 (Fig.1.) are each made of two pieces of tubing. Butt welding was allowable on part F because of its thickness. Excessive

run-through occurred in this weld but it was impractical to attempt to remove it due to limited accessability.

A thin collar was machined to slip over the joint of the two pieces of tube 1 and the collar was then welded to the upper and lower pieces. This method was dictated since the wall of the tube was much too thin to allow butt welding.

The affixing of the four lower flanges of tube 2 to that tube and the joining of tube 1 to head flange 2 were made with the use of epoxy. This method was required due to insufficient space to allow welding.

The remaining flanges were welded to their appropriate tubes by welding the inner-most portion of the non-sealing surface of the flange to the tube. This produced some warping in the flanges. As anticipated however, the warp was such that after cooling, the sealing surface of the flange was convex. This in fact improved the loading ability of the flange bolts.

Each "O" ring groove was machined as a 90° "V" cut in the lower flange of each pair of flanges. The sealing surface of the upper flange was allowed to remain flat. This method eliminates the need for tight tolerances on the concentricity of the groove.

Elbows C and D were made by cutting four pieces of tubing at a 45° angle and butt welding the angled cuts together. The run-through on both welds was removed to

permit the passage of offset E through the two elbows. Extreme care was exercised in the cutting and welding of the various parts of elbows C and D and offset E since any misalignment would be magnified at the ends of items A and B.

To construct offset E, an 18 inch length of the tube was solution heat treated to reduce the heat treatment from T6 to T1. The tube was then filled with molten lead which was allowed to solidify. After bending the tube to an angle of 90° in a hydraulic press, it was placed in a furnace to melt out the lead. The tube was then cut in the knee of the bend and one leg rotated 180° about the center line of the tube. A one inch piece of the tube was placed between and butt welded to each of the legs.

III. SUPPORTING EQUIPMENT

It was decided early in the design of the cryostat to use tried and proven, commercially available equipment whenever possible to maximize dependability and to minimize down time when malfunctions occur. Figure 2 is a schematic diagram of the supporting equipment and its relationship to the cryostat.

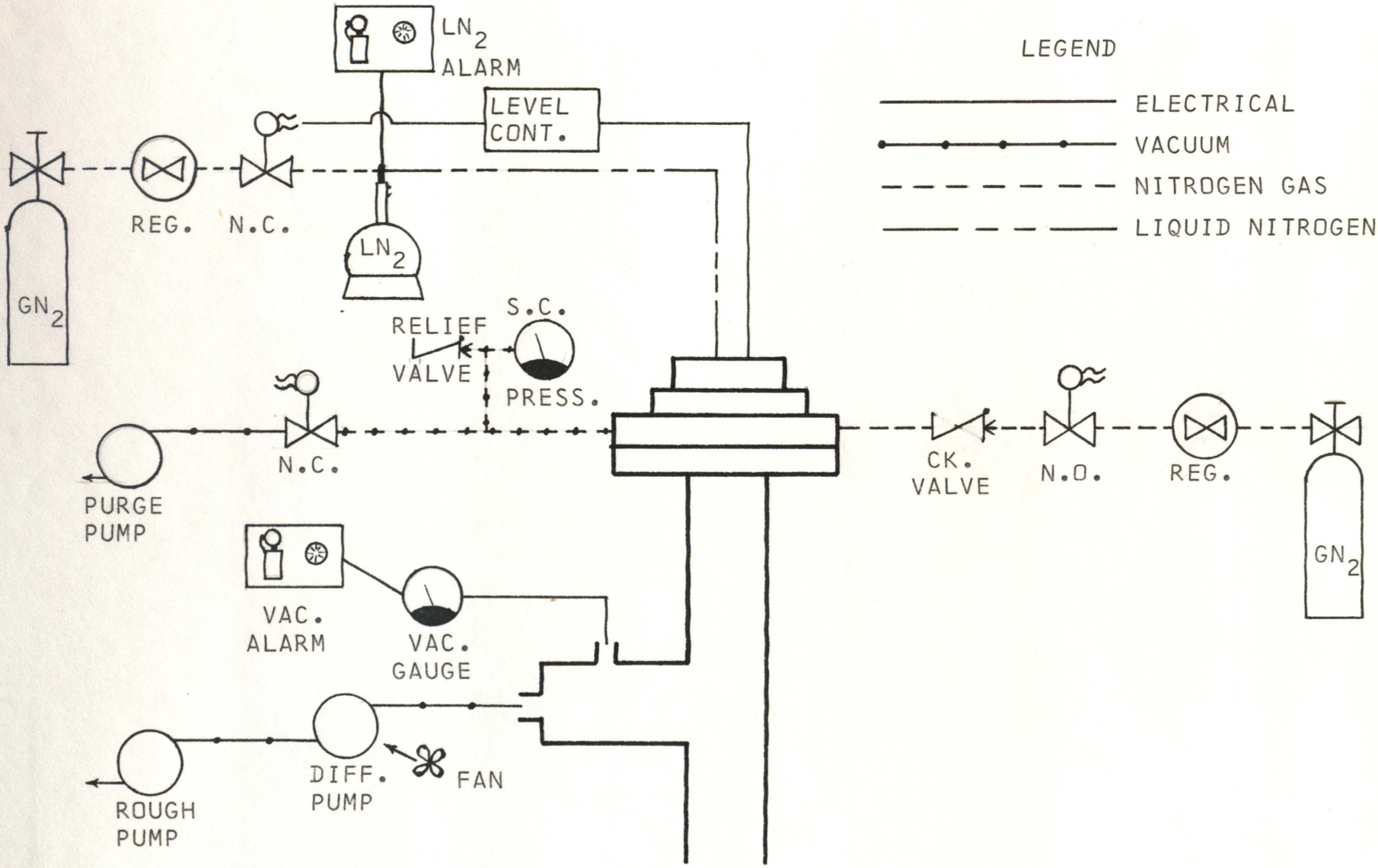
A. Mechanical Equipment

1. Vacuum System

The vacuum system consists of a Welch Duo Seal 1400 roughing pump and a Consolidated Vacuum Corporation type VMF air-cooled diffusion pump. The roughing pump has a free air speed of 21 liters per minute and the diffusion pump has a speed of 600 liters per minute at 1×10^{-4} torr. This combination yields an ultimate vacuum of 1×10^{-6} torr which is more than satisfactory for the purpose intended. A pumping time of at least five hours is required to establish a vacuum of 20 microns (20×10^{-3} torr) which is recommended as the maximum safe operating pressure.

2. Purge and GN_2 Supply System

The purging system consists of a Welch 1400 vacuum pump connected through an Automatic Switch Company model 8262 B90 (normally closed), 2-way solenoid valve to head flange 3. As shown in Figure 1, the horizontal holes in head flange 3 lead to tube 2.



SCHEMATIC OF THE CRYOSTAT
AND SUPPORTING SYSTEMS
FIGURE 2

The nitrogen gas supply system consists of a standard tank of nitrogen connected to a pressure regulator and an Automatic Switch Company model 8262 B34 (normally open), 2-way solenoid valve. High pressure reinforced rubber tubing conducts the gas from the supply tank to head flange 3.

A check valve is installed at the head on the GN_2 line to prevent a back flow of radioactive gas should there be a rupture in the supply line. This check valve is set to allow gas into the sample chamber when a pressure differential of one (1) psi exists and is leak tight to a back pressure of 3,000 psig.

A light-weight vinyl tube carries the GN_2 from the head to the bottom of the sample chamber. This was done in an effort to maximize the effectiveness of the purging system.

A relief valve set to open at 35 psig is installed on the sample chamber side of the purge exhaust line to relieve the pressure in the sample chamber if it should become excessive.

3. LN_2 Supply System

The liquid nitrogen supply system includes a 50 liter stainless steel, Hofman Dewar flask designed for a maximum working pressure of 50 psig, a Johns Technology LN_2 transfer line kit, a Johns Technology Model L-20 Cryo-Miser liquid nitrogen level control, and a tank of gaseous nitrogen with a regulator.

A signal from the max-min level sensors in tube 1 operates the solenoid which allows nitrogen gas to pressurize the Dewar flask. The liquid nitrogen is then forced into tube 1 by the pressure differential. A 10 psig relief valve is included in the Dewar head assembly to prevent over pressurizing the system.

B. Instrumentation

The control and instrumentation of the cryostat and its supporting systems has been kept as simple and reliable as possible. The layout of the control panel is shown in Figure 3 and the wiring diagram for the control panel is drawn in Figure 4.

The Cooke Vacuum Gauge (Model 1300) is of the cold cathode discharge type. This type of gauge, though found lacking in accuracy, is well-known for its simplicity and ruggedness. Since the purpose of the vacuum in the cryostat is to provide sufficient insulation between tubes 2 and 3, only a rough indication of the quality of the vacuum is required.

A Cenco Model 94035 pressure gauge is connected to the purge outlet line in parallel with the relief valve. This gauge is of the positive-negative type reading continuously from 30 inches Hg vacuum to 30 psig and gives an indication of the pressure condition in the sample chamber at all times.

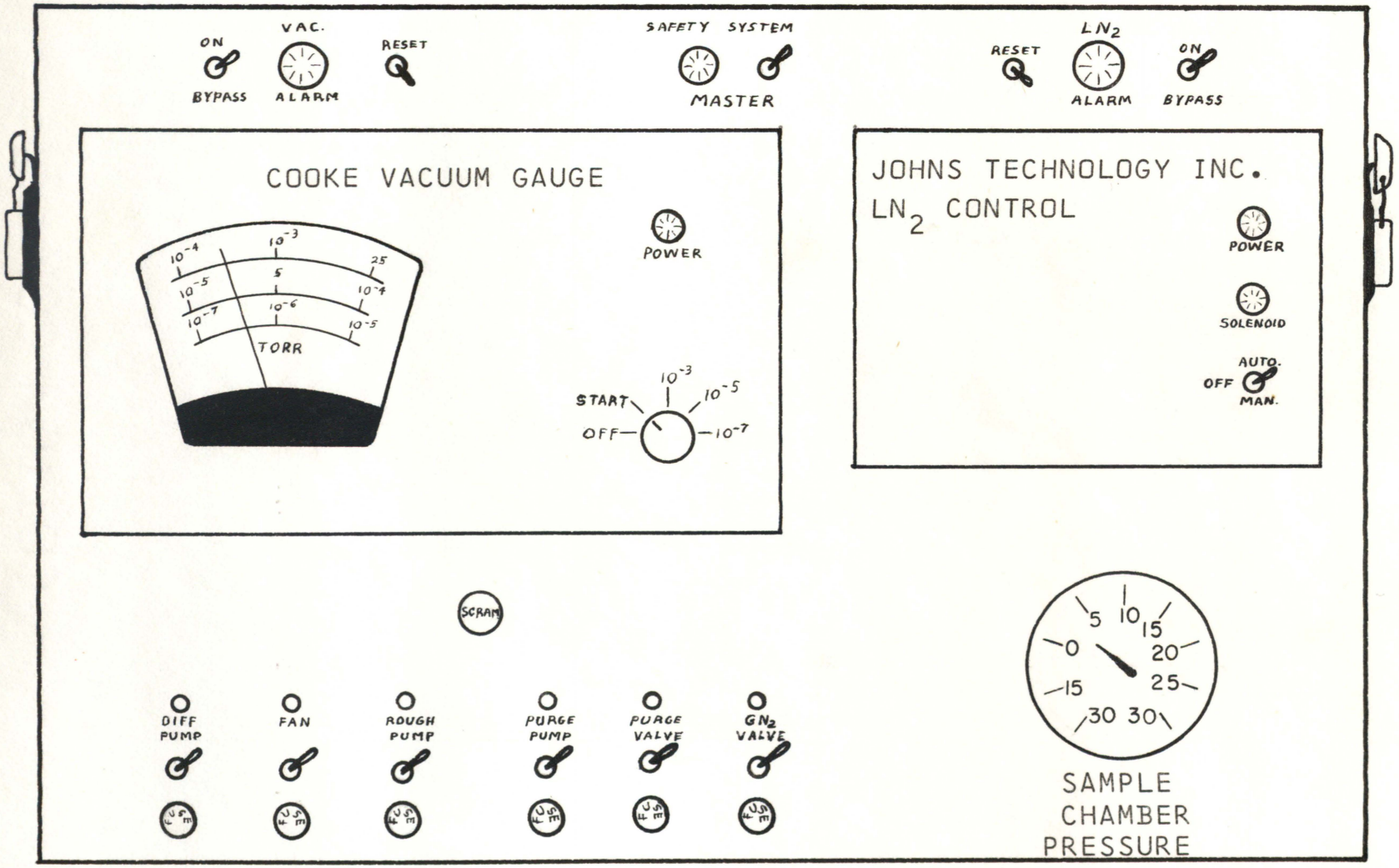
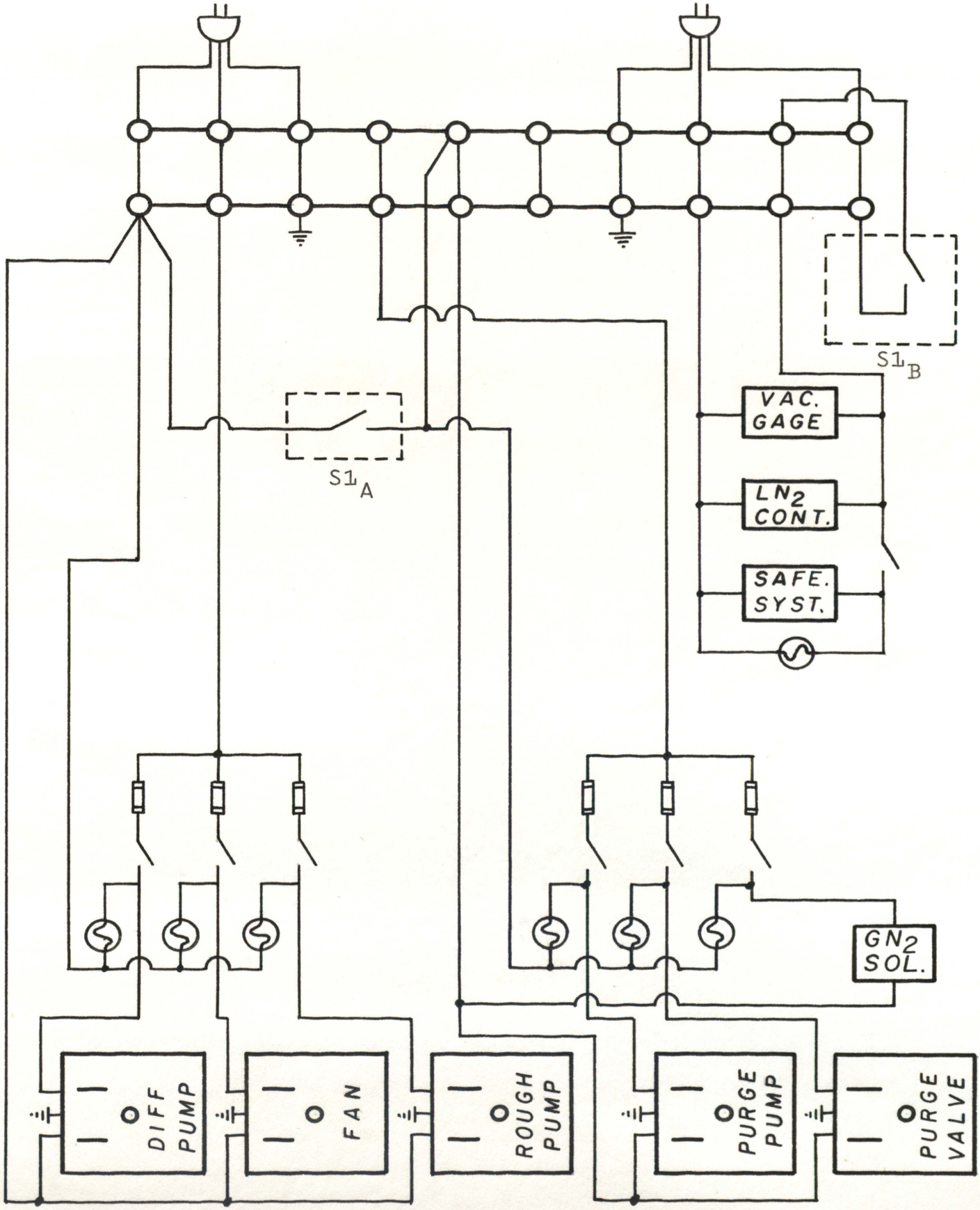


