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NATURE OF THE INTERACTION BETWEEN ELECTRONS AND WELL-DEFINED SURFACES
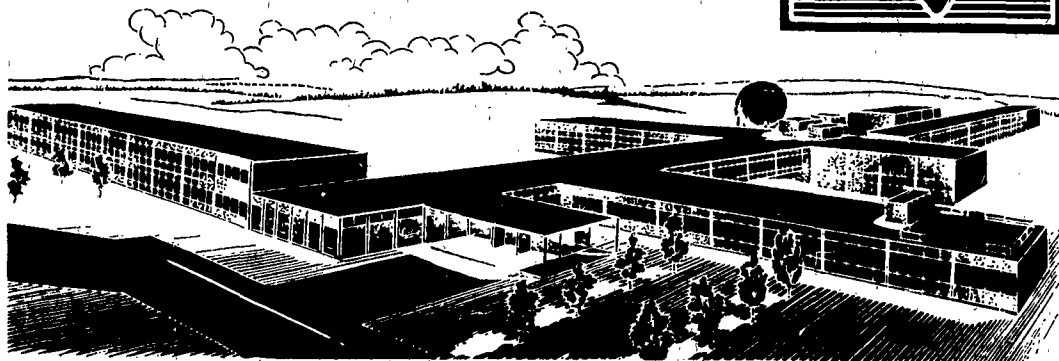
III. ULTRA-HIGH VACUUM SYSTEM AND SAMPLE OUTGASSING

IV. GAS SOURCE FOR THE ULTRA-HIGH VACUUM STUDY OF
ADSORPTION OF KNOWN-GAS LAYERS ON CLEAN SURFACES

E. L. KERN
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CHEMISTRY RESEARCH LABORATORY

MAY 1963

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FOREWORD

This report describes the equipment and techniques used to obtain ultra-high vacuum conditions needed in the study of clean surfaces. Its purpose is to inform scientists in the clean-surface research field of the techniques to be used in surface studies to be performed and described in the future. The work and planning described herein was performed between September 1960 and February 1963. This internal effort was performed at the Chemistry Research Laboratory of the Aeronautical Research Laboratories, Wright-Patterson Air Force Base, Ohio, under Task 702201, "Research in Physical Properties of Surfaces and Films"; Project 7022, "Surface and Interface Phenomena of Matter".

Mr. Waller L. Sands of the Aeronautical Research Laboratories' Glass Shop (AROSG) did much of the design and construction of the initial development of the main experimental vacuum system; his basic techniques are used to effectively obtain the ultimate vacua described.

Mr. Victor L. Johnson of Dayton, Ohio partially designed and totally constructed the gas handling system and modified main chamber components.

Parts I and II of this report series: I "Analysis of Surface Forces by a Helical Electron Beam" and II "Interaction Between a Small Cross-Section Helical Electron Beam and Uniform and Nonuniform Electrical Fields Perpendicular to the Helical Axis" describe one of the surface analysis efforts of the Surface Chemistry Group.

ABSTRACT (PARTS III AND IV)

The obtaining and maintaining of atomically clean surfaces is a necessary basic step in the study of well characterized surfaces. The initial part of such cleaning is to incorporate the sample and necessary measuring components in an ultra-high vacuum system, capable of maintaining a pressure of $1 \cdot 10^{-9}$ torr (1 torr = 1 mm Hg) to $1 \cdot 10^{-10}$ torr. This report (III) describes the means of obtaining these conditions. A double pumping arrangement, using a diffusion pump and a getter pump, with appropriate vapor trapping, valves and gauges, is described. Chamber bakeout and high-frequency heating of metal components is described. Equipment such as liquid nitrogen and oven controllers, which provide around-the-clock maintenance, is also described.

Studies of the influence of known-gas layers on a clean surface necessitates an ultra-high vacuum gas manifold system. In Part IV, the vacuum pumping, valving, measuring equipment and gas manifold are described. Ion gauges can be calibrated absolutely against a high vacuum McLeod gauge. By this system, capable of 10^{-9} to 10^{-10} torr, partial pressures of gases of the order of 10^{-12} torr can be introduced into the main testing chamber.

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III. ULTRA-HIGH VACUUM SYSTEM AND SAMPLE OUTGASSING

A. NEED FOR CLEANING THE SURFACE

How good should a surface be so that experimental results are reproducible, and are consistent with work of other laboratories? Modern vacuum technology and recent research on how to clean and produce undisturbed crystal surfaces permits this question an answer that has meaning. Surface research has been performed at high vacuum conditions (10^{-6} to 10^{-8} torr) for the last two decades, but the exact nature of the surfaces has not been known. Even if the surface--however one defines it--can be completely cleaned, we now know that it will be contaminated to the extent of one molecular layer in a time span of seconds. If reliable measurements are to be made, this changing surface condition must be controlled and eliminated. Therefore, either use of lower pressures or work with a consistently contaminated surface is required. But how reproducible is the contamination and what is the long range effect of the substrate and the contamination on the surface? This question has had the effect of invalidating most of the details of investigations which were performed at pressures of 10^{-8} torr or higher, the vacuum range of almost all work done up to several years ago. Reliable surface property measurements, if the few monomolecular layers at the surface of a solid are important, should be done in ultra-high vacuum (10^{-9} torr or better) and the surfaces should be monitored by techniques which can give surface conditions with sufficient reliability.

The ARL surface research approach to a clean, reproducible surface is to clean the surface and produce the surface structure to be studied; monitor this structure, maintaining the desired conditions for a sufficient time to measure surface properties with a high degree of reliability. Surface perfection should be within a small fraction of one molecular layer, since the phenomena being studied (see Part II of this report series) is a surface averaging effect.

The time required for measuring and monitoring is on the order of 30 - 60 minutes or more. At a vacuum of 1 x 10^{-10} torr, a period of 3 hours is available until the surface is contaminated (sticking coefficient = 1; for lower coefficients, the time is likewise increased), (Ref. 1). At 10^{-9} torr, only 15 minutes or so is available. There is currently much controversy over the value of sticking coefficient, but recent quantitative studies on clean nickel (Ref. 2) shows a coefficient of unity on the very clean surface, decreasing as the coverage increases toward 1 molecular layer.

Cleaning the surfaces requires not only heat (1000° C or less) and vacuum (Ref. 2) but auxiliary means to remove chemisorbed species and semiconductor or metal disturbed layers due to preparing the surface in the desired orientation and shape. Argon ion bombardment cleaning techniques to be used will be discussed in a later report.

The method selected to monitor the cleanliness or order of the surface is low energy electron diffraction (Ref. 3,4). This will permit observance of surface structure to a small fraction of a monomolecular layer (Ref. 5,6)

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over an appreciable area of the sample. The equipment, techniques and initial results of the low energy electron diffraction investigations will be described in future reports. In addition, contact potential measurements between the surface being studied and a reference surface will indicate reproducibility of initial surface conditions and small changes thereafter.

The following is a description of the ultra-high vacuum generating system, including the oven outgassing for the chambers and high frequency heating of metal chamber components.

B.   ULTRA-HIGH VACUUM AND PUMPING SYSTEM

A double pump system has been designed to handle the pumping requirements (speed, ultimate vacuum and maintenance of good operating characteristics) of the test chamber that will study electrical properties of atomically clean surfaces. This features a titanium ion getter pump (to obtain and maintain ultimate vacuum with minimum maintenance) and a 720 l/sec oil diffusion pump with appropriate ultra-high vacuum valves to do initial and outgassing pumping. This arrangement allows keeping the getter pump in prime condition through periodic outgassing and by alleviating the need for it to pump a large amount of gases or condensible gases. (See Figure 1).

The liquid nitrogen traps, valves and gauges are all designed for the low 10^{-10} torr range. At the time of planning, optimum vacuum technology components and techniques gave working vacuums in the 10^{-10} torr range. Recent techniques going to higher vacuums are generally not applicable in this experimental set-up. Very initial tests with some of the components on a glass chamber, a fraction of the current chamber's size, indicated a vacuum of 3×10^{-10} torr.

The ion-gettering pump can be isolated from the chamber by an indium-alloy-seat valve (Kane Engineering 1" I.D.) to allow pumping by the diffusion pump, while keeping the getter pump sealed at u.h.v. (ultra-high vacuum) (See Figure 2). The purpose of using the ion getter pump only at u.h.v. is to minimize equilibrium displacement of already adsorbed gas species, such as has been found with desorption of argon ionically pumped onto glass by bombarding krypton (Ref. 7), and thereby maintain a lower ultimate pressure. When the pressure is sufficiently low, the magnet is removed from the getter pump and the latter is heated by an oven (Litton Size No. 3 operating at 450° C). The trapped oil diffusion pump pumps on both the chamber and the getter pump. The chamber must be at $10^{-6} - 10^{-7}$ at the termination of baking in order to obtain ultimate vacuum in the 10^{-10} torr region. Since the getter pump is used for clean pumping, the gases from it should not contaminate the main chamber. The Kane valve can also be baked in a similar manner.