FIGURE 3 CONTROL PANEL

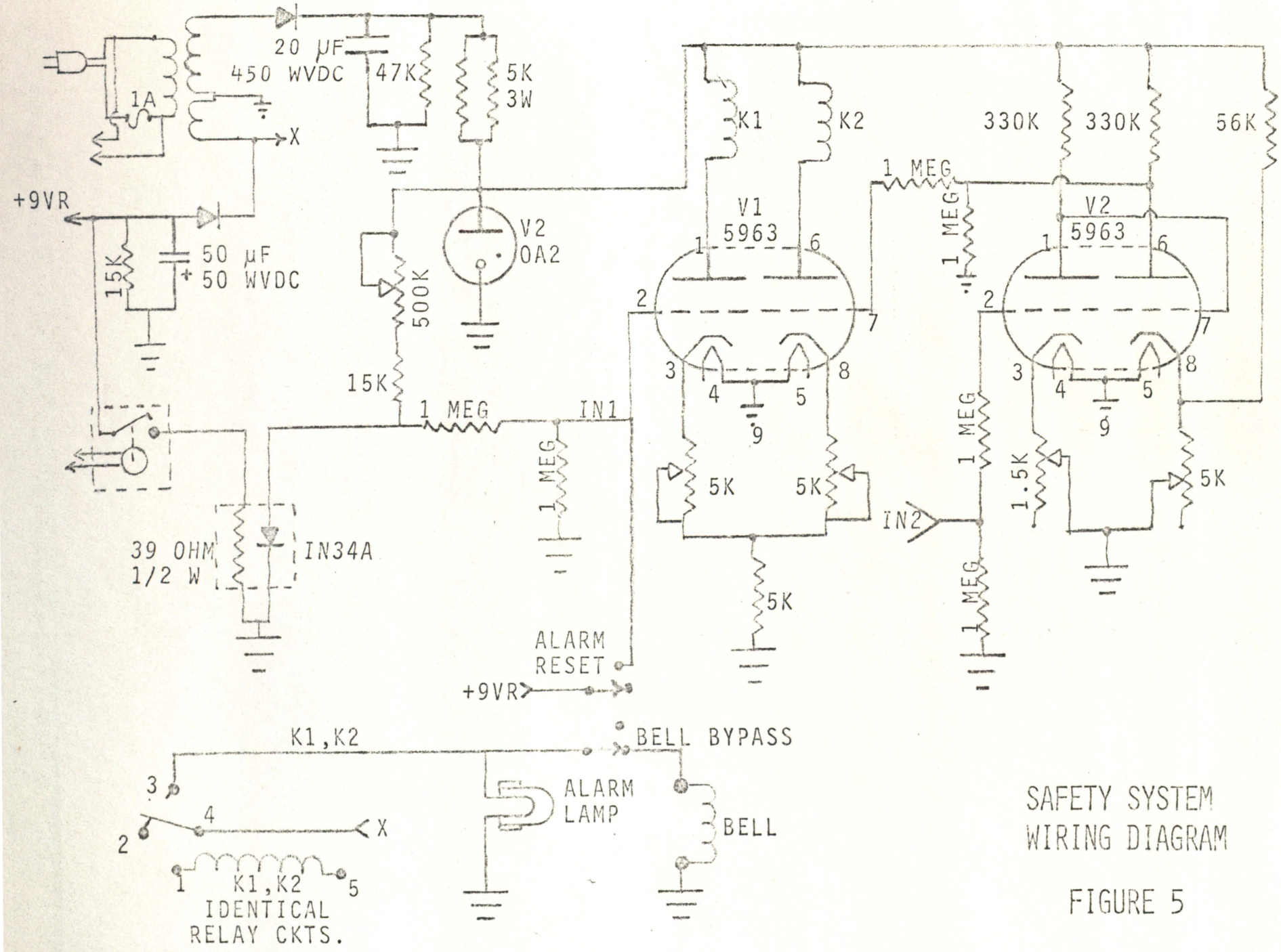


CONTROL PANEL WIRING DIAGRAM

FIGURE 4

A special electronic safety system was designed for use with the cryostat to provide audio and visual annunciation in the event of a malfunction in the operation of the cryostat. The Cooke Vacuum Gauge has an output for a 100 mV recorder. This signal is fed into an amplifier and applied through a potentiometer to a relay. When the vacuum in the cryostat raises above a specified pressure, the relay will be tripped causing a bell and a light on the control panel to function. A bypass switch is provided to silence the bell while the situation is being corrected. The light will stay on until the situation is corrected and the relay is reset by the appropriate switch on the control panel. The pressure at which the vacuum alarm is actuated can be controlled by adjusting the potentiometer.

While the cryostat is in operation, it is important to maintain liquid nitrogen in tube 1 to prevent a temperature or pressure rise in the sample chamber. For this reason a level sensing device has been constructed to indicate when the level of liquid nitrogen in the Dewar flask falls below 10% (5 liters). The basic concept for this system was obtained from a simple device suggested by Hyman, Sheppard and Spinka.[13] The original device would only operate a voltmeter. The concept is that a diode is periodically heated by a resistor. When the diode and resistor are immersed in a cryogenic liquid the heat from the re-



SAFETY SYSTEM
WIRING DIAGRAM

FIGURE 5

sistor is carried away by that liquid. When the level of the liquid drops below the sensor, the resistor heats the diode thus changing the potential across the diode. This change in potential is used to drive a relay for the LN_2 alarm system. When this relay is tripped, a light and a bell on the control panel are actuated. As with the vacuum alarm system, a switch is provided to by-pass the bell while the situation is being corrected. As before the light will remain on until the situation is corrected and the relay is reset by the appropriate switch. A schematic diagram of the complete safety system is drawn in Figure 5.

IV. CALCULATIONS AND EXPERIMENTS

A. Calculations

Before any experiment may be placed in the core of the reactor, an approximate value for its reactivity worth must be obtained. Several methods for approximating this value are available. In the case of the cryostat, the reactivity worth was approximated by relating the aluminum to the known effect of cadmium and by relating the volume displaced by the aluminum, vacuum, and nitrogen to the void coefficient. The known values were obtained from the UMR Reactor Technical Specification. The execution of these calculations is elementary Nuclear Engineering and will not be included in this report. The calculations indicated that the reactivity worth of the cryostat would be approximately -0.18% ΔK/K when placed adjacent to the core. Experiment No. 6 was performed to obtain the actual value of the reactivity worth of the cryostat.

For health physics purposes it was desired to know the expected activity of the nitrogen in the sample chamber. The most prominent reaction was determined to be,

$$^{14}N(n,p)\ ^{14}C$$

The activity of the irradiated nitrogen was calculated to be 23.6 microcuries after operation in the core at 200 KW for 8 hours.

It was desired to know the explosive containment ability of the cryostat. The "TNT Containment Law for Real

Vessels" was derived by Wise and Proctor of the United States Naval Ordnance Laboratory while investigating the response of the Enrico Fermi Reactor to TNT simulated nuclear accidents. The equation as given in reference 23 is;

$$A = 0.1563\ \epsilon_u\ w^{0.85}\ (3.41 + 0.117\ R_i/h_o)$$
$$B = (R_e^2 - R_i^2)^{1.85}$$
$$C = 10^5\ (2\sigma_y + \sigma_u + \sigma_u\epsilon_u)^{-1}$$
$$D = (1.47 + 0.0373 R_i/h_o)^{0.15}\ R_i^{0.15}$$
$$(W_R)_{max} = \left[\frac{A \times B}{C \times D}\right]^{0.811} \qquad (9.4)$$

where the nomenclature and units are,

W . . . . charge weight (TNT or pentolite), lb

w . . . . weight density of vessel material, lb/ft^3

R_i . . . . initial internal radius of vessel, ft

R_e . . . . initial external radius of vessel, ft

h_o . . . . initial wall thickness of vessel, ft

ϵ . . . . permissible strain of vessel material, in/in

ϵ_u . . . . ultimate strain of vessel material, in/in

σ_t . . . . true stress, psi

σ_y . . . . yield stress of vessel material, psi

σ_u . . . . ultimate stress of vessel material, psi

All ingredients of the Containment Law are in terms of conventional materials and properties. Values for 6063 T6 schedule 40 aluminum pipe were used in this equation to determine the containment ability of tube 2 and tube 3.

The results indicated that tube 2 could contain an explosion equal in magnitude to 1.95×10^{-4} pounds of TNT and that tube 3 could contain an explosion equivalent to 7.56×10^{-4} pounds of TNT. No provisions are made in the equation for the length of the vessel and dimensional analysis indicates that the units on $(W_R)_{max}$ are actually pounds per foot rather than pounds as indicated in the report. Therefore, the calculated values were multiplied by the lengths of the respective tubes to obtain the corrected values of 0.0049 pounds of TNT for tube 2 and 0.0204 pounds of TNT for tube 3.

B. Experiments

The nature of the experiments to be performed on the cryostat were in four distinct catagories. The first phase which included experiments 1 through 4, served as a general check on the operation and safety of the cryostat. The second phase, covered by experiment 5, proved that the cryostat could operate in the pool. Phase three was to determine the reactivity worth of the cryostat. This was achieved in experiment 6. The final phase was to operate the cryostat in the core of the reactor. This was accomplished in experiments 7 and 8.

The purpose, data, and results of the individual experiments are listed below. In each case the conditions existing at time zero are that; 1) an insulating vacuum

of less than 20 microns has been established, and 2) tube 1 has been filled with liquid nitrogen and was operating on automatic. Timing of the experiment started when gaseous nitrogen was admitted into the sample chamber.

EXPERIMENT NO. 1

Purpose: To perform a general check on the operation of the cryostat.

Data: None

Results: An insulating vacuum of better than 1 micron was established and the sample chamber was effectively purged. The liquid nitrogen admitted into tube 1 would not flow to the bottom of the tube. This was caused by the fact that the LN_2 was admitted into tube 1 through a diffuser installed near the top of the tube. The warm air in the tube vaporized the liquid nitrogen causing the major portion of it to be forced up and out the vent hole in head flange 1. No cooling was experienced in the sample chamber.

The experiment was terminated and the situation was corrected by removing the diffuser on the end of the short stainless steel inlet tube for the LN_2 and replaced with a 21 foot polyethylene tube which conducted the LN_2 to the bottom of tube 1. This was accomplished before experiment 2 was performed.

EXPERIMENT NO. 2

Purpose: The same as Experiment 1.

Data:

Time (Minutes)	S.C. Temp. (millivolts)	(^{o}F)	Vacuum (Microns)
0	+1.035	+79^{o}	1.0
5	-4.5	-230	X X
10	-5.450	-318	X X
15	-5.434	-316	X X
20	-5.465	-319	X X

Results: As expected, the temperature in the sample chamber remained at room temperature as long as that tube remained evacuated, regardless of the level of liquid nitrogen in tube 1. When the GN_2 was admitted to the sample chamber, the temperature dropped rapidly. For this experiment the pressure of the GN_2 in the sample chamber was maintained at 0.0 psig.

As indicated by the loss of insulating vacuum, a leak was present in tube 2. After 10 minutes the temperature varied from -319 to -316^{o}F depending upon the level of the LN_2 in tube 1.

EXPERIMENT NO. 3

Purpose: During the review of this project by the Hazards Committee it was suggested that since the condensed liquid nitrogen in the sample chamber was so near its boiling point, a heating load such as that imposed by the β and γ radiation from the reactor might cause a sudden pressure build up in the sample chamber. The purpose of this

experiment was to impose a heat load on the sample chamber by filling the insulating vacuum with gaseous nitrogen. Heat from the atmosphere would thus be conducted to tube 2.

Data: None

Results: The experiment was not performed due to erratic readings on the vacuum gauge. The malfunction was traced to foreign particles in the vacuum gauge cathode discharge tube which resulted in arcing within the tube. The situation was corrected by cleaning the tube according to the manufacturers' directions.

EXPERIMENT NO. 4

Purpose: Same as Experiment No. 3.

Data:

Time (Min.)	S.C. Press (psig)	S.C. Temp (mV)	S.C. Temp (°F)	Pressure (psig)
0	-14.5	+0.85	+71	5.5 Micron
5	4	-4.50	-230	25 Micron
10	6	-5.40	-312	+35 psig
15	0	-5.45	-319	35 "
20	0	-5.40	-312	35 "
25	4	-5.40	-312	35 "
30	10	-5.35	-307	35 "
35	4	-5.42	-315	35 "
40	26	-5.28	-300	35 "
45	10	-3.90	-186	35 "
50	5	-3.20	-139	35 "
55	3	-2.80	-114	35 "
60	2	-2.00	-69	35 "

Results: Gaseous nitrogen was admitted to the vacuum chamber at 7 minutes. There was no serious pressure change

in the sample chamber during the first half hour. A heavy coat of frost was present on the exterior of tube 3. It was evident then that during normal operation no serious pressure rise would be anticipated.

At 35 minutes the LN_2 supply to tube 1 was disconnected to determine what effect this might have. As the level of LN_2 dropped in tube 1, the pressure in the sample chamber increased. At 42 minutes the sample chamber pressure exceeded 30 psig. To prevent a rupture in tube 2, the purge pump was turned on and the purge valve opened to reduce the pressure in the sample chamber. At 43 minutes the pressure stabilized at 20 psig, after which the purge valve was closed and the pump turned off. The pressure continued to drop slowly until it became stable at 51 minutes.

The results of this experiment were as expected. The Dewar flask which is the supply vessel for the LN_2 is equipped with a level sensor to notify the cryostat operator when the supply is low.

EXPERIMENT NO. 5

Purpose: To operate the cryostat in the reactor pool.

Data:

Time (Min.)	S.C. Press (psig)	S.C. Temp (mV)	S.C. Temp (^{o}F)	Vacuum (Microns)
0	-14.5	+1.30	+91	0.7
5	12	-5.40	-312	10
10	7	-5.32	-304	15
15	10	-5.35	-307	18
20	8	-5.40	-312	21
25	7	-5.35	-307	24

Results: As can be seen from the data, the cryostat operated successfully with the exception that a slight leak was present in tube 2. That leak was later found and repaired.

Undersized Buna N "O" rings were used between the flanges of tube 2 for the first five experiments as a temporary measure. These items were replaced by Teflon "O" rings of the proper size before additional experiments were performed.

EXPERIMENT NO. 6

Purpose: To determine the reactivity worth of the cryostat in core positions A1, B2, and C3.

Data: None

Results: The reactor was operated at 2 watts for this experiment. The reactivity worth of the cryostat was determined by comparing the relative positions of the regulating rod with the cryostat in the core, and removed. The worths of the cryostat in core positions A1 and B2 were found to be negligible. The worth of the

cryostat in position C3 was found to be -0.198% ΔK/K which compared favorably with the approximation of -0.18% ΔK/K discussed in section IV A of this paper.

EXPERIMENT NO. 7

Purpose: To operate the cryostat in position C3 of the core of the reactor with the reactor at 50 KW.

Data:

Time (Min.)	S.C. Press (psig)	S.C. Temp (mV)	S.C. Temp (^{o}F)	Vacuum (Microns)
0	-14.5	+2.5	+141	17
5	15	+1.5	+100	13
10	8	-0.2	+22	13
15	20	-5.4	-312	14
20	15	-5.4	-312	12
25	10	-5.38	-310	11
30	10	-5.3	-302	12
35	3	-5.4	-312	12
40	3	-5.4	-312	12
45	10	-4.8	-255	12
50	15	-4.2	-207	12
55	2	-3.15	-158	13
60	7	-4.3	-214	12
65	12	-5.0	-273	11
70	15	-5.32	-302	10
75	10	-5.38	-310	12
80	8	-5.4	-312	12

Results: The experiment was relatively successful. The slight variation of the vacuum was attributed to the fact that fresh oil was employed in the rough pump and in the diffusion pump. These oils had not been operated long enough to complete their outgassing.

As indicated by the data, a great deal of variation of the pressure and temperature occurred in the sample chamber. This was caused by the great separation between

the max-min LN_2 level controls in tube 1. To correct this situation the separation was changed from 12 feet to about 5 feet and the maximum level indicator was moved to within 4 feet of the head of the cryostat.

EXPERIMENT NO. 8

Purpose: To operate the cryostat in position C3 of the core of the reactor with the reactor at maximum power (200 KW) for two hours.

Data:

Time (Min.)	S.C. Press (psig)	S.C. Temp (mV)	S.C. Temp (^{o}F)	Vacuum (Microns)
0	-14.5	+2.97	158	10
5	20	+1.2	86	10
10	6	-5.4	-312	10
15	10	-5.3	-304	10
20	12	-3.7	-172	10
25	5	-5.4	-312	10
30	8	-5.4	-312	10
35	25	-5.4	-312	10
40	3	-5.4	-312	10
45	3	-5.4	-312	10
50	3	-5.4	-312	10
55	3	-5.4	-312	10
60	3	-5.4	-312	9.5
65	3	-5.4	-312	9.5
70	3	-5.4	-312	10
75	5	-5.4	-312	10
80	4	-5.4	-312	10
85	3	-5.4	-312	10
90	3	-5.4	-312	10
95	3	-5.4	-312	10
100	3	-5.4	-312	10
105	3	-5.4	-312	10
110	3	-5.4	-312	10
115	3	-5.4	-312	10
120	3	-5.4	-312	10

Results: The experiment was successful. During the first 40 minutes of the run, the cryostat operator varied

the pressure in the sample chamber to determine the best operating pressure. A pressure of 5 psig or less was found to be satisfactory.

At 55 minutes the pool temperature became excessive and the power level of the reactor was reduced to 160 KW. The power reduction was required due to the combination of the effects of the negative temperature coefficient and the negative reactivity worth of the cryostat.

The results of this experiment indicated that the cryostat had satisfied all of the original goals and that further experimentation to prove the operational ability of the cryostat would be pointless.

V. CONCLUSIONS

The cryostat described in this paper has satisfied all of the initial requirements. It is safe, relatively simple to operate, will operate automatically, and will maintain a constant cold temperature in the sample chamber with the reactor at any desired power level. The cost of this unit including supporting equipment and labor was on the order of $3000. It is felt that this expenditure is well within the financial reach of most research reactor facilities in the United States. Any institution desiring to duplicate this cryostat may obtain a complete set of detailed shop drawings from the Director of the UMR Reactor at this institution.

It is hoped that this unit and duplications of this unit will do much to advance low temperature irradiation investigations.

VI. RECOMMENDATIONS

Other fluids may be used in the cryostat as primary or secondary coolant mediums. As an example, a slurry of dry ice and acetone in tube 1 with nitrogen or helium in tube 2 would produce a sample chamber temperature of -108°F. In this situation the cooling ability of the cryostat would be greatly reduced since the secondary medium would not condense on the outside of tube 1. To improve this situation the sample chamber could be wrapped on the outside with several layers of super insulation assuming that the insulation was compatible with the reactor and did not seriously reduce the radiation flux.

If at some later time the permissible power level of the reactor is increased, it might be necessary to use gaseous helium in the sample chamber as the secondary coolant rather than nitrogen. Helium is essentially inert to radiation. Although the thermal conductivity of gaseous helium is better than that of nitrogen, the cooling capacity of the cryostat would be reduced since the helium would not condense on the outside of tube 1.

The cryostat could be operated at liquid helium temperature by using gaseous and liquid helium in place of the gaseous and liquid nitrogen. This is not feasible at this time because of the high cost of helium, but may become attractive as the cost and availability of helium improves.

It has been reported[5] that an insulating vacuum of 10^{-2} mm Hg is sufficient insulation where liquid nitrogen economy is of no great importance. If builders of future cryostats can tolerate this additional financial burden, some savings can be realized in selecting aluminum tubes with a slightly reduced wall thickness and by using a thermocouple vacuum gauge rather than a cold cathode discharge tube as employed with the system described in this paper.

At some later date it is possible that a transfer vessel will be required which will keep radioactive samples at liquid nitrogen temperatures while providing satisfactory shielding for personnel. To date, no such device is in existence.

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VITA

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He has received his college education at the University of Missouri at Rolla, where he received a Bachelor of Science Degree in Mechanical Engineering in 1965.

He entered the Graduate School of the University of Missouri at Rolla in June, 1965. He has served as Graduate Assistant from September 1965, to June 1967.

VIII. APPENDICES

APPENDIX A

HAZARD'S SUMMARY REPORT

for the

UNIVERSITY OF MISSOURI AT ROLLA CRYOSTAT

Submitted to

THE REACTOR SAFEGUARDS COMMITTEE

UNIVERSITY OF MISSOURI AT ROLLA

November 1967

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I. THE CRYOSTAT AND ITS SUPPORTING SYSTEMS