For initial operation the Vac-ion and main chamber are pumped with the oil diffusion pump (Consolidated Vacuum Corp. (CVC) Model PMC 720 3-stage fractionating using DC-704 silicone fluid) trapped by a liquid nitrogen cooled baffle (CVC Model BC-41 modified by mating Varian Associates 7" O.D. u.h.v. copper crush ring flange on top). After reaching 1×10^{-6} torr, the
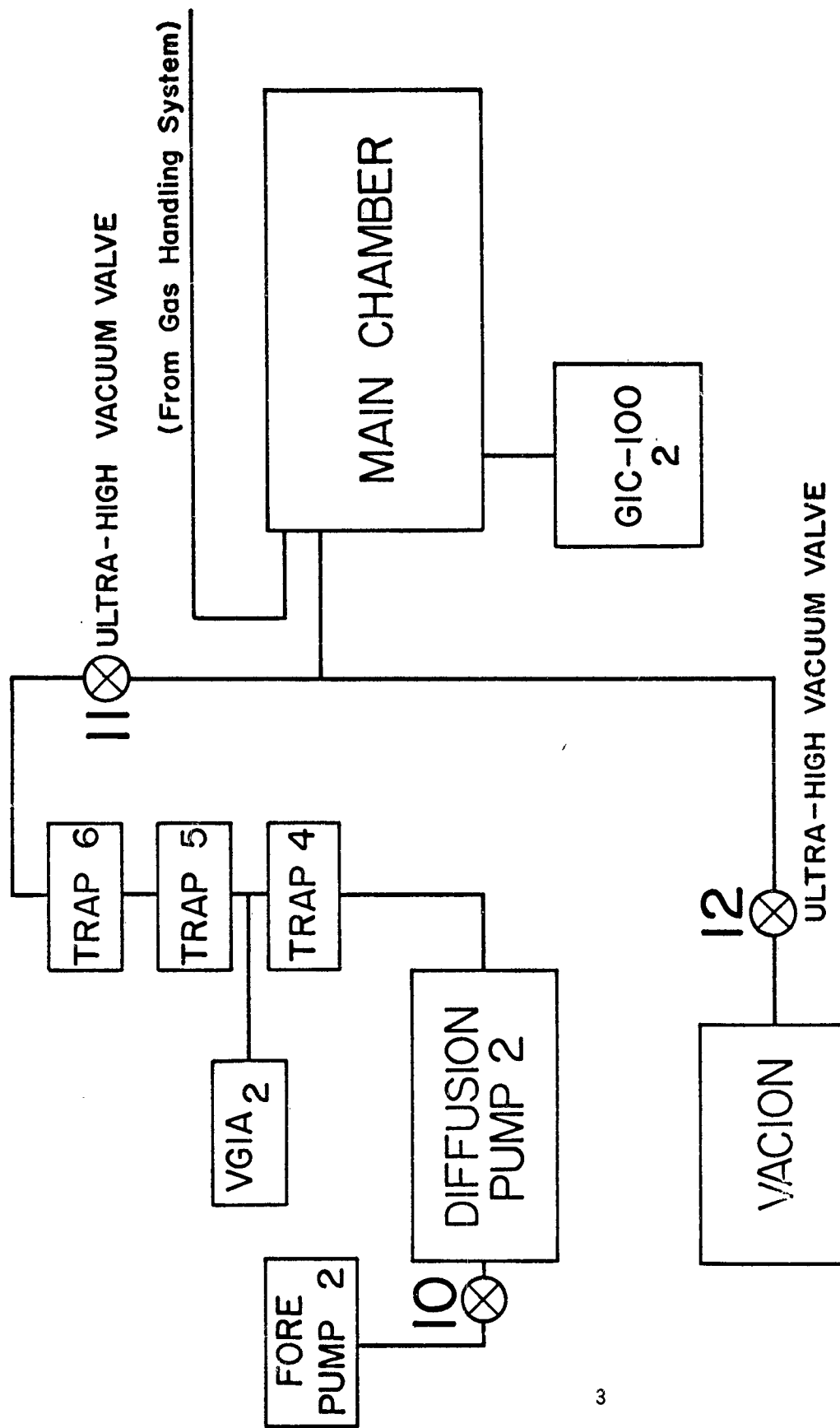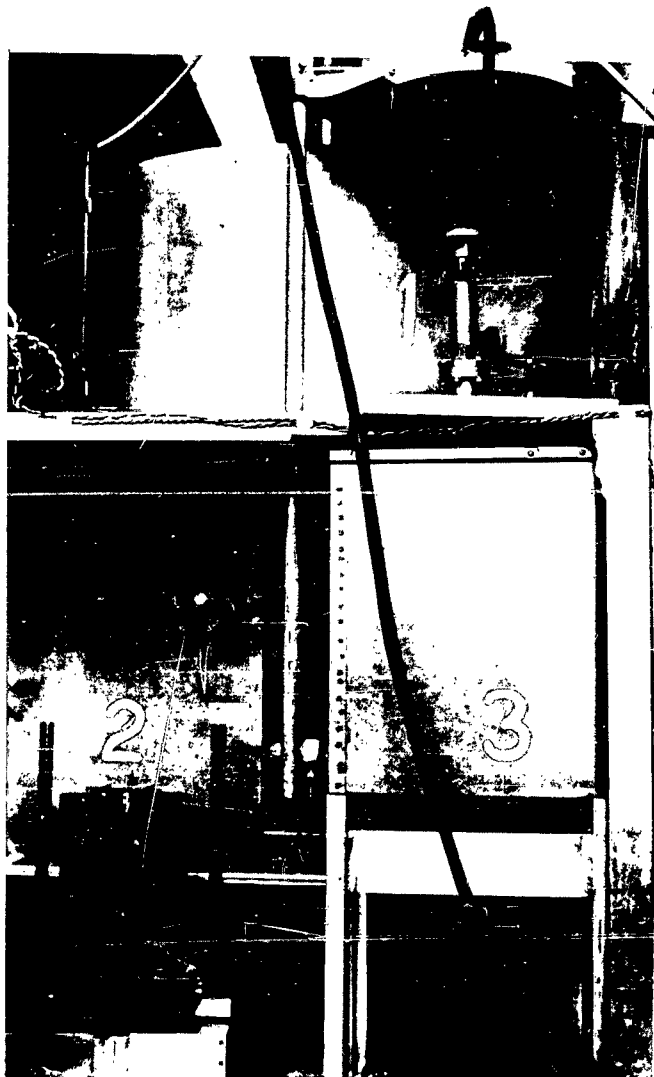
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Figure 1. Function diagram of the Main Chamber System.

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Ion-Getter Pumping Section

Figure 2.   Showing (1) Kane Valve; (2) Vac-Ion Magnet and jack; (3) Magnetic
Shielding inside of which is the Vac-Ion pump body. (Baking oven
or magnet can go inside shielding and around ion-pump). (4) Valve
Baking Oven

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chamber and Vac-ion, including Kane valves, are baked out. The chamber is baked at 450° C until 1×10^{-7} torr is obtained, with 1 hour at 480° C near the end of the period. This usually takes 48 - 72 hours. The Vac-ion and its Kane valve are baked at 450° C for a maximum of 5 hours, which automatically reflows the seat of the open Kane valve.

The chamber is allowed to cool, a second liquid N_2 trap is filled, and, afterwards, with the pressure indicated by the GIC - 100 gauge at 1×10^{-8} torr, the getter (tantalum) is flashed (at 20 amps). This reduces the concentration of O_2, CO_2 and CO in the chamber before switching to ion-pumping. During this period, the electron tube filaments are outgassed and high-frequency outgassing of metal components is performed (see page 7.) The tube is alternately flame fired between h.f. firings.

When the above initial conditioning is done, usually taking 4-7 days, the pumping is switched to the ion-pump for obtaining ultimate vacuum. The system is designed to obtain 5×10^{-10} torr or below, in order to facilitate 1 - 2 hours surface investigation time.

Farnsworth (Ref. 5) indicates that, at 10^{-7} torr, on germanium, a monomolecular layer of oxygen is adsorbed, (confirmed by Green) (Ref. 8), indicating that the outgassing procedures described should be capable of reducing surface contaminants to a monolayer. Analysis techniques will be able to study this at a later stage of the equipment development.