A. CRYOSTAT

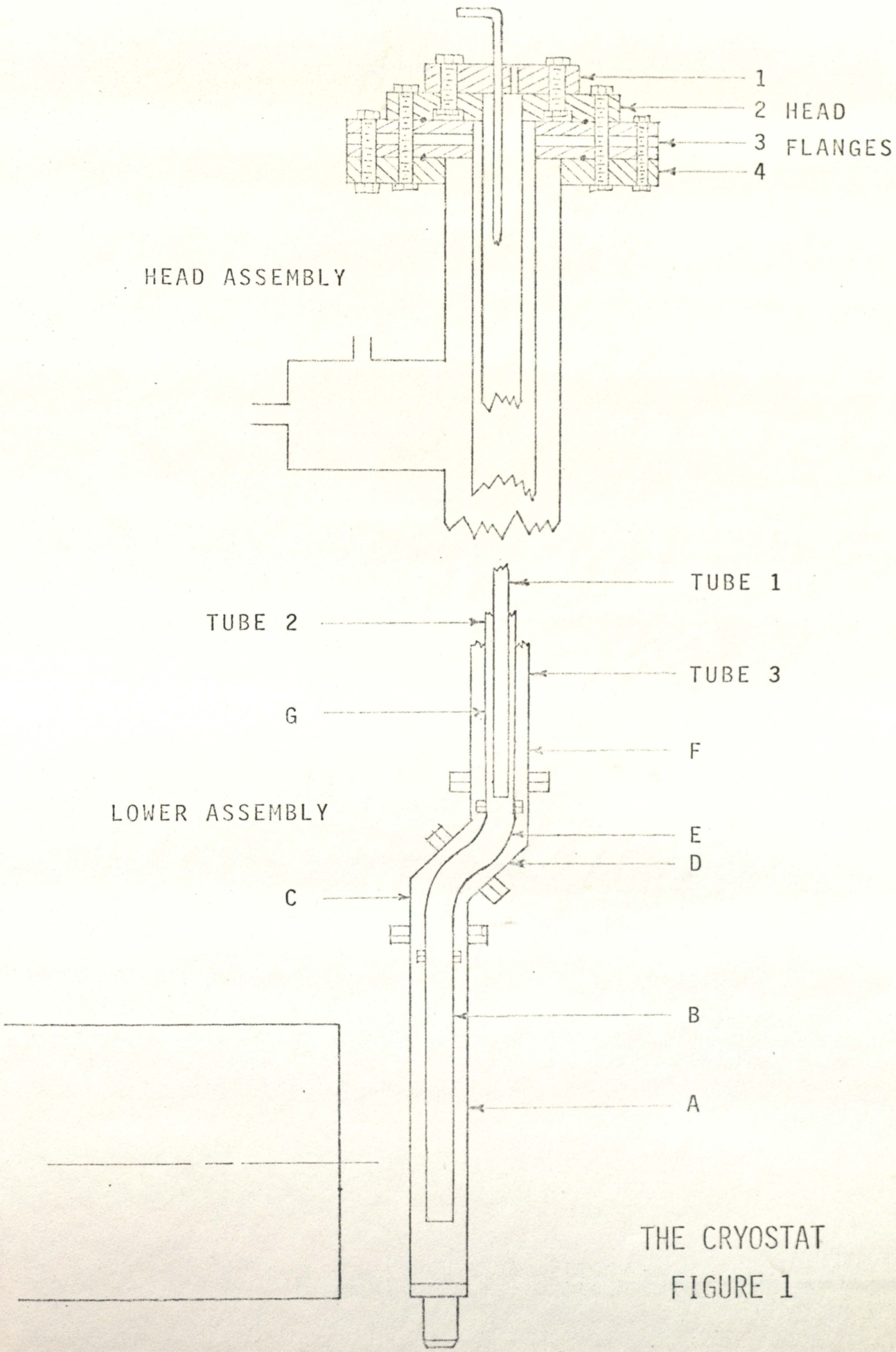
The purpose of the cryostat, designed for use in the UMR reactor, is to establish a low temperature environmental condition within a sample chamber that can be placed in the core of the reactor. Safety of operation, minimal temperature fluctuations and knowledge of the temperature within the sample chamber were the basic considerations in this design. Preliminary experiments with the cryostat indicate satisfactory accomplishment of these goals.

A number of cryostats for use in reactors have been constructed in the past decade with varying degrees of success. The Oak Ridge National Laboratory has the most experience in this field and observed two explosions in cryostats in 1956. Their findings indicated that the cause for these explosions was due to a concentration of liquid or solid oxygen in the presence of ionizing radiation.

In the design of the UMR cryostat, it is felt that sufficient precautions have been taken to reduce, if not completely eliminate, the possibility of such an occurrence.

1. Description

The cryostat itself consists of three (3) concentric aluminum tubes and eleven (11) aluminum flanges. The assembled cryostat is shown in Figure (1).



THE CRYOSTAT

FIGURE 1

The choice of aluminum was based upon its strength, weight, low activation, short half-life and machinability. Tube 2 and tube 3 are 6063 T6 Schedule 40 pipe. Tube 1 is 5052-0 x.049 wall. Nine of the flanges are 1100F Al and two are 5052-0 Al.

All three types of aluminum are compatible with the reactor (i.e., low activation and short half-life.) The particular selection was determined by availability of the various types. The order of preference was 1100, 6063 and 5052.

It was felt that aluminum bolts and nuts of sufficient strength to accomplish the various tasks would contain alloying elements of sufficient type and quantity to make them the radioactive equivalent of stainless steel. Therefore, due to the higher strength and dimensional stability, stainless steel bolts are employed throughout the assembly.

Teflon "O" rings are recommended as the most suitable flange seals although Buna-N "O" rings may be used as a temporary substitute.

Argon arc welding with 1100F aluminum rods was used to weld all flanges on tube 3 and to accomplish the offset in the lower portion of the same tube. Some re-welding was required where leaks were found. Several very small leaks were sealed through the use of Glyptol Paint, a commercial product developed for that purpose and distributed by the Cenco Company.

The flanges on tubes 1 and 2 were affixed to the tube with epoxy. The use of this material was dictated by insufficient space to permit welding. An activation analysis was performed on the epoxy used to insure that it did not become excessively radioactive.

2. Theory

A vacuum established between tubes 2 and 3 serves as thermal insulation to isolate the cold temperature in the sample chamber from the fuel elements of the core. A vacuum of 20 microns is considered to be the minimum allowable whereas a vacuum of 1 micron or less is preferable. The present fore pump and diffusion pump are sufficient to maintain a vacuum of better than 1×10^{-5} torr.

As mentioned previously, it is desired to minimize the quantity of oxygen present in the sample chamber. To accomplish this, a purging sequence is used wherein the sample chamber (tube 2) is pumped to a vacuum of -26 in. Hg or better and back filled with gaseous nitrogen to a pressure of 15 psi. This sequence is carried out four (4) times. The sample chamber is then evacuated and held at a pressure of -26 in. Hg or less while tube 1 is filled with liquid nitrogen. Gaseous nitrogen is then admitted to the sample chamber.

The pressure within the sample chamber may be maintained at the operator's discretion, but must not exceed 15 psig during initial filling and must not exceed 30 psig at any time.

Preliminary experiments outside the pool indicate that the temperature in the sample chamber can be maintained at -319° F -0° F $+ 1^{\circ}$ F if desired. The temperature fluctuation is determined by the distance between the max-min level sensors of the automatic liquid nitrogen level unit.

B. SUPPORTING SYSTEMS

It was decided early in the concept of the cryostat to use tried and proven commercially available equipment whenever possible to maximize dependability and minimize down time when malfunctions occur.

1. Vacuum System

The vacuum system consists of a Welch Duo Seal 1400 fore pump and a C.V.C. (Consolidated Vacuum Corporation) type VMF air cooled diffusion pump. The roughing pump has a free air pumping speed of 21 liters per minute and the diffusion pump has a speed of 600 liters per minute at 1×10^{-4} torr. This combination yields an ultimate vacuum of 1×10^{-6} torr which is more than satisfactory for the purpose intended. Preliminary experiments have indicated that the required pumping time is longer than initially desired but tolerable.

If during operation the pressure should exceed 20 microns, an alarm and indicator on the control panel will notify the operator of the condition.

2. Purge and GN_2 Supply System

The purging system consists of a vacuum pump connected through an ASCO (Automatic Switch Co.) 8262 B90 normally closed,

2-way solenoid valve. The particular vacuum pump employed in this system is of little consequence since a vacuum of -26 in. Hg is all that is required.

The gaseous N_2 supply system consists of a standard tank of nitrogen connected to a pressure regulator and an ASCO 8262 B-34 normally open, 2-way solenoid valve. High pressure reinforced rubber tubing conducts the gas from the supply tank to the head of the cryostat.

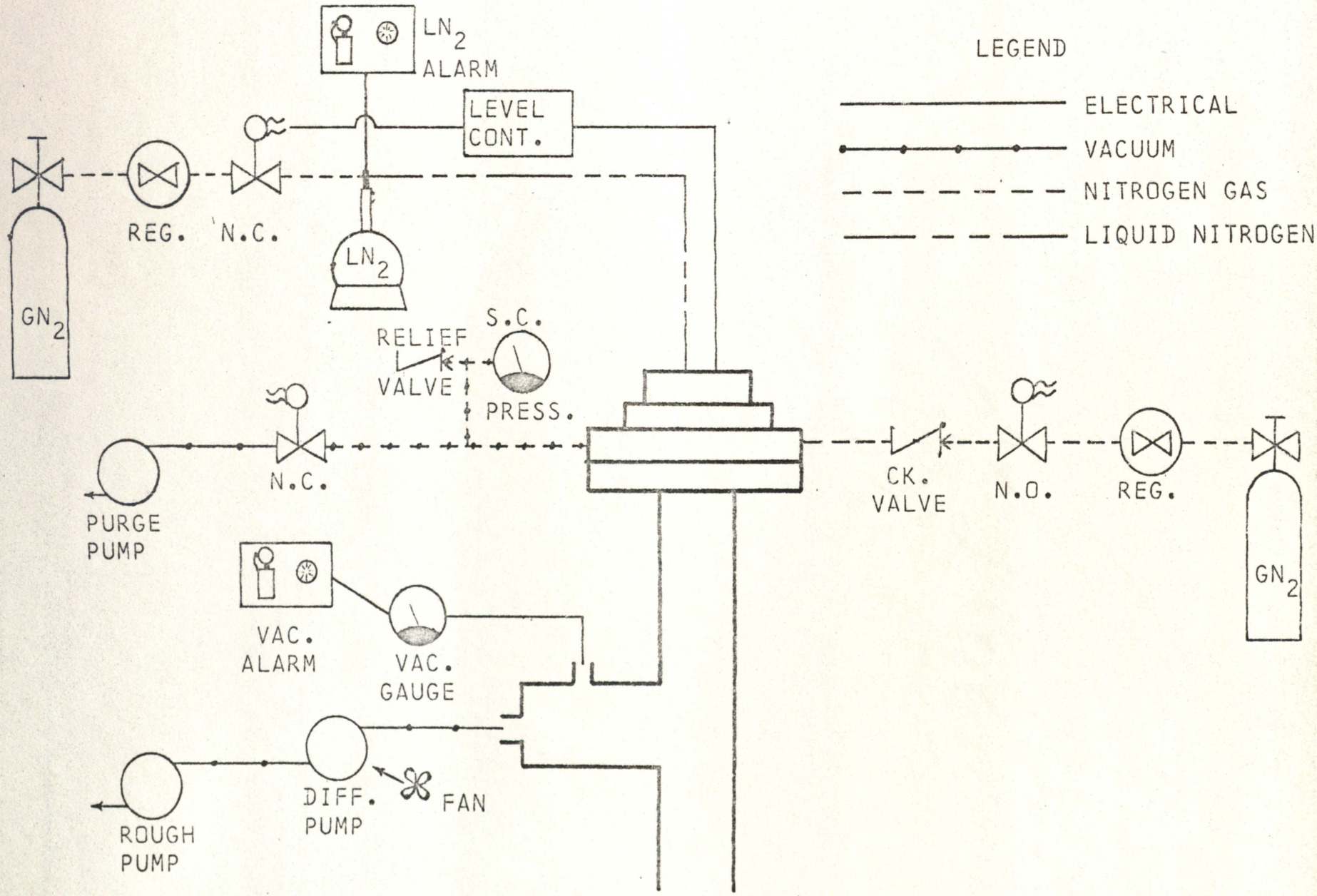
A check valve is installed at the head on the GN_2 line to prevent a back flow of radioactive gas should there be a rupture in the GN_2 supply line. This check valve is set to allow gas into the sample chamber when a pressure differential of one (1) psi exists and is leak tight to a back pressure of 3,000 psig.

A relief valve set at 35 psig is installed on the sample chamber side of the purge system to relieve the pressure if the pressure in the sample chamber becomes excessive.

A light weight vinyl tube carries the GN_2 from the head to the bottom of the sample chamber. This was done in an effort to maximize the effectiveness of the purging system.

3. LN_2 Supply System

The liquid nitrogen (LN_2) supply system includes a 50 liter stainless steel Hofman Dewar flask designed for a maximum of 50 psig working pressure, a Johns Technology LN_2 transfer line kit, a Johns Technology Model L-20 Cryo-Miser liquid nitrogen level control, and a tank of nitrogen with a regulator. Figure (2) shows a layout of the system.



SCHEMATIC OF THE CRYOSTAT
AND SUPPORTING SYSTEMS
FIGURE 2

To initially fill the LN_2 tube in the cryostat (tube 1) the level control unit is switched to automatic which opens the solenoid gas valve thereby supplying a pressure to the Dewar flask and forcing the LN_2 through the transfer line into the cryostat. The solenoid will close when the level of the LN_2 in tube 1 reaches the upper level sensor. A light on the control panel indicates when the normally closed gas solenoid has been energized.

The upper most flange of the head contains a 1/4" hole which allows the evaporated nitrogen to escape. This presents no serious radiation hazard but does require cautious respect since the vapor is extremely cold.

A 10 psi relief valve is included in the Dewar head assembly to prevent over pressurizing the system. If the over pressure in the system is so great that the relief valve cannot handle the situation, the next weakest link should prove to be the rubber stopper fitted to the Dewar head.

A level sensing device in the Dewar will indicate when the Dewar is near empty by actuating the alarm and an indicator light on the control panel.

II. INSURING SAFE OPERATION OF THE CRYOSTAT

A. CRYOSTAT CONTROL AND SAFETY SYSTEMS

1. Instrumentation

The control and instrumentation of the cryostat and its supporting systems has been kept as simple and rugged as possible. The layout of the control panel can be seen in Figure (3).

The Cooke Vacuum Gauge (Model 1300) is of the cold cathode discharge type. This type of gauge, though found lacking in accuracy, is well known for its simplicity and ruggedness. Since the only purpose of a vacuum in this system is to provide sufficient insulation to isolate the warm pool and the cold sample chamber, only a rough indication of the quality of the vacuum is required. The gauge has been standardized with a high quality ionization gauge.

Two gas regulators are employed in the system. Both are of the two gauge type indicating the pressure and quantity of gas in the tank and the pressure in the transfer lines.

A Cenco model 94035 pressure gauge is connected to the vacuum line of the purge system between the normally closed solenoid and the head of the cryostat. This gauge is of the positive-negative type reading continuously from 30 inches Hg vacuum to 30 psig. The gauge gives an indication of the pressure condition in the sample chamber at all times.

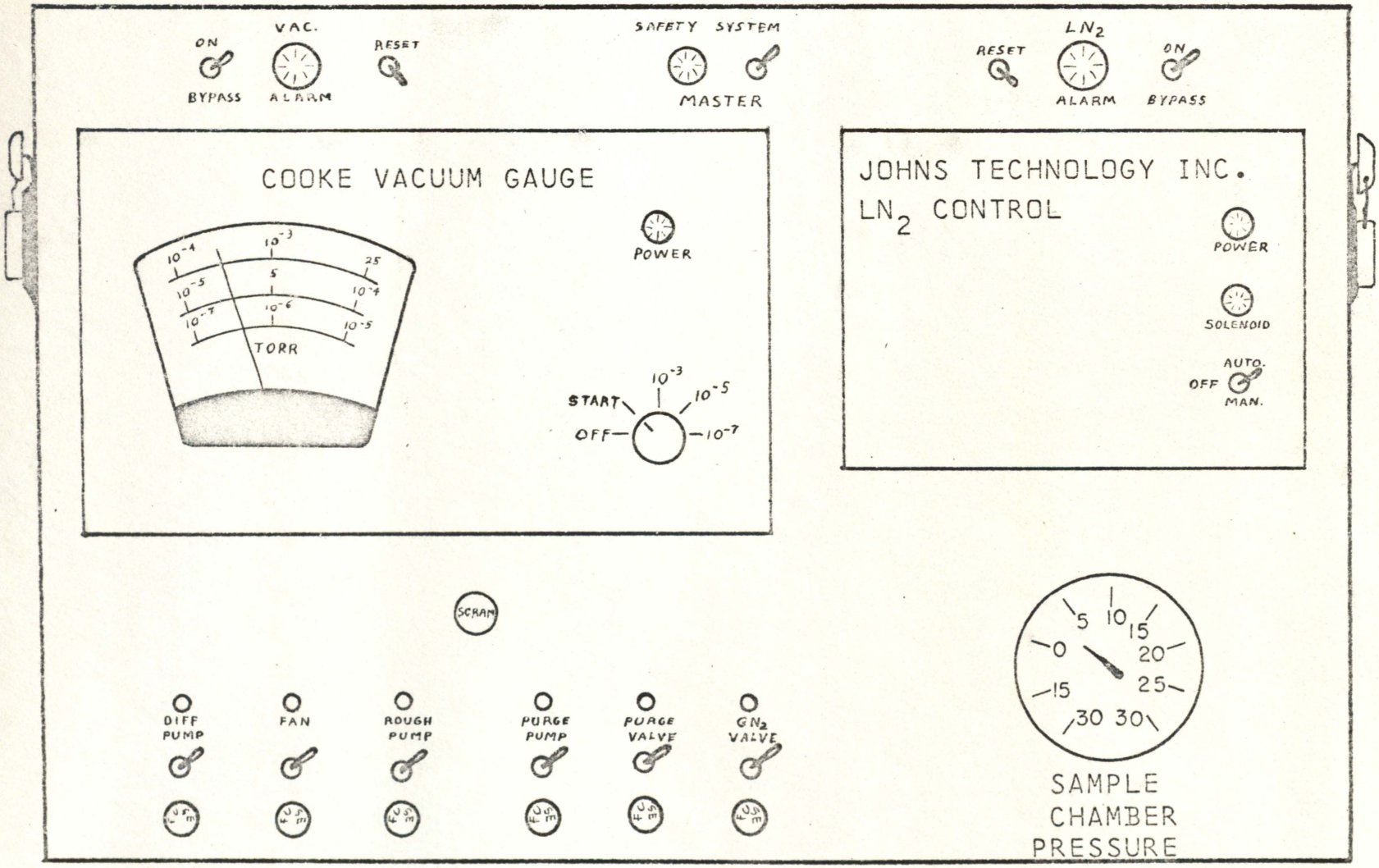


FIGURE 3 CONTROL PANEL

When it is desired to know the temperature in the sample chamber, it is recommended that a copper constantan thermocouple be used. This type of thermocouple is generally accepted to be the best for cryogenic applications. Extreme caution must be urged in handling the thermocouple after irradiation since the copper will be somewhat radioactive. It should be kept in mind that the irradiated copper produces two radioactive isotopes, Cu^{64} and Cu^{66}, with half-lives of approximately 13 hours and 5 minutes respectively. In conjunction with the thermocouple it is recommended that a high quality millivolt potentiometer be used such as a Leeds and Northrup Model 8686 or a Model 8690.

2. Automatic Operation

It is anticipated that the cryostat will be in use for continuous periods of up to 16 hours. For this reason it is desired that the operation of the system be automatic after the start-up procedure has been completed, that audio and visual annunciation be included should any phase of the cryostat go amiss, and that the system be fail-safe in case of a cryostat or reactor accident.

It is suggested that when the reactor is being operated adjacent to the thermal column that the control panel for the cryostat be placed at the east end of the pool. Should a cryostat annunciation then occur, it would be readily apparent to the reactor operator as well as the area of difficulty.

The vacuum system will be in operation at all times. Should the reactor and/or the cryostat be scramed during operation, it is felt that maintaining the required insulating vacuum is of primary importance. If during operation of the cryostat the vacuum should rise above 20 microns, the alarm on the control panel will sound and a red light on the instrument panel will flash.

The level of liquid nitrogen in the cryostat is maintained by the Johns Technology Model L20 automatic control. Should the level of LN_2 in the Dewar flask drop below 10% (5 liters) the cryostat alarm will sound and a red light on the control panel will flash. To correct this situation the LN_2 control unit is switched off and the Dewar flask should then be refilled.

The pressure of the GN_2 in the sample chamber is controlled by the regulator on the appropriate tank and that pressure is indicated on the regulator line gauge and on the sample chamber pressure gauge. Should the supply tank become empty, the check valve in the line will prevent any back flow of the radioactive GN_2.

B. ADMINISTRATIVE CONTROLS

Since the cryostat is a potentially dangerous apparatus, the likelihood of an accident involving the cryostat can be greatly reduced by the clear definition of responsibility for the various phases of operation. At all times, regulations and procedures of reactor operations take precedence over cryostat regulations and procedures.

C. DUTIES OF PERSONNEL

1. Director of Reactor Facility

The Director will have the primary responsibility of over-seeing all cryostat activities. He shall make the final decisions relating to utilization of cryostat time, feasibility of experiments and operational procedures.

2. Senior Operator

a. Assume primary responsibility for safe operation of the cryostat and insure that experimental requirements do not unduly compromise safety.

b. Supervise all cryostat operating personnel.

c. Supervise training of new operating personnel.

d. Take appropriate corrective action to prevent recurrence of any significant malfunction, violation, or accident in connection with cryostat operation.

e. Cooperate with the Director in scheduling and coordinating experimental programs and service irradiations.

f. Review requests for irradiation time. Approval or disapproval shall be based strictly upon consideration of operational safety rather than the merits of the experiment.

3. Cryostat Operator

A cryostat operator shall be a person imminently familiar with the cryostat and its operation. He shall be

approved to operate the cryostat by the Director.

a. Accept responsibility for safe operation of the cryostat at all times.

b. Make all minor decisions regarding operation of the cryostat and all decisions required immediately.

c. Remain in the reactor building at all times except when relieved by another qualified operator.

d. Carry out appropriate checks of the safety circuits and supervise routine maintenance.

e. Manipulation of controls and surveillance of instrumentation.

f. Advise the supervisor of any unusual behavior on the part of the cryostat and its controls, taking any necessary action to prevent damage to the cryostat, the reactor and protect health.

4. Reactor Operator

a. The reactor operator shall not be the cryostat operator simultaneously.

b. In addition to his normal duties, the reactor operator will notify the cryostat operator of any abnormality in the operation of the reactor which might be caused by or affect the operation of the cryostat.

III. ACCIDENTS INVOLVING THE CRYOSTAT

The utmost care has been taken in the design of the cryostat to guard against accidents of any type. However, not all possibilities can be anticipated. Listed below are several "major" accidents that might occur, the results of each accident, the measures taken to prevent such an occurrence, and the appropriate emergency or corrective action to be taken in each situation.

A. RUPTURE OF OUTER TUBE (TUBE 3)

1. Cause

The most likely cause for a leak in the outer tube would be cracks in the tube-flange welds near the offset or in the welds of the elbow bend of the tube.

If the cryostat were in position in the core without any connections or tubes in place it would be possible to apply a horizontal force to the head of sufficient magnitude to rupture the tube at or near the tail piece.

If an explosion were to occur in the sample chamber of a magnitude equal to or greater than 0.0204 lbs. of TNT, the outer tube would burst. Any explosion of lesser magnitude will be contained.

2. Effect

If the cryostat were not in operation at the time there would be no apparent effect other than the presence of the cracks. However, if the cryostat were in operation, a leak of almost any size would be sufficient to raise the vacuum above 20 microns which would set off the vacuum alarm system.

3. Corrective or Emergency Action

If the crack or rupture is located below the water it is suggested that operation be terminated with a normal shut down procedure. If the leak is above the water level it might be possible to complete the experiment in progress providing the leak is small.

In the event of an explosion, the cryostat should be scramed. Further action will be dictated by the particular situation.

4. Preventative Action

The outer tube is schedule 40, 6063 T6 Aluminum pipe which should be more than sufficient to withstand any mechanical load that might be imposed upon it. All welds have been made extra strong and a double pass on each weld insures sufficient penetration. The tail piece extends 2 inches into the outer tube to increase the strength of the tube.

B. RUPTURE OF SAMPLE CHAMBER (TUBE 2)

1. Cause

The flanges of tube 2 (and the sample chamber) are solidly bonded to the tube with a high grade of epoxy cement. Although the flanges are almost a force fit over the tubes and therefore the amount of epoxy required is minimal, it is felt that sooner or later cracks will develop in the epoxy.

An explosion occurring in the sample chamber equivalent to or greater than 0.0049 lbs. TNT will cause the sample chamber to burst.

2. Effect

The first situation listed above will be readily apparent during the start up procedure, et. sec. some difficulty will be experienced in obtaining a vacuum between tubes 2 and 3. When the sample chamber is evacuated during the purge sequence the main vacuum will drop. However, when the sample chamber is back filled with gaseous nitrogen, the vacuum gauge will indicate an increase in the pressure. This abnormality will be readily apparent to the cryostat operator.

An explosion in the sample chamber of sufficient magnitude to rupture tube 2 but not tube 3 will cause the vacuum system alarm to be activated.

3. Corrective or Emergency Action

The size of a particular leak will determine whether the experiment can or should not be continued. At any rate, the leak should be repaired at the earliest possible date. For very small leaks a coat of Glyptol paint may be sufficient to stop the leak. This should be regarded as a temporary measure only and the flange should be removed, cleaned, and re-epoxyed as soon as time permits.

In the event of an explosion the system should be scramed but the insulating vacuum should be re-established as quickly as possible. The reason for this is to minimize any cooling of the outer tube while it is in the vicinity of the core.

4. Preventative Action

Tube 2 and the sample chamber are made of extra strong, schedule 40, 6063 T6 Aluminum pipe. This should be more than

adequate for any anticipated situation. The flanges were designed and machined so that they fit very snugly on the pipe and so that the flanges carry all of the bolt load.

C. RUPTURE OF INNER TUBE (TUBE 1)

1. Cause

When tube 1 is initially filled with liquid nitrogen, large but short lived thermal stresses are set up in the tube. This may eventually cause some cracks to appear.

2. Effect

In the unlikely event that a sizable rupture should occur in tube 1, it might not be readily apparent. A decrease in the pressure in the sample chamber might occur but the pressure would more than likely be restored to normal by the GN_2 supply to the sample chamber. In addition to the excessive GN_2 consumption, other indications would be a rise in the sample chamber temperature and an increase in the LN_2 consumption. It is possible that the radiation monitor on the bridge may be activated due to the presence of N^{16}.

3. Corrective or Emergency Action

In the event that a sizable rupture does occur in tube 1, the system should be scramed. The reactor should also be shut down as quickly as possible. Care should be exercised in approaching the cryostat since some N^{16} activity should be present due to the oxygen impurities in the commercial LN_2.

4. Preventative Action

Tube 1 was selected to be as thin as possible to reduce the effect of the thermal shock and to maximize the rate of

heat transfer. The tube supports no load other than its own weight.

D. CONCENTRATION OF OXYGEN IN SAMPLE CHAMBER

1. Cause

Improper purging of the system could cause a very high concentration of oxygen in the sample chamber. Gross negligence could occur such as a failure on the part of the operator to perform the purging sequence or inadvertantly connecting an oxygen tank rather than a nitrogen tank to the sample chamber.

2. Effect

The most serious consequence of a concentration of oxygen in the sample chamber is the imminent possibility of an explosion such as those that occurred at Oak Ridge in 1956.