Vacuum Measuring

Foreline pressure is monitored by a thermocouple gauge (CVC GTC-004 measured by GIC-110 unit). The outgassing characteristics, as related to pressure changes, are noted by following the changes shown by the VG1A ionization gauge in the main bakeout oven, which is baked with the chamber. Operating in the 10^{-4} - 10^{-8} ranges (as measured by GIC-110 unit), the VG1A measures the pressure above the first liquid nitrogen trap (baffles), (See Figure 3). Vacuum of the main chamber is measured by a Bayard-Alpert gauge (CVC No. GIC-013) which is baked with the chamber and is turned on only after the VG1A indicates 1×10^{-8} or lower. Both of the filaments in the B-A gauge are outgassed during bakeout.

The two ion-gauge measuring units are connected to an automatic con-tinuous recording unit. The switching unit switches the inputs of the gauge unit concerned to the appropriate scale; i.e., to a more sensitive scale if the recorder is recording below 10% full scale or to a less sensi-tive scale when it reaches 100% scale. If the vacuum drops below 10×10^{-5}, the unit switches to the VG1A output and turns the GIC-013 unit off. If the VG1A indicates pressure higher than 10×10^{-4}, it is turned off and the diffusion pump is turned off. This circumvents lengthy, involved and seri-ous decontamination procedures in case the vacuum system develops a leak.

The Vac-ion can register its own vacuum, logarithimically to 1×10^{-8} torr or linearly to 1×10^{-9} torr. This provides some check on the GIC-013 at ultimate vacuum and also indicates how much gas volume is being pumped by noting the difference with the GIC-013.
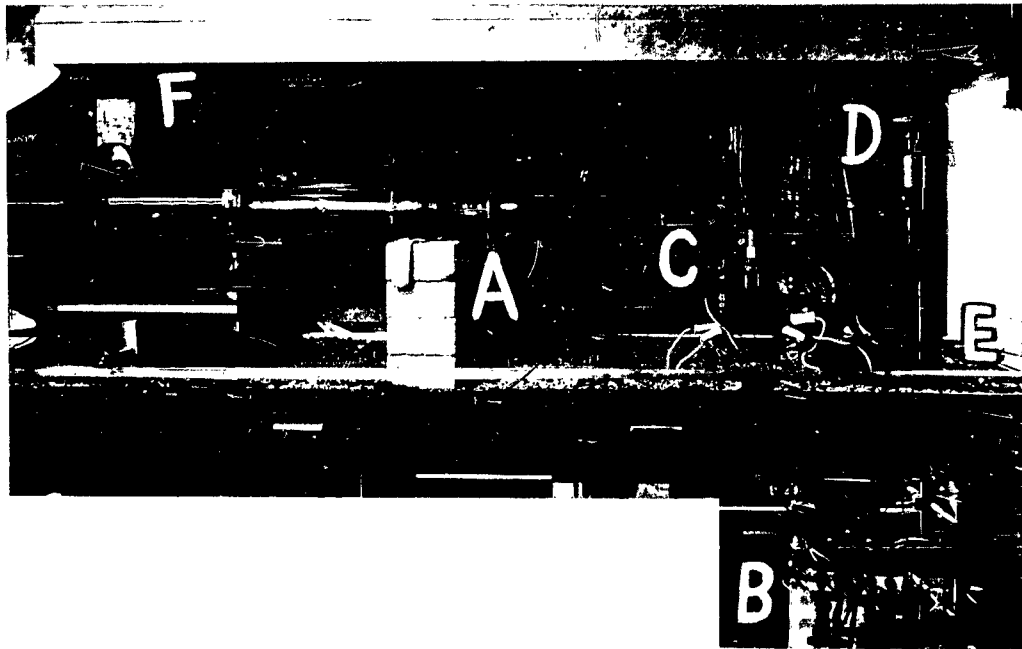
Experimental Chamber and Oil Diffusion Pumping Section

Figure 3.   An intermediate Helical Electron Beam tube with sample
            (A) is being pumped. The intermediate oil diffusion pump (B)
            was replaced by pump described on p. 4. Ionization gauge VGlA
            measures vacuum above pump and trap (C) and the Bayard-Alpert
            gauge (to right of VGlA) measures chamber pressure. Kane 1"
            valve (D) opens diffusion pumping system to the experimental
            chamber. Ion getter pump is below table at E and oven (F)
            lowers onto chamber for bakeout.

Liquid Nitrogen Traps and Supply

The effectiveness of a diffusion pumping system in reaching ultimate vacuum is dependent upon the ability to prevent the pump-fluid vapors from migrating into the vacuum chamber being pumped. At the temperature of the fluid which is pumping, its vapor pressure is the pressure in the system. Fluid vapors will condense on colder surfaces as they begin to migrate toward the chamber; this process depends on putting an obstacle before a particle with very long path length. An optical baffle is fairly efficient, but a cold optical baffle (at liquid nitrogen temperature) reduces the vapors by lowering their vapor pressure so that they are not free to move on. In addition to fluid vapors, decomposition of the fluid produces more volatile gases which must also be trapped. For these reasons, a liquid nitrogen trap can reduce ultimate pressure by two orders of magnitude. Additional traps in the line will increase trapping efficiency toward 100% and will allow warming of traps beneath it, permitting removal of accumulated adsorbed layers.

The first trap described in the previous section is a cooled, metal baffles. Above this, within the bakeout oven, is a double-walled, glass, spherical trap, in series with a glass cold-finger trap; these are baked with the chamber to ensure future efficient trapping. The ion-pump requires no traps since no fluid is used.

The liquid nitrogen is supplied to the traps from dewars with 25 or 50 liter capacity. Thermistors in each trap indicate level. When the liquid level drops below the thermistor, a relay circuit energizes solenoid valves, allowing the nitrogen to flow (see Appendix 2 for circuit diagram). The dewar is pressurized by its own evaporating gas, the pressure being controlled by a pressure switch (see Appendix 2). This system, which operates continuously, also supplies the Gas Handling System (Part IV; report following). Nitrogen consumption is approximately 25 liters/day.

Outgassing by Bakeout

In a wide range of temperatures (room to elevated temperatures) and of pressure (atmospheric to below 10^{-6} torr), many layers of gases are adsorbed on surfaces. When higher vacua are attained, these gases slowly desorb, except for the monomolecular layer at the surface, being thereby a source of gas inside a vacuum system. In addition to the surface adsorbed gas, volatiles (gases or atoms which can react to form gases in u.h.v.) lie inside the solid. The latter are reduced before the experimental tube is assembled by firing all accessible metal parts in dry hydrogen at elevated temperatures. To speed the process of obtaining ultra-high vacuum, these solids may be heated to supply necessary desorption and diffusion energies. For "Pyrex" glass systems with graded glass-to-metal seals, the maximum safe temperature is 480° C, while normally used maximum temperature is 450° C. Volatile impurities are driven from the u.h.v. side of the glass, water is entirely driven from ceramics and metals are fairly well outgassed. After suitable baking out, after which the pressure has dropped to 10^{-6} to 10^{-7} torr at these temperatures, and cooling of the system, the vacuum is in the 10^{-8} torr region. The more

complex the system and the less adequate the pumping system, the harder
it is to reach ultimate vacua. Therefore, in our systems, this bakeout
must be followed by alternately flaming the glass, and high frequency
firing the metal. (See next section).

The bakeout facility includes one large oven for the experimental
chamber, ion gauges, Kane valve, the diffusion pump, two cold traps and
getter, and two small ovens, one for the Vac-ion and one for its Kane valve.
The large oven is a Wilt Oven with a 60" x 17" x 20" cavity operating on
220 v.a.c. It is controlled by a Minneapolis-Honeywell off-on set con-
troller and an over-temperature safety shutoff. (See Figure 4). The small
ovens are Litton cylindrical bakeout ovens operating on 220 v.a.c. with
temperature time-proportionating controllers and variable transformers
monitored by wattmeters to allow control of rate of temperature climb.
(See Figure 5).

Outgassing Following Bakeout

The vacuum existing after bakeout, subsequent cooling, and final
degassing of filaments is usually 10^{-8} torr. On previously degassed systems
this may be lowered to 10^{-9} with the very well trapped, diffusion pump.
Pumping is then switched to the Vac-ion which pumps to 10^{-10} torr. In the
$10^{-9} - 10^{-10}$ torr region, the gas sources can easily equal the pumping
capabilities unless further steps are taken in the outgassing of materials.
The glass envelope, traps, gauges, etc. can be further outgassed by slowly
flame heating the end farthest from the pump, and progressing toward the
pump, keeping the formerly heated part hot. Particular care must be used
around graded seals and feedthroughs, but two or three repeated applica-
tions will produce low 10^{-9} torr region, while reducing the gas sources in
the glass walls appreciably. Filling of the top two traps (the middle one
has been previously kept at a low level) will then bring the system into
the 10^{-10} torr region.