3. Corrective or Emergency Action

If it is realized that one or more of the above errors have been committed, the reactor should be shut down immediately. The LN_2 system and the GN_2 supply to the sample chamber should be shut off and the purge pump should be used to evacuate the sample chamber. Great care should be exercised if the reactor has been operating since N^{16} activity will be present.

4. Preventative Action

The entire design of the cryostat has been built around the concept of reducing or eliminating the possibility of an accident of this nature. It is felt that if the purging

sequence is followed as prescribed, the likelihood of such an occurrence is extremely small. In addition, the materials employed in the cryostat were selected to be much stronger than required so that if an explosion did occur, it could be contained.

E. RUPTURE OF LN_2 SUPPLY LINE

1. Cause

A very severe force applied to the transfer line while it is cold could possibly break the line.

2. Effect

Should the transfer line be broken the liquid nitrogen will flow from both sides of the break into the surrounding area.

3. Emergency or Corrective Action

No attempt should be made to stop or retard the flow of LN_2 at the break. The LN_2 level control should be shut off and after the transfer line has warmed up, it should be replaced.

4. Preventative Action

The polyethylene transfer line used with the cryostat is extremely satisfactory for this application. Experiments with this hose have proved it almost unbreakable whether warm or cold. Past experience with the hose has shown that once connected to the barbed hose fittings, the tube, more often than not, must be cut to be removed.

F. OVERLOAD ON ELECTRICAL CIRCUIT

1. Cause

The maximum instantaneous electrical load of all the supporting systems is 26 amps and the normal operating load is roughly 15 amps. The load imposed by the starting torque of the vacuum pump motors is rather large and may trip a breaker. An electrical short is always a possibility.

2. Effect

The most undesirable effect of a power loss is a loss of vacuum. This constitutes a more undesirable condition by increasing the thermal conductivity between the sample chamber and the outer tube thereby creating a cold spot in the vicinity of the core.

3. Emergency or Corrective Action

Scram the system if necessary to reduce the load on the line and reset the breaker. Re-establish the vacuum in the system as quickly as possible. Isolate the area of difficulty and correct it.

4. Preventative Action

Each of the sub-systems is equipped with a fuse. Therefore, if any individual load becomes excessive it will not affect the operation of the other systems. Two completely separate electrical supply circuits are employed. All instrumentation runs off of one line and all "power" equipment off the other. This was done so that if the "power" side should go out, the instrumentation and safety systems will still be in operation to indicate that fact. Each sub-system

also operates an indicating light on the control panel so that if a fuse is blown it will be readily apparent.

G. RADIATION AND NUCLEAR ENGINEERING CONSIDERATION

The greatest concern in this area is the amount and type of radioactivity to be anticipated with the system. Calculations have shown that the nitrogen in the sample chamber, after operation for 8 hours at 200 KW in a position adjacent to the core, will reach a maximum activity of 23.6 microcuries. This activity will be in the form of C^{14} with a half-life of 5730 years. An activity of this magnitude requires respect but does not constitute a serious health hazard.

The aluminum in the cryostat has a half-life of 2.30 min. and can be handled in relative safety after a cooling-off period of one hour. As per reactor safety regulations, the cryostat may not be removed from the pool without the presence of the health physicist, the reactor supervisor, or the director.

An important consideration of any experiment to be placed near the core is the reactivity worth of that experiment. According to AEC regulations, any experiment in the UMR reactor is limited to ± 0.20% reactivity worth without performing an approach to critical with the experiment in place. Calculations have shown the reactivity worth of the cryostat to be -0.18%.

IV CONCLUSIONS

The cryostat described in this report has been designed and built with safety and reliability of operations as the primary goals. Five preliminary experiments have been performed including one run in the pool. Satisfactory results have been obtained and the experiments that simulated cryostat accidents proved the ability of the cryostat to handle such occurrences. It is felt that the system is safe and reliable and should prove to be a valuable addition to the reactor facility.

APPENDIX B

OPERATIONAL AND MAINTENANCE PROCEDURES FOR THE UNIVERSITY OF MISSOURI AT ROLLA REACTOR CRYOSTAT

Prepared By

BRUCE B. JOINER

December 1967

University of Missouri at Rolla

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LIST OF ILLUSTRATIONS

I. INTRODUCTION

The cryostat is a relatively simple piece of research equipment. It does, however, require great respect since it is potentially hazardous. The operator* should become completely familiar with the design of the cryostat, its principle of operation, and the contents of this manual. Approval to operate the cryostat must be obtained from the reactor supervisor after satisfactory completion of a training and familiarization program.

Any modifications or improvements to the cryostat, its supporting systems, the maintenance procedure or the operating procedure must be included in this manual. Such modifications must be approved by the director of the reactor and the reactor supervisor.

*The term operator refers to the cryostat operator unless otherwise specified.

II. ASSEMBLY AND DISASSEMBLY

Great care should be taken to insure that the various tubes and hardware have not become excessively radioactive. The cryostat consists of three concentric tubes which must be assembled and disassembled in a given order. Always tighten and loosen the nut, not the bolt, to minimize damage to the bolt holes. The aluminum flanges are easily damaged by the stainless steel bolts. Always use flat washers between the faces of the bolts and nuts, and the flanges. The faces of the flanges are very soft and care should be exercised to protect these surfaces from scratches.

A. Complete Disassembly of Lower Portion (See Fig. 1.)

The cryostat should be laid flat on the floor.

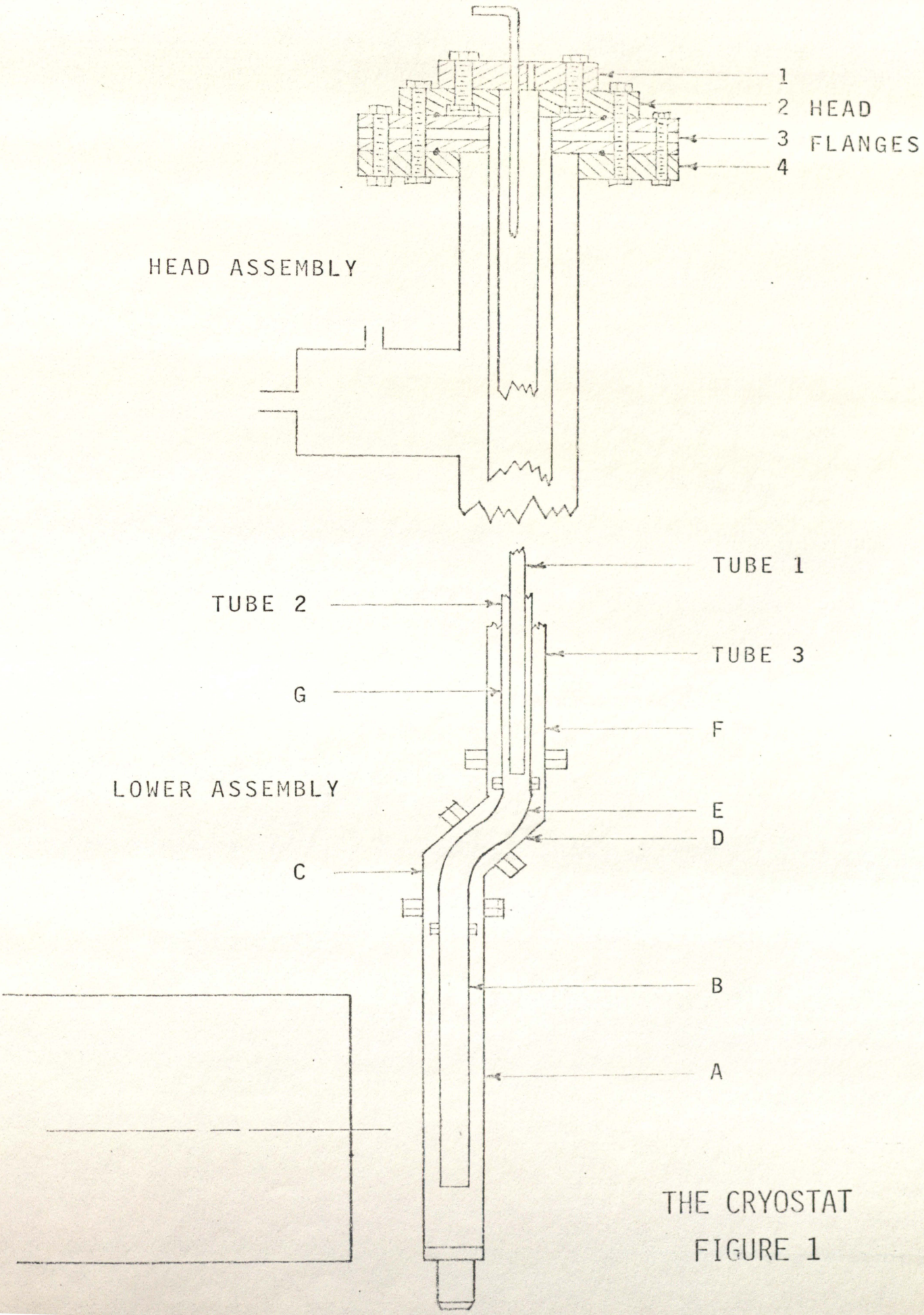
(1) Unbolt and remove tube A from elbow C. Some difficulty can be expected since the flanges of B and E bind against tube A.

(2) Unbolt and remove sample chamber B from offset E.

(3) Unbolt and remove elbow C from elbow D.

(4) Unbolt elbow D from down tube F and slide the elbow away from the down tube. The elbow <u>cannot</u> be removed at this time.

(5) Unbolt offset E from down tube G. Elbow D and offset E can now be removed together.



THE CRYOSTAT

FIGURE 1

B. Assembly of Lower Portion (See Fig. 1.)

(1) Clean all flange sealing surfaces with a dry, soft, clean cloth. Wipe all "O" rings to remove vacuum grease and dirt. Replace damaged "O" rings. Spread a thin layer of vacuum grease on the sealing surface of all flat faced flanges and in the "O" ring groove of the other flanges. Use a minimum amount of grease spreading it evenly with a finger.

(2) Place the 8 "O" rings in position in all of the grooved flanges.

(3) The holes in the flanges will allow the offset to be rotated in 45° incriments. Tube A can also be rotated in 45° incriments with respect to the offset. Give careful consideration to the orientation desired between the head, the offset, and the tail piece of tube A.

(4) Place offset E inside elbow D (elbow D is longer than elbow C and the grooved flange of offset E goes up)

(5) Bolt offset E and down tube G together.

(6) Bolt elbow D and down tube F together.

(7) Bolt elbow C to elbow D.

(8) Bolt sample chamber to offset E.

(9) Bolt tube A to elbow C.

Note: Always make sure that the various parts are dry before assembling the cryostat. Water

vapor will turn the vacuum pump oil to a heavy sludge.

C. Disassembly of Head Portion (See Fig. 1.)

The head of the cryostat consists of 4 flanges. Head flange 1 permits access to tube 1 and is secured to head flange 2 by the use of 4 bolts. To detach head flange 1 remove the 4 nuts and flat washers holding that flange. It is not necessary to remove head flange 2 first.

Head flange 2 is affixed to tube 1. This sub-assembly can be detached by removing the 8 long stainless steel bolts that hold head flanges 2, 3, and 4 together. If desired, as in cold sample removal, (see III A) head flanges 1 and 2 can be removed together.

Head flange 3 is welded to tube 2 and head flange 4 is welded to tube 3. These two flanges should not be unbolted unless absolutely necessary. Tube 2 can not be removed from inside tube 3 because of welding "run through" on the lower portion of tube 3 which prohibits the passage of the lower flange on tube 2.

D. Assembly of Head Portion (See Fig. 1.)

If head flanges 3 and 4 have been separated and it is found necessary to replace the "O" ring, this can be accomplished in two ways. (The "O" ring is Buna N, 4 inch dia.x 1/8 inch thick) First, an ordinary 4 inch Buna N "O" ring can be stretched over head flange 3. The "O" ring will return to its approximate original size within an hour. The second method is to use an "O" ring making kit and place the

strip of "O" ring material (Buna N) around tube 2 before glueing the ends together.

Wipe the sealing surfaces of all flanges with a soft dry cloth and apply a thin coat of vacuum grease to each of the sealing surfaces. Head flanges 3 and 4 can now be bolted together. (Note: It is not possible or necessary to use lock washers on these bolts.)

The 4 bolts which join head flanges 1 and 2 must be placed in head flange 2 before this flange is bolted to flanges 3 and 4. Rubber washers should be placed in the 4 bolt holes in head flange 2 to prevent damage from the stainless steel bolt heads.

Place a 4 inch Buna N or Teflon "O" ring on the face of head flange 3 before inserting tube 1 into tube 2. Use 8 of the 3 1/2 inch stainless steel bolts to secure head flanges 2, 3, and 4. Use lock washers on these bolts.

Head flange 1 can now be fastened to head flange 2. Neither an "O" ring nor lock washers should be used in joining these two. The nuts should be 1/2 turn more than finger tight.

III. SAMPLE INSERTION AND REMOVAL

Access to the sample chamber may be gained by removing tube A and sample chamber B or by removing tube 1 and lowering the sample into the sample chamber with a string. Wires for electrical connections can be run from the sample chamber to the head and out the electrical lead hole in head flange 3. This hole must then be filled with some type of sealant such as epoxy, Dow Corning RTV Rubber Flexible Mold Compound, or Cope Plastics' Castolite, to prevent leakage of air into the sample chamber.

Normal sample removal can be accomplished by unbolting tubes A and B or by removing tube 1 and pulling the sample up by the string.

A. Cold sample removal

Cold sample removal can be accomplished by initially attaching a string to the sample and lowering the sample into the sample chamber. The upper end of the string should then be tied to the upper portion of tube 1 as close as possible to head flange 2.

(Note: The reactor MUST be shut down before removing the sample. Gamma streaming might otherwise create a health hazard. As per reactor regulations the health physicist or senior operator must be present when a sample is removed from the pool.)

B. Procedure

The use of an overhead crane is required to accomplish cold sample removal.

(1) Reduce the pressure in the sample chamber to 2 psig.

(2) Turn the LN_2 level control off.

(3) Disconnect the GN_2 transfer line to the Dewar.

(4) Disconnect the LN_2 transfer line from the Dewar to the head. Use extreme caution. Liquid nitrogen or very cold vapors will spurt from the head and from the Dewar. Wear heavy gloves, a long sleeve shirt, protective glasses, and a hard hat when performing steps 4 through 8.

(5) Disconnect the LN_2 level control leads.

(6) Remove the eight bolts that hold head flange 2 to head flanges 3 and 4.

(7) Attach the overhead crane to the tube 1 assembly and remove carefully.

(8) Retrieve the sample by pulling it up with the string.

(Warning: The liquid nitrogen in tube 1 will boil rapidly as the tube is warmed by the air and this may cause it to "rain" liquid nitrogen briefly. This is not a hazardous condition but the individuals in the area should be alerted to reduce the element of surprise. Both the cryostat and tube 1 must be warmed to room temperature before they can be reassembled. Thoroughly dry tube 1 before reassembling.)

IV. PREPARING THE CRYOSTAT FOR OPERATION IN THE CORE

The cryostat should be cleaned with alcohol before being placed in the core to remove substances that might contaminate the pool water or create radioactive hot spots on the cryostat.

Three people are required to place the cryostat in the pool due to its size. The easiest way to accomplish this is to move the head of the cryostat towards the freight door in the bay and carefully lower the tail portion into the water from the north side of the pool. It is recommended that a safety line be attached to the head during this operation.

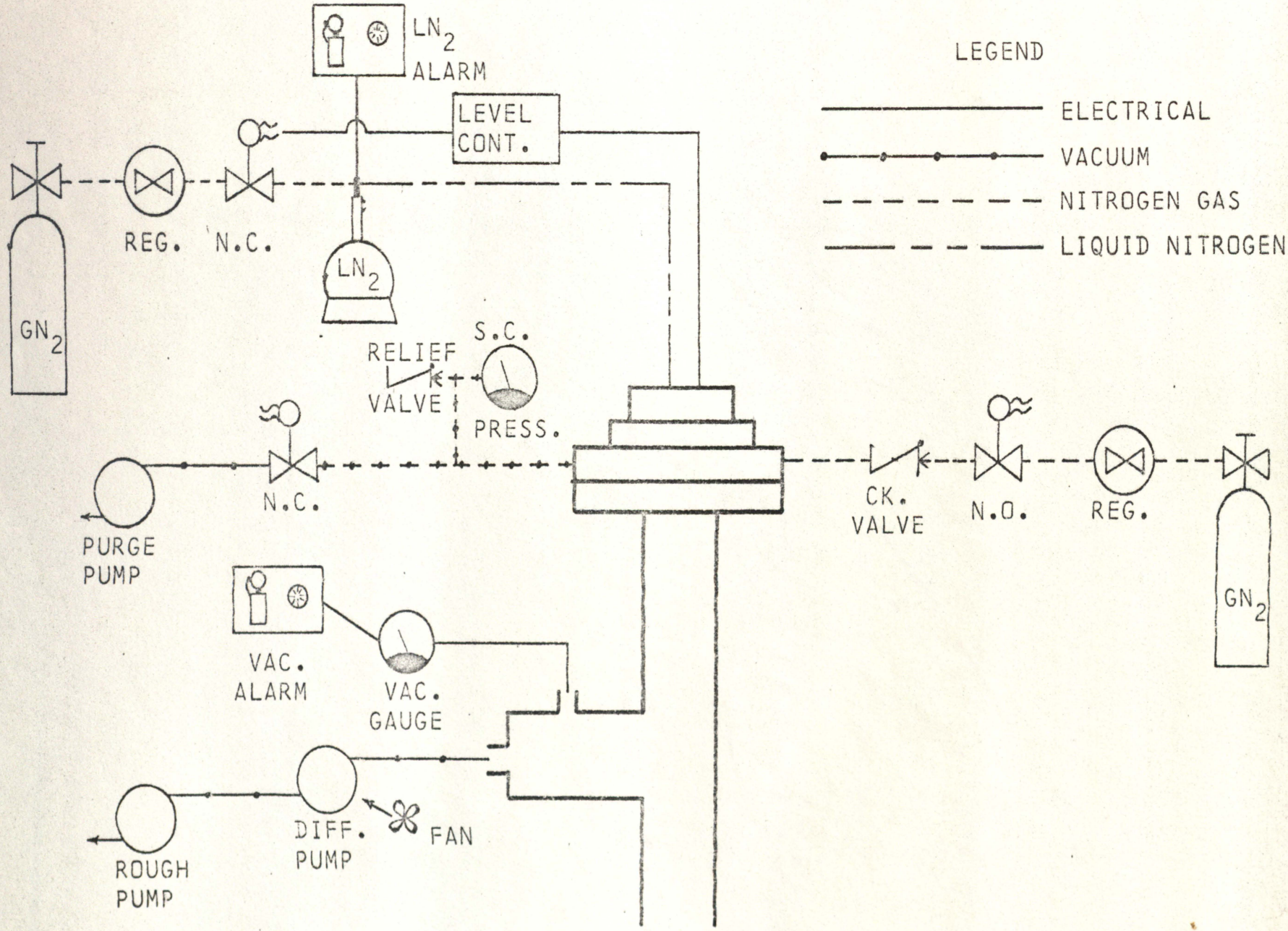
After the tail piece has been placed in the desired position in the grid plate, the cryostat should be secured to the bridge with the bridge clamp.

The following list is intended as a guide for making the proper mechanical and electrical connections to the cryostat. (See Fig. 2.)

(1) Apply a thin coat of vacuum grease to the vacuum outlet near the head of the cryostat and to the diffusion pump inlet.

(2) Connect the vacuum outlet to the diffusion pump with the thick Tygon tubing. Use one clamp on the vacuum outlet and two clamps on the diffusion pump inlet.

(3) Connect the purge outlet to the purge pump with a piece of clear plastic tubing. Use two clamps



SCHEMATIC OF THE CRYOSTAT
AND SUPPORTING SYSTEMS
FIGURE 2

on the purge outlet and one clamp on the pump inlet.

(4) Apply a thin layer of vacuum grease to the vacuum gauge tube and to the vacuum measuring part. Connect these two with a short piece of clear plastic tubing. Use one clamp at the port and two on the tube.

(5) Two tanks of high purity nitrogen gas should be positioned next to the bridge steps and tied in place.

(6) Attach a pressure regulator to each tank.

(7) Attach the sample chamber GN_2 solenoid to one tank and the LN_2 solenoid to the other.

(8) Use an appropriate length of reinforced gas hose to connect the GN_2 solenoid to the sample chamber GN_2 inlet. Clamp the hose at both ends to the barbed fittings.

(9) Make the electrical connections between the GN_2 valve and the control panel. (Wires are appropriately marked.)

(10) Make the electric connection between the LN_2 valve and the control panel. (Wires are appropriately marked.)

(11) Place the electrical distribution box on the bridge.

(12) Connect the gas hose from the control panel to the sample chamber pressure outlet on the head. Use a hose clamp.

(13) Plug the power cords of the rough pump, diffusion pump, fan, purge pump, and purge valve into the appropriately marked receptacles in the distribution box.

(14) Make the electrical connections to the vacuum gauge tube.

(15) Connect a suitable length of gas hose to the LN_2 solenoid and lead it up to the bridge. DO NOT connect it to the Dewar at this time.

(16) Connect the gray LN_2 level control leads from the head to the control panel.

(17) Double check all connections.

V. START UP AND SHUT DOWN PROCEDURE

The following check lists for start up and shut down should be followed as stringently as those pertaining to the reactor. Figure 3 shows the control panel layout.

A. Start Up

The first 14 items listed on the start up sheet provide a systematic check of the mechanical and electrical sub-systems. Any abnormality which shows up during this check out should be corrected before proceeding. Any serious malfunction should be reported immediately to the senior reactor operator on duty.

With respect to the first item on the start up check list i.e., both power cords plugged in, these should NOT be placed in the same wall receptacle box. The total electrical load of the cryostat system is greater than the maximum allowable for a single outlet box.

The last 14 items on the start up check list pertain to the actual operation of the cryostat. It is recommended that experiments performed with the cryostat be started early in the morning and that the start up procedure be initiated the previous afternoon. This will allow a suitable vacuum to be established overnight. An operator's presence is not required during this period.

Purging the sample chamber is extremely important in order to remove oxygen from the sample chamber. Oxygen liquifies at a higher temperature than nitrogen and the

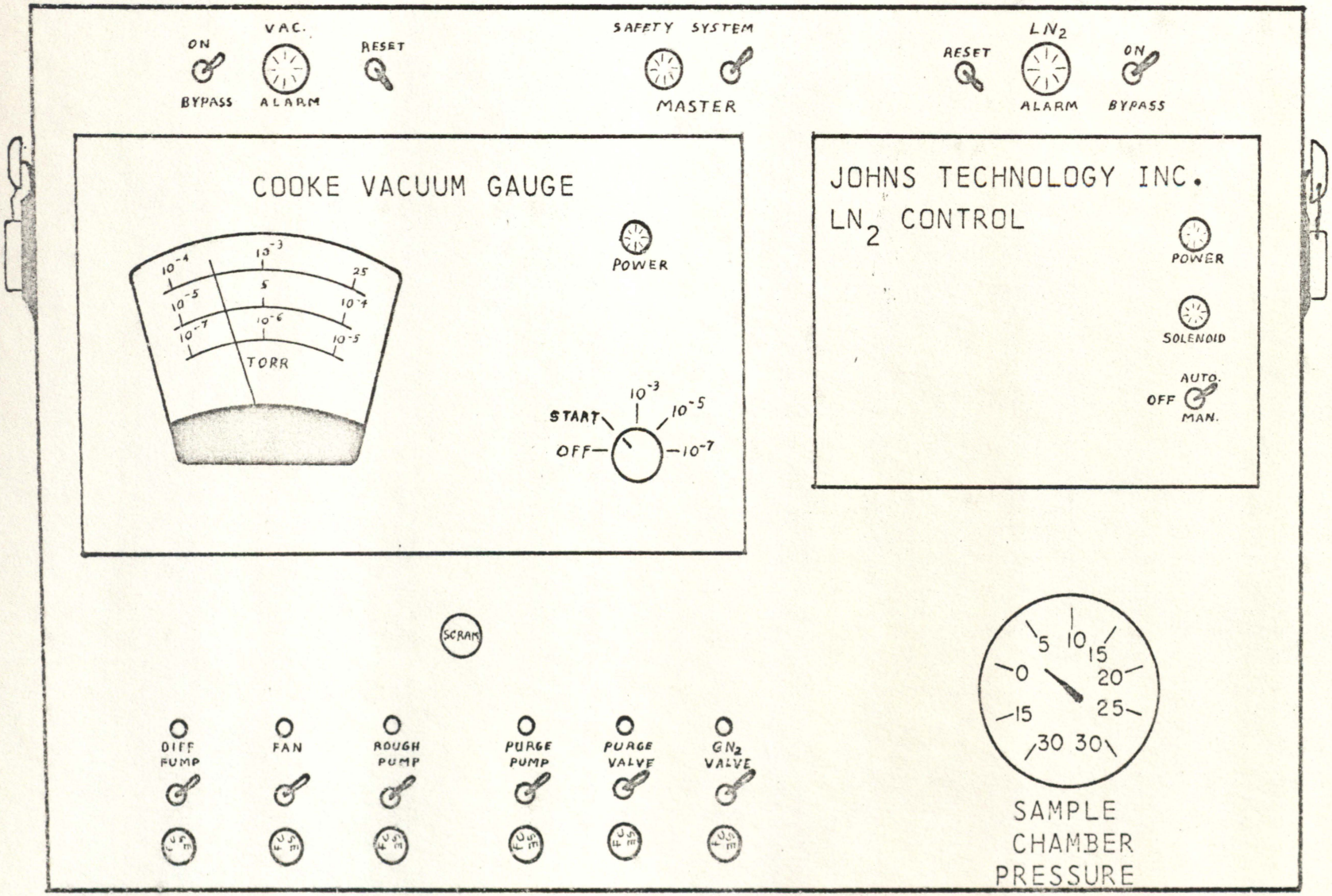


FIGURE 3 CONTROL PANEL

intense ionizing radiation from the reactor will convert this liquid oxygen to unstable ozone. Several cryostats of early design exploded due to this situation. (See e.g. ORNL 2188) Therefore, pump the sample chamber down as far as possible, usually to about -27 inches of Hg, and then back fill the sample to +15 psig with gaseous nitrogen. Repeat this step four times. Evacuate the sample chamber once more and continue pumping during the next four steps.