The size and complexity of metal parts in this vacuum system is a
large source of gas. The gas problem, including the oxidation of molybde-
num parts at elevated temperatures and vacua on the order of 10^{-4} torr,
can be greatly reduced by hydrogen firing components before assembly.
With the complexity of the system and the procedures for modifying and
assembling, and with the cathode structures, etc., ideal hydrogen firing
cannot always be carried out. Bakeout at 450° C (or thereabouts) cleans
the metal surfaces fairly well and speeds up diffusion of gases from their
volume, but they still constitute a large source of gas.

Reducing the gas in the metal parts may be done by high frequency (h.f.)
heating of the parts through the glass wall. The ability to heat depends
upon coupling the h.f. field to the metal parts, which is facilitated by
several specially designed coils. Due to complexity of the metal parts,
coupling is still not good to many of the sections. Hence, depending upon
the geometry and location with respect to the glass walls, metal part
temperatures can be raised from 450° C to 800° C. The largest metal
component is the two inch inconel tube, supported by Vycor plates that will
endure higher temperatures. A coil designed and constructed in the labora-
tory is very effective in heating this complex structure. Heating must be
done slowly so the pressure does not rise above 10^{-7} torr.
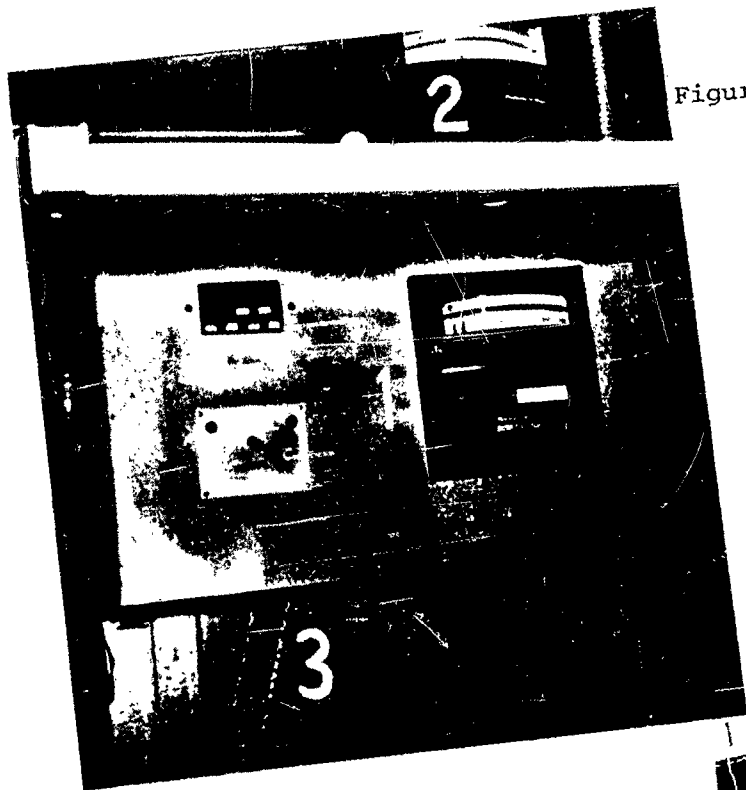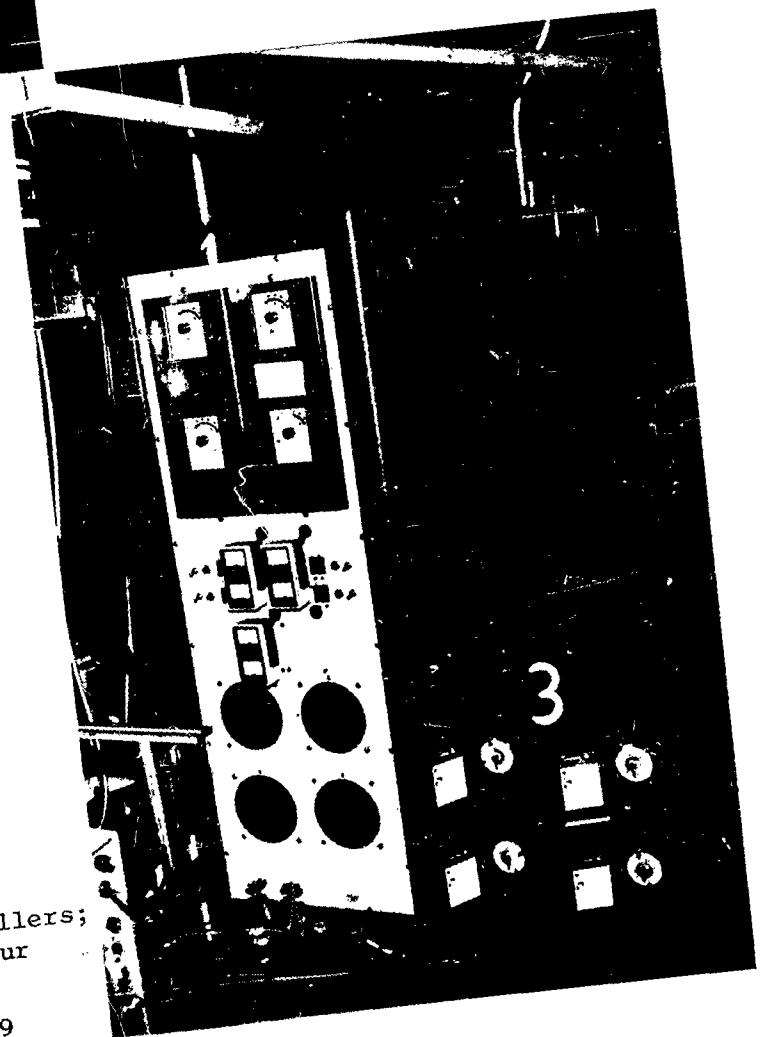
Chamber Bakeout

Figure 4.   Oven control panel shows
the (1) temperature con-
troller and (2) the oven
temperature protection
control. The oven (3)
lowers by chain movement.

Small Oven Control Panel

Figure 5.   (1) Temperature controllers;
(2) Variacs; (3) 24-hour
timers.

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The coils are powered by a radio-frequency generator (180 to 450 k.c. depending upon the coil and load being heated and the coupling to them)-- Lepel Model T-30-3, rated at 30 kw output power (See Figure 6). Grid and power controls, and an incrementally changeable oscillator tank which couples to the work coil facilitate good power output control and, therefore, good temperature control. Measurement of temperature of the heated metal parts will be approximated by a photometer and indirectly indicated by the sample temperature (after r.f. power is turned off). Controlling will be guided by pressure rise on the vacuum gauge.

The Lepel power generator is located close to the work coils and 8 ft. flexible water cooled leads are used. Distilled water is supplied to the generator and coils (to reduce maintenance and prolong coil and tube life) by a pressure water-recirculator (See Figure 7). Panel and foot controls allow pulsing operation of the coils to more effectively carry out out-gassing procedures. The power leads are held by plastic holders on the side of the oven in order to relieve strain on the glass chamber.

Alternate heating of the glass by flameout and the metal by r.f. heat-ing should drive gas out of the volume of both materials and sequentially onto the surface of the components not being heated. By alternate heating, each time to a lower temperature, the ill effect of one on the other should be reduced and the source of gases thereby be progressively reduced.

These outgassing procedures at 10^{-8} torr or below and extensive flam-ing out (especially with r.f. heating) should be sufficient to reduce the oxygen layer on germanium to less than a monomolecular layer. Farnsworth (Ref. 6) indicates that at 10^{-7} torr germanium has a monomolecular layer of oxygen (forms on clean surface in 25 minutes) and that it is removed by heating at 500° C for 30 minutes. Although flaming out will not raise the temperature of the sample that high, less than a monomolecular layer should adhere at, for example 350° C, if the layer is desorbed in any way comparable to oxygen on nickel (Ref. 2), for which 350° C lowers coverage to near 1/2 a monolayer.

C. SAMPLE OUTGASSING

All of the described outgassing procedures for the vacuum chamber should reduce inert gas surface layers or even strongly adsorbing gases to a mono-layer or less, as described in the preceding sections. Tenaciously held electrolyte or chemical etch or other strong molecular interaction product layers could be held to a monolayer or slightly more. Direct heating of the sample holder, thereby indirectly heating the sample, can reduce such layers to small fractions of a monolayer. Electron gun assemblies to do this will be incorporated in the low energy electron diffraction equipment and with the argon ion bombarding unit (actually, the annealling gun). These will be described with the future report on these components, and will be used in later stages of investigations.