To connect the Dewar flask, attach the gas hose from the GN_2 tank to the gas inlet on the Dewar and then attach the LN_2 outlet of the Dewar to the LN_2 inlet of tube 1 on top of the head of the cryostat. Use a short piece of polyethylene tubing for this purpose. Also connect the Dewar LN_2 level indicator leads.

Past experience has shown that the best sample chamber pressure for steady state operation is 3 psig. Further experimentation may prove this to be too low for some applications, but do not exceed a steady state operating pressure of 25 psig. To prevent over pressurization of the chamber, the sample chamber relief valve on the head of the cryostat should be set to open at 35 psig.

B. Shut Down

The shut down procedure warrants a few words of advice. Use extreme caution when disconnecting the GN_2 line to the Dewar since cold nitrogen vapor will be emitted from the head of the Dewar.

If the cryostat is to be left in the core while the reactor is in operation, it is necessary to back fill the sample chamber with gaseous nitrogen. If it is not desired to leave the vacuum system in operation, then this void must also be back filled with gaseous nitrogen. If this is not done the oxygen in the air will be converted to radioactive nitrogen-16. Even though the half-life of N^{16} is very short (7.4 sec.) this may present problems during the disassembly of the cryostat.

VI. MAINTENANCE

Routine maintenance to the cryostat will be apparent as the need arises. Listed below are items which will need periodic attention.

After 25 runs or 60 days, whichever comes first;

1) Change the vacuum pump oil in the rough pump and in the purge pump.*
2) Change the diffusion pump oil.*
3) Descale the vacuum gauge tube following procedures in section A. below.
4) Check the electronic tubes in the safety system.
5) Clean the inside of the sample chamber and the inside of tube A.
6) Polish the outside of the sample chamber and tube A.

After 75 runs or 6 months, whichever comes first;

1) Descale the vacuum gauge tube and calibrate against a high quality ionization gauge.
2) Spray the threads of the stainless steel nuts and bolts with Teflon. Descale first if necessary.

A. Vacuum Gauge Tube Cleaning

Prepare a solution of 8-12% by volume of sulfuric acid with water and add 5% by weight of ferric sulfate. Heat the solution to 150-170°F. Remove the magnet from the tube. Pour the heated solution into the tube and

*Follow manufactures recommended procedures.

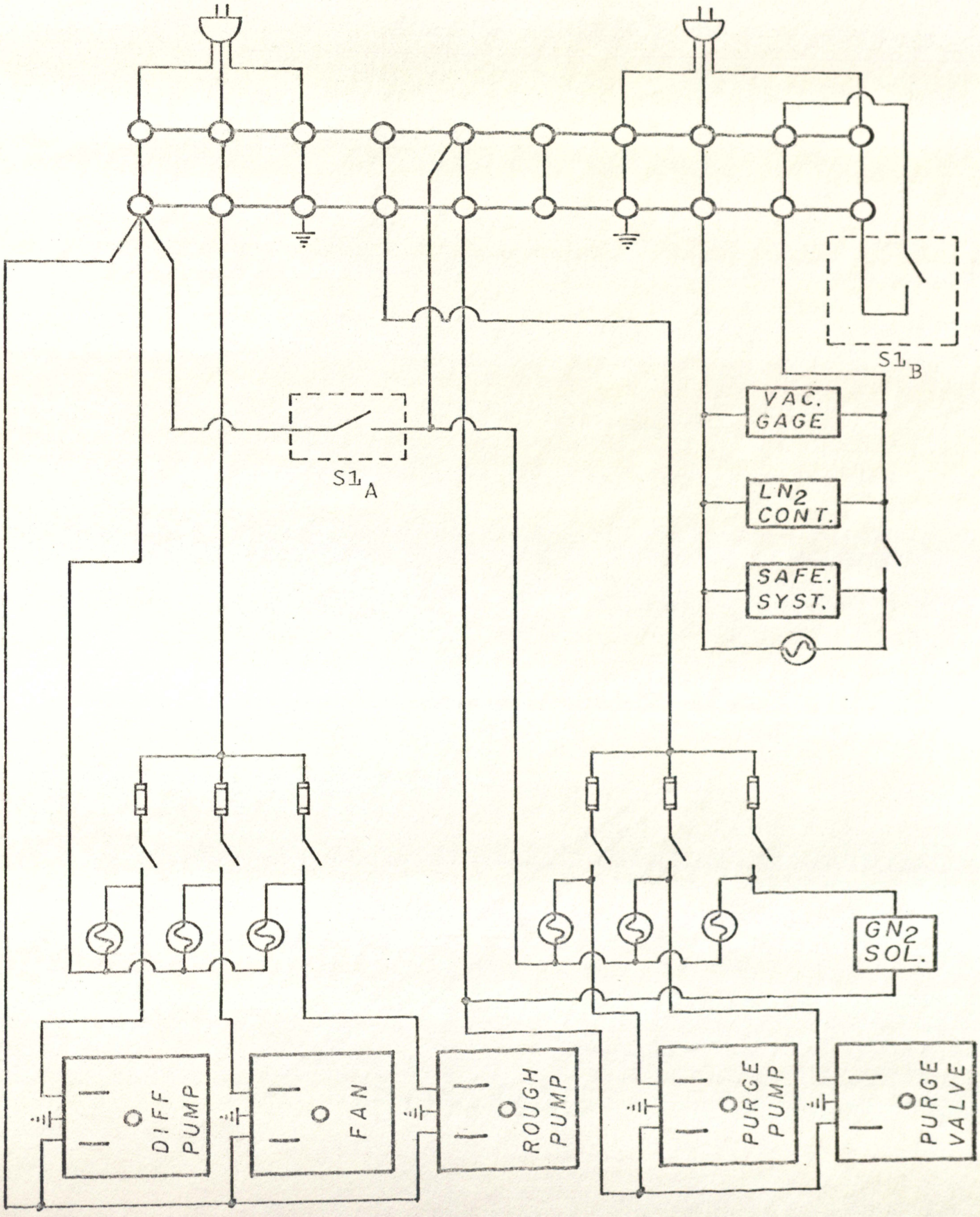
allow it to stand for 15-20 min. Remove the solution and flush thoroughly with water. Rinse the tube with isopropyl alcohol and allow to dry before replacing the tube in the cryostat. The orientation of the magnet on the tube is of no consequence.

B. Leak Detection

The simplest and most effective method of locating a leak in the cryostat is to pressurize the particular piece or tube suspected of leaking and submerge it in the pool. Do not excede 60 psig when checking tube 2 or any portion of that tube.

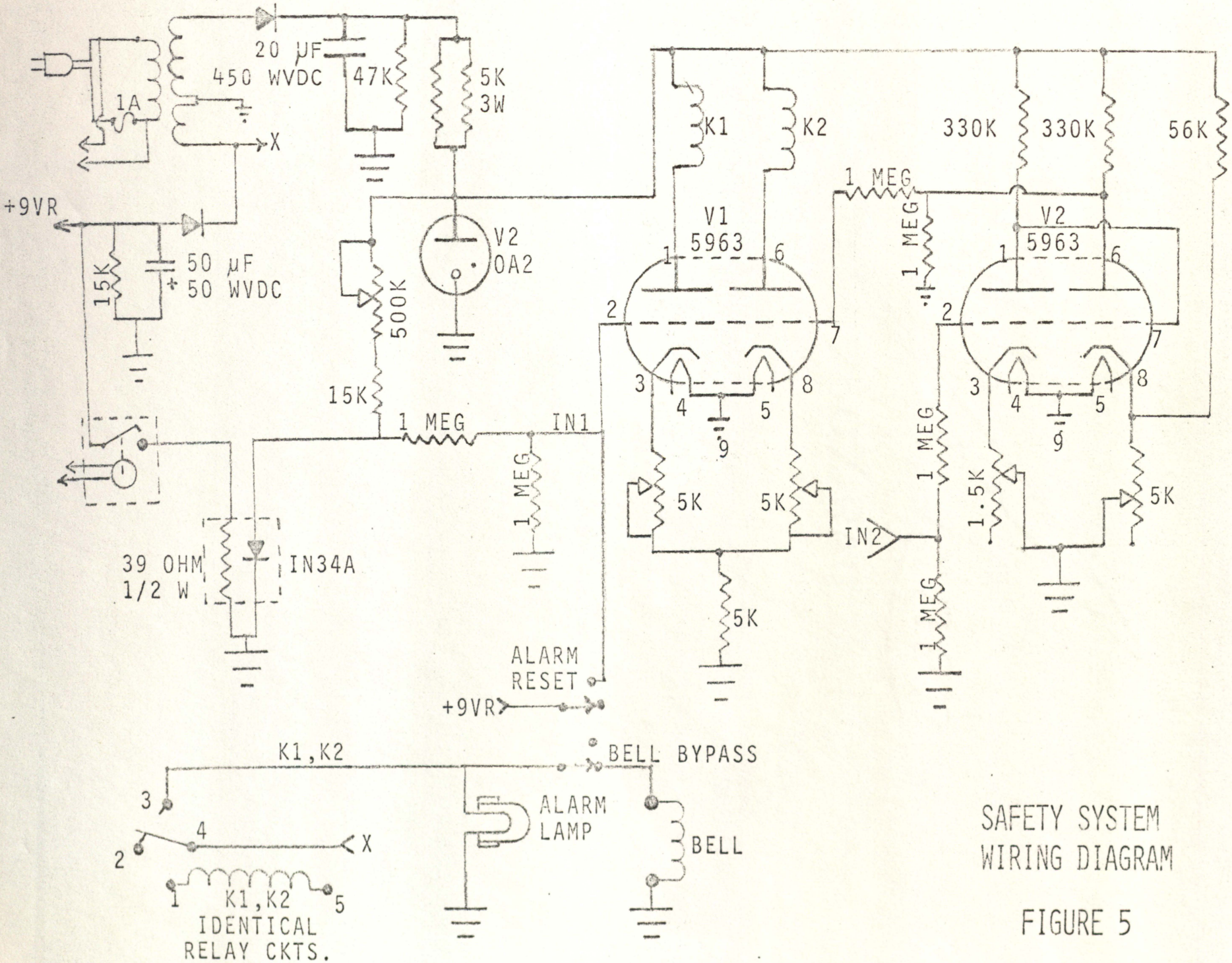
C. Wiring and Electronics

Figure 4 is a schematic of the control panel wiring. The fuses of the six pieces of power equipment are placed in the line before the respective indicating lights. Thus, if a fuse burns out, that indicating light will not work. Figure 5 is a schematic of the safety system.



CONTROL PANEL WIRING DIAGRAM

FIGURE 4



SAFETY SYSTEM
WIRING DIAGRAM

FIGURE 5

VII APPENDICES

APPENDIX A

CRYOSTAT START UP CHECK LIST

Date						
Operator						
Both power cords plugged in						
Rough Pump - oil level / operational						
Diff. Pump - operational						
SCRAM Button - ON						
Purge Pump - oil level / operational						
Purge Valve - operational						
Sample Chamber GN_2 Valve						
LN_2 Level Control						
LN_2 Safety Alarm on BYPASS						
Vac Safety on BYPASS						
Safety System Master ON						
LN_2 Safety System - light / bell						
Vac Safety System - light / bell						
Safety System Master OFF						
Operate Rough Pump 30 minutes						

Fan ON						
Diffusion Pump ON						
Operate Vac System $\geq$ 6 hours						
Vac Gauge to START for 1 hour						
Vac $\leq$ 20 microns						
Safety System Master ON						
Vac Alarm System ON						
Purge Sample Chamber 4 times						
LN_2 Dewar full & connected						
LN_2 Safety System ON						
LN_2 Level Control to AUTOMATIC						
GN_2 Valve open (OFF)						
Purge Valve closed (OFF)						
Purge Pump OFF						
Fill Sample Chamber to (GN_2 press)						

APPENDIX B

CRYOSTAT SHUT DOWN CHECK LIST

LN_2 level control OFF						
GN_2 solenoid closed (ON)						
Purge pump ON						
Purge valve open (ON)						
GN_2 to Dewar off - $\frac{\text{tank}}{\text{reg.}}$						
GN_2 line to Dewar disconnect						
Wait 30 min.						
Purge valve closed (OFF)						
Purge pump OFF						
GN_2 solenoid open (OFF)						
Sample chamber press.=0 psig						
Sample chamber GN_2 OFF - $\frac{\text{tank}}{\text{reg.}}$						
Safety system master OFF						
Vacuum gauge OFF						
Diffusion pump OFF						
Wait 5 minutes						
Fan OFF						
Rough pump OFF						
Scram button OFF (in)						