Radio Frequency Generating Unit

Figure 6. Shows (1) the main con-
           trol panel; (2) a small
           work coil for induction
           heating of metal compo-
           nents within a glass
           walled vacuum chamber.
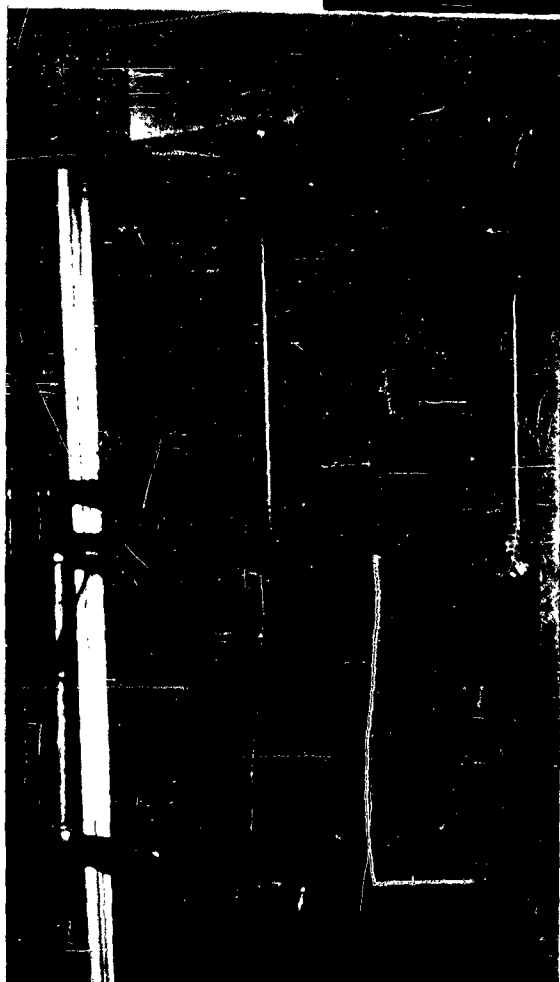
Closed Water Recirculator for R.F. Generator

Figure 7. Shows the closed-water-recircu-
           lating unit used to cool the
           generator and work coils.

IV. GAS SOURCE FOR THE ULTRA-HIGH VACUUM STUDY OF
ADSORPTION OF KNOWN-GAS LAYERS ON CLEAN SURFACES

Study of the variation of surface properties, such as energy and
force field investigations currently in progress, with adsorbed gases re-
quires a clean surface and an orderly way of putting controlled gas layers
on the surface. Procedures described in the last section and in future
reports (ion bombardment), allowing the atomic cleaning of the surface and
the work function determinations coupled with low energy electron diffrac-
tion (both to be described later), permit analyzing the surface cleanliness.
A well-defined gas layer may be obtained by slow introduction of pure gas
at ultra-high vacuum. A very low partial pressure of the gas can be pro-
vided, enabling adsorption onto the surface as a function of this pressure
and the sticking coefficient of the gas for the type of surface and its
current surface coverage. The rate of adsorption can be followed by either
low energy diffraction or this technique coupled with contact potential
measurements.

The following sections describe the manner of introducing known pres-
sures of pure gases. An ultra-high vacuum (u.h.v.) system, separate from
the experimental chamber pumping system, is used with an ion gauge and the
means for calibrating the ion gauge. A gas manifold contains bottles of
a variety of gases of interest in the surface studies.

A. ULTRA-HIGH VACUUM PUMPING AND GAS MEASURING CHAMBER

An u.h.v. pumping system is coupled to a measuring chamber and to the
gas manifold and McLeod gauge for calibrating the ion gauges (see Figures 8
and 9). A CVC Model GF-25 three-stage, glass, fractionating diffusion pump
is used with Octoil-S fluid. This is trapped by a 2-liter capacity, double-
wall trap, above which is a VG1A ion gauge to monitor the progress of the
vacuum condition. Above this gauge (above the table) are two 1-liter
capacity double wall traps (see Appendix 2 for liquid nitrogen controls).
The VG1A is outgassed by its "degas" mode of operation and by heating with
a heat gun. The top two traps and gas measuring chamber can be baked by
pulling down a Litton Model 4 oven (for control description, see Appendix
4).

The measuring chamber is connected to the pumping system by a glass
conductance valve (see Figure 9) which is actuated by a magnetic positioner,
spherical joint (15 mm. throat) sealed in the vacuum line. The measuring
chamber is also connected to the bottle manifold and McLeod gauge through
a Granville-Phillips (G-P Type C u.h.v. valve).

The gas measuring chamber is mounted above the traps on a stainless
steel support and is capable of routine operation in the 10^{-9} - 10^{-10} torr
region. To provide the control needed when introducing a gas into the main
experimental tube a Granville-Phillips Variable Leak Valve is used. This
leak valve is capable of variable conductances ranging from 10^{-13} 1/sec to
10 1/sec. Variable leaks 1 and 2 permit a calculable amount of gas to be
admitted to the system. Variable leak 3 provides the means by which large
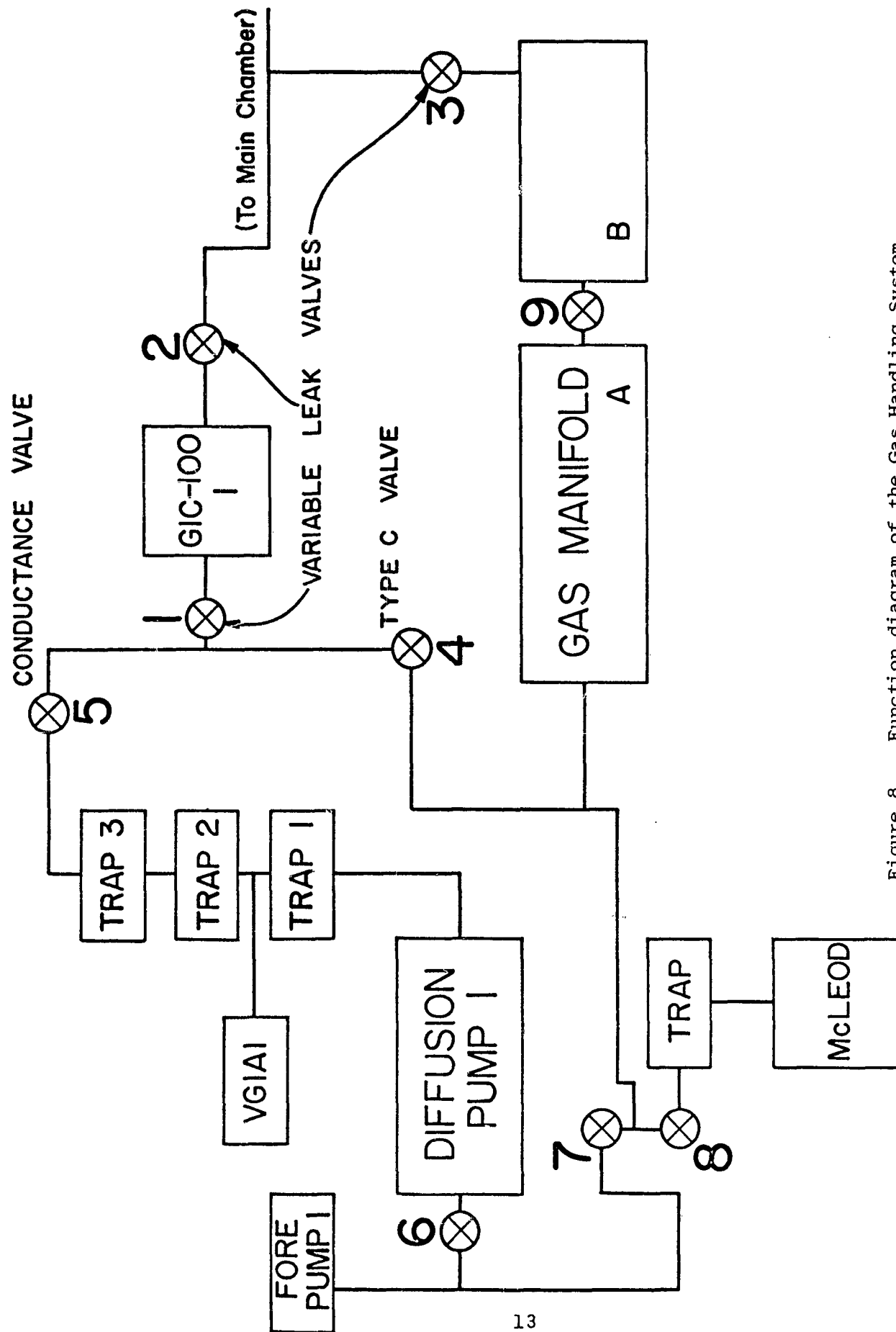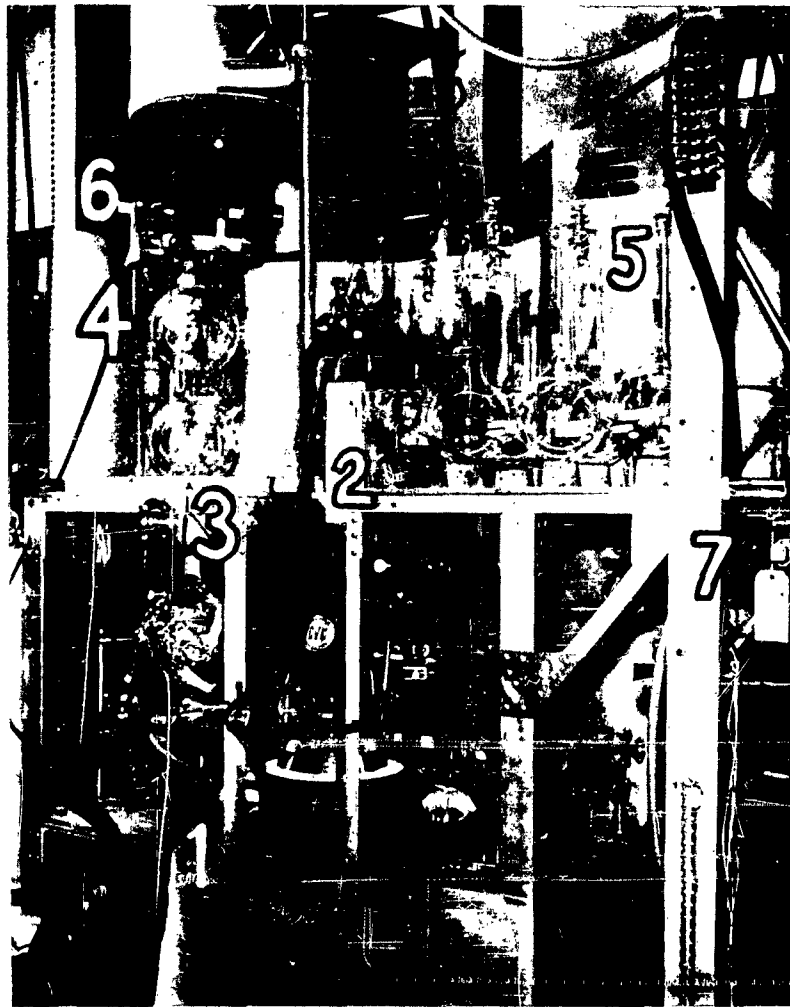amounts of Argon can be admitted to the main experimental tube when the

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Figure 8. Function diagram of the Gas Handling System.

Gas Manifold and Measuring System

Figure 9. (1) Diffusion pump; (2) McLeod Gauge; (3) Type C valve;
(4) Conductance Valve; (5) Gas manifold; (6) Ultra-
high vacuum measuring chamber; (7) Variable leak on
Argon manifold.

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Argon Ion Bombardment cleaning method is used. The relatively small opening of these valves allows the effective pumping speed of the system to u.h.v. conditions and the introducing of gas to and flow from the chamber, but the digital indicators of opening size are very useful in controlling flow to the main chamber.

A Bayard-Alpert ion gauge (CVC-GIC-013) measures the pressure in the chamber. Pumping by the gauge can be minimized by using pulse measuring techniques.

B.   ION GAUGE CALIBRATION

The ion gauges, and in particular, the measuring chamber gauge, can be calibrated for the gas being used. Factory calibration is for air and conversion factors are included for some of the gases used, but not all. A McLeod gauge (CVC-GM-110) is used as the absolute standard and can be read to an accuracy of from 2% in 10^{-4} torr range to 20% in the 10^{-6} to 10^{-7} torr ranges. A forepump is used to lower the mercury in the gauge. A cold finger liquid nitrogen trap prevents mercury vapors from contaminating the rest of the system and a h.v. stopcock closes the gauge from the manifold. When this gauge is opened to the manifold and the manifold opened to the measuring chamber, the gas pressure slowly reaches equilibrium in the volume between the McLeod and ionization gauges. By this means, meter readings can be calibrated with absolute pressure readings. Appendix 3 details the measuring procedure and calibration of the gauge currently being used.

C.   GAS MANIFOLD

The high vacuum manifold consists of 11 one-liter Airco assayed reagent gas bottles (see Appendix 4 for assay and manifold configuration) with impurities equal to or less than a couple ppm. Four argon bottles are separated from the rest of the manifold by a h.v. stopcock on one side and a G-P variable leak on the other side. This arrangement allows a relatively higher pressure of argon to be introduced into the main chamber, enabling the very low conductance of the measuring chamber to be bypassed when ion bombardment is being performed. The entire manifold is outgassed with a heat gun (blower type) and can maintain 10^{-6} torr for months. The gas from the bottles is introduced into the manifold by breaking the bottle breakseal with a glass enclosed magnet.

D.   INTRODUCING KNOWN GAS PRESSURES TO THE EXPERIMENTAL CHAMBER

Study of the effects of adsorbing gas on surfaces is facilitated by introducing known pressures of pure gases to the experimental chamber containing the sample through a variable leak valve. A desired partial pressure of the gas (of interest) can be introduced into the sample chamber if the pressure and volume of gas in the introducing chamber is known and the volume of the sample chamber. The introducing chamber pressure is measured by an ionization gauge (Bayard-Alpert type calibrated as described in B above) and its volume can be measured.

The volume of the sample chamber, V_2, can be determined by measuring the pressure of gas introduced from the introducing chamber described above. This pressure P_2 is measured on an ionization gauge (the partial pressure of the gas used in studies can also be measured by this gauge but the method of the last paragraph is used to predetermine it). At time (t_1) the gas measuring chamber has pressure and volume

(1) $P_1 V_1$ = const.

and the sample experimental chamber has pressure and volume

(2) $P_2 V_2$ = const.   (V_2 to be determined)

The two chambers are opened mutually to one another and equilibrium established:

(3) $P_1' V_1$ = V_1 $(P_1 + \Delta P_1)$ P_1' is the resultant pressure in the gas measuring chamber.

(4) $P_2' V_2$ = V_2 $(P_2 + \Delta P_2)$ P_2' is resultant pressure in the sample chamber.

Since P_1 was greater than P_2, the pressure in chamber (1) (gas measuring chamber) diminishes (ΔP_1 negative) and the pressure in the chamber (2) (sample chamber) increases (ΔP_2 positive). Since the moles of gas before and after equilibrium are the same:

(5) $P_1 V_1$ + $P_2 V_2$ = $P_1' V_1$ + $P_2' V_2$

Adding (3) and (4) and subtracting (5):

(6) $V_2 \Delta P_2$ = $V_1 \Delta P_1$

Since, from (3) and (4):

$$\Delta P_1 = P_1' - P_1$$

$$\Delta P_2 = P_2' - P_2$$

(6)   may be rewritten:

(6') $P_2' V_2$ = $P_2 V_2$ + $V_1 P_1' - V_1 P_1$

or the unknown volume of the sample chamber may be determined as

(7) $V_2 = V_1 \dfrac{P_1' - P_1}{P_2 - P_2'}$

An analysis of the size and shape of the gas measuring chamber gives a value for V_1 of 173.5 cc. The value for V_2 has yet to be measured and will be different for each different configuration of the sample chamber envelope.

Since the main chamber has roughly 20 liters (taking tube diameter 110 mm and 60 in. length and adding in for side arms), a pressure reduction of roughly 100 occurs between gas handling chamber and main chamber. If a partial pressure of gas in the measuring chamber is 1×10^{-8} torr, introduction into the main chamber results in 1×10^{-10} torr. By this means, pressures and ultimate vacua very easily obtainable in the measuring chamber correspond to very low pressures of the gases being used for study where very slow adsorption can take place. Of particular concern are the reactive gases where partial pressure in the main chamber should be below 1×10^{-11} torr. Residual reactive gases after extensive bakeout procedures should be kept below this value. This will allow very good determination of adsorption rates and characteristics.
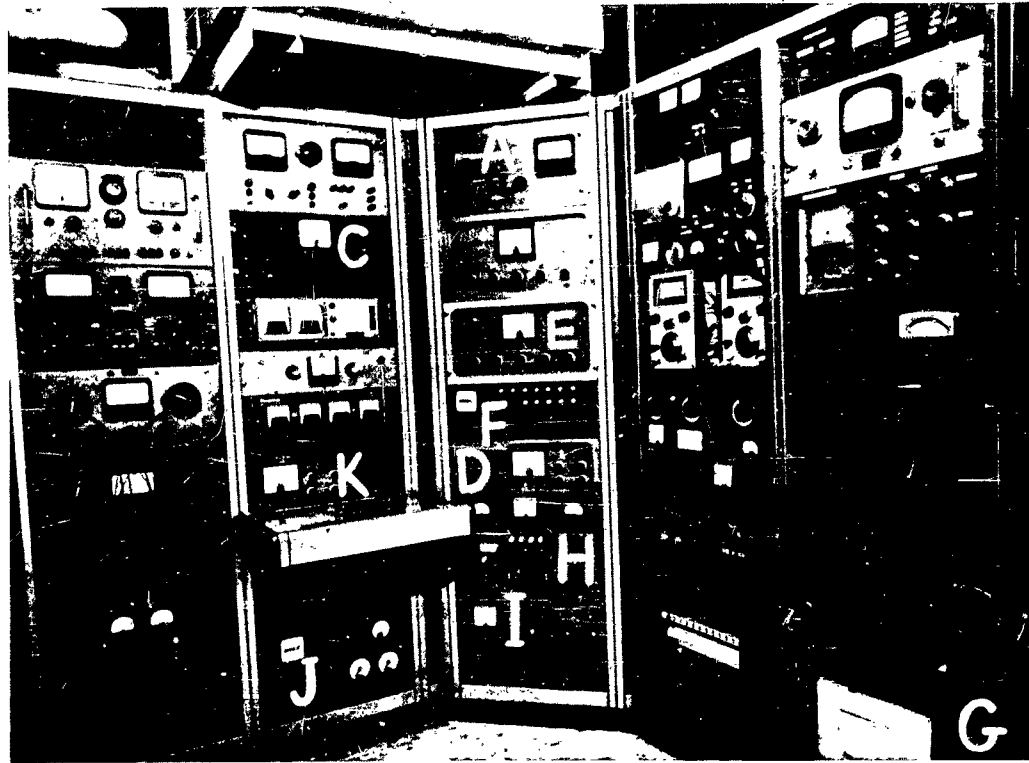
Main Electronic Control Panel

Figure 10.   A. Vac-Ion Control. Gas handling system gauges
VG1A (C), GIC-100 (B) and vacuum recorder (G).
Experimental chamber controls VG1A (D), GIC-100
(Bayard-Alpert gauge) (E) and automatic scale switch-
ing for recording (F). Diffusion and fore pump
controls (H) and outgassing getter control (I). An
automatic protection circuit (J) protects against
extensive damage to pumps. Preliminary test chambers
for electron gun development are measured by VG1A
Unit (K). Other panels provide control of the elec-
tron gun and analysis units.

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LIQUID NITROGEN FLOW CONTROL CIRCUIT

The automatic control of liquid nitrogen level in the vacuum system traps facilitates reliable 24 hour a day operation of the traps. The liquid nitrogen, pressurized in its delivery dewar by its own evaporated gas, can flow into the traps when the thermistor therein indicates a low level and the circuit opens the liquid line solenoid. Excess dewar gas pressure is let off by a pressure sensitive valve. The unique feature of this equipment is its ability to control and fill five traps simultaneously from the same dewar. A description of the control circuit follows.

Figure 11 shows the five parallel control circuits and the common power supply. Since constant current through the thermistros would not allow cooling, even when surrounded by liquid nitrogen, a cam timer interrupts thermistor power every 30 seconds for about 3 seconds (settable between 0 and 15 seconds). D.C. power (200 volts) goes through the timer, through the thermistor to a d.c. relay which activates 6 v.a.c. relays to control line voltage going to the liquid line solenoids.

Operation

The thermistor is placed in a liquid nitrogen trap. Power supply is turned on, the thermistor control switch is on, valve switch is on (see components list). When the nitrogen level drops to the point where the thermistor resistance drops, the thermistor current activates the d.c. activating relay 1 (2, 3, 4, and/or 5). This in turn activates relay 1a (2a, 3a, 4a, and/or 5a), which opens the solenoid valve 1 (2, 3, 4, and/or 5). The flow time is controlled by the cam timers. When the nitrogen level rises so that the thermistor resistance rises, the relays (1, 1a) and valve, are deactivated, cutting off the nitrogen flow.

Components List

        Power Supply - General Radio, Type 1204-B; output 6.3 VAC, 0-300 V.D.C.
        Cam timers - Industrial Timer Corp. Mod. MC-J2395
        Relays. (1-5) - Potter and Brumfield. KCP11
                (1a-5a) - Potter and Brumfield. KR11A
        Thermistors - Fenwal Type GB32J1
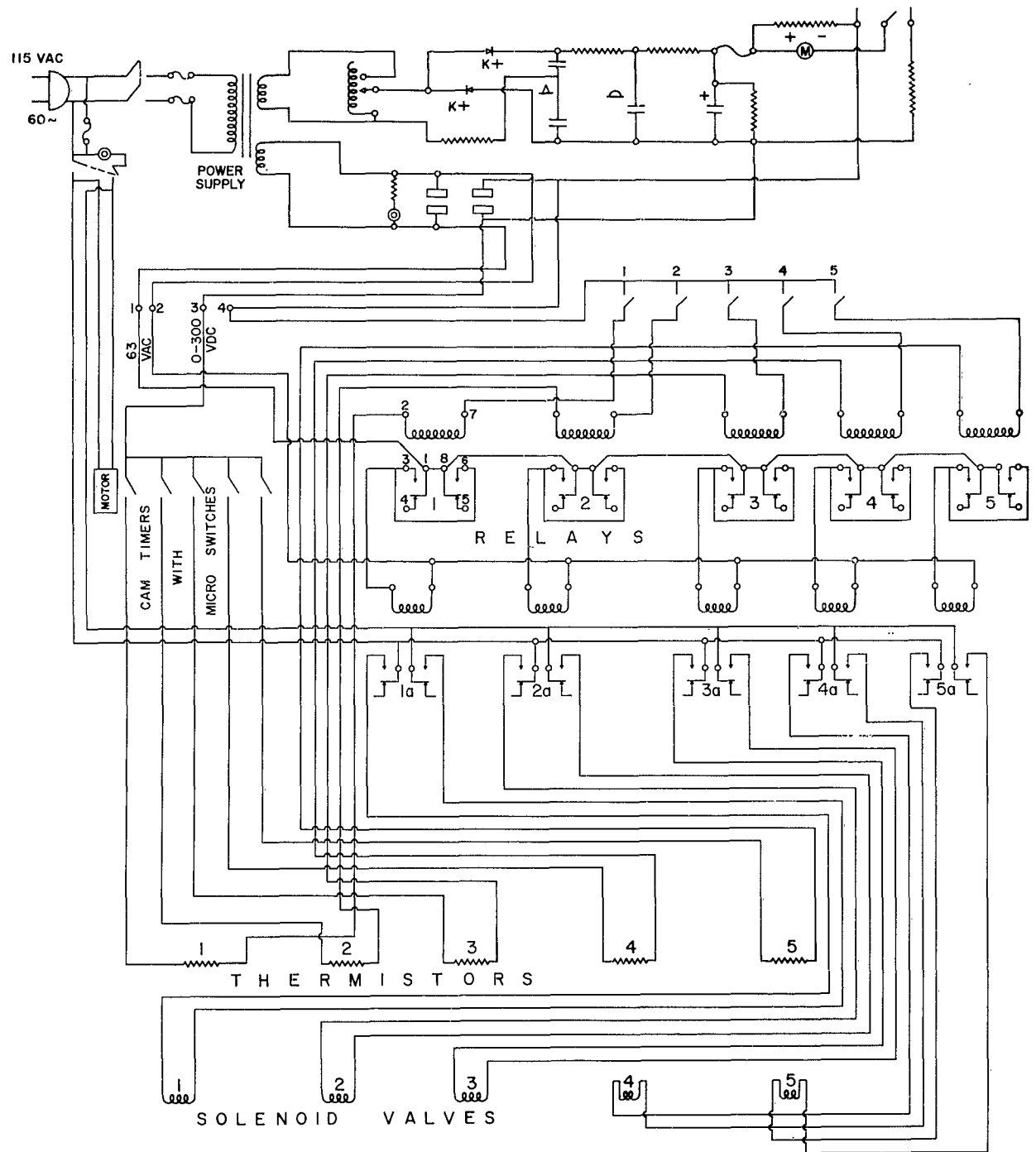   Solenoid Valves - Skinner V52DB2022; 115 VAC

Figure 11.   Nitrogen Control Circuit

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CALIBRATION OF IONIZATION GAUGES USING A MCLEOD GAUGE AS STANDARD

Calibration of Gauges in GHS

To determine the accuracy and reliability of the VG1A and Bayard-Alpert ionization gauges, the readings indicated by the gauges are compared with those of a McLeod gauge. Since the ionization current is dependent upon the ambient gas in the gauge, correction factors peculiar to the gas must be used. With both gauges, a manufacturer's list of correction factors is supplied. In the case for which the correction is unknown, it can be determined by noting the difference between the gauge reading and that of the McLeod. It is necessary to have pressure equilibrium in the system when the readings are made. Also, care must be taken to prevent mercury vapor from the McLeod from entering the vacuum chambers. This is accomplished by immersing a cold finger trap in liquid nitrogen (see Figure 9). The McLeod gauge, a CVC Type GM-110, used as a standard, is capable of measuring pressures of 4.10^{-3} to 1.10^{-7} torr.

Preliminary Steps Required Prior to Calibration

The preliminary steps applicable to both the VG1A and Bayard-Alpert gauges consists in the following:.

1. When the pressure in the systems is low, at a minimum value, the cold finger should be immersed in liquid nitrogen, and the McLeod opened to the rest of the vacuum system through valves 8 (stopcock) and 4 (Type C), the conductance valve (5) being normally open. (See Figure 8). The opening of valve 4 (Type C) should be done slowly, since the pressure gradient may be three or four orders of magnitude.

2. When the Type C (5) is fully opened and the pressure has once again dropped to a low value, the gas manifold lines should be flamed out, to remove any contaminants which possibly would invalidate readings.

VG1A Calibration

When the pressure is at a minimum, the calibration gas is let into the system from one of the gas bottles. The pressure as indicated on the VG1A is noted; then the absolute pressure is determined with the McLeod gauge. The two pressures are compared; the correction factors for the VG1A readings due to the type of gas used are determined.

Because the system is constantly pumping, it is sometimes difficult to obtain readings in the VG1A. Comparison of the two pressures, however, have been reasonably good.

Bayard-Alpert Gauge Calibration

When constant vacuum is obtained after the initial steps have been followed, valve 8 (the glass stopcock) is opened, valve 2 (variable leak) is closed, valve 1 (variable leak) is opened. The Type C (valve 4) is opened slowly so that the rise in pressure is small.

After valve 4 (Type C) is completely opened and the pressure is constant at a low value, the conductance valve (5) is closed and a gas is let into the system from the manifold. The Bayard-Alpert gauge is read when equilibrium has been established. The absolute pressure is then determined by the McLeod gauge. Comparison of the two readings is made, noting the correction factor for the Bayard-Alpert gauge for the gas used.

The calibrations made so far have indicated good agreement between the two gauges, i.e., agreement to less than meter and/or scale uncertainties of each gauge.

TABLE A2-I

TABULATION OF CALIBRATION DATA FOR BAYARD-ALPERT IONIZATION GAUGE

Pressure x 10^{-4}

Meter	Corrected	McLeod
6.4	4.2	4.1
5.4	3.5	3.5
1.5	.98	0.75
8.0	5.26	5.3
7.4	4.87	4.75
6.55	4.31	4.0
4.0	2.63	2.8
7.7	5.07	8.75
7.3	4.80	4.8
6.8	4.47	4.4
8.4	5.53	6.0
7.4	4.87	5.25
6.15	4.05	4.25
5.25	3.46	3.50
4.5	2.96	2.90
3.5	2.30	2.20
2.9	1.91	1.80
2.65	1.74	1.60
2.2	1.45	1.35
2.1	1.38	1.26

Correction factor is 0.658, as taken from manufacturer data for N_2 gas. Calibrations made on 25 and 28 August, 5 and 7 September 1962. Average uncertainty ± 4%.

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GAS MANIFOLD CONFIGURATION AND GAS ASSAY

The gas manifold supplies a known-composition gas to the gas handling system, which, in turn, introduces it to the sample chamber at a known pressure. The systematic study of the effect of a gas upon surface properties relies upon a minimum of contaminants in the gas, since different types of gases react to produce different surface products at different rates. The high vacuum manifold (10^{-6} - 10^{-7} torr) introduces assayed gas (see Table A3-1) into the u.h.v. chamber. The individual gas bottles may be easily replaced when empty or contaminated. Figure 12 shows initial configuration of bottles.

TABLE A3-I

IMPURITIES LEVELS OF GASES USED IN THE GHS MANIFOLD

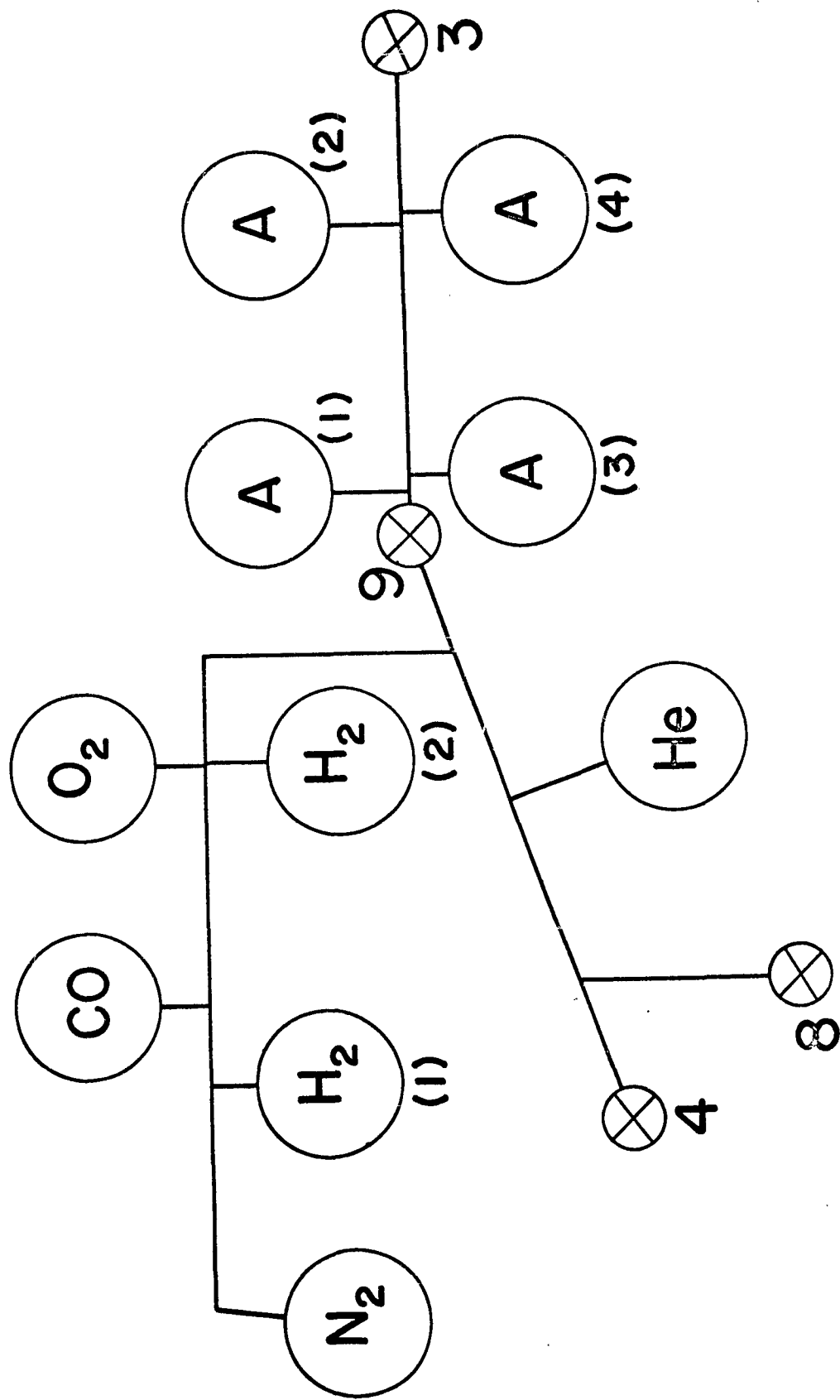| A	(1)	<0.0010 M% H_2;	<0.005M% N_2;	<0.005M% O_2
A	(2)	N_2 0.0014 M%		
A	(3)	N_2 0.0014 M%		
A	(4)	<0.0010 M% H_2;	<0.0005 M% N_2;	<0.0005 M% O_2
O_2		A_2 0.06 M%;	<0.60 M% CO_2;	<0.20 M% N_2; <1.5N% CO; 0.01 M% H_2
H_2	(1)	<0.005 M% H_2O		
H_2	(2)	<0.005 M% H_2O		
CO		0.2 %CO_2; 0.037% H_2; 0.0012% O_2		
N_2		None		
He		No Assay		

Figure 12. Diagram illustrating configuration of gas bottles of Gas Manifold.

SMALL BAKEOUT OVEN CONTROLS

A group of four small ovens provides a convenient bakeout facility for small parts of the GHS. Two ovens (Model 3) are $9\frac{1}{2}$" in diameter, 15" deep, two (Model 4) are 11" in diameter, 18" deep. Power to the ovens is regulated by a time-proportionating thermocouple-operated potentiometer-controller, with variable cycle. The circuit is designed for 24 hour operation and requires minimum operator attention.

Figure 13 is a function diagram of the control system. There are four such systems, one for each oven and each is independent of the other.

Components

    Variac: General Radio W20H Variac Autotransformer (220 v.a.c. 16 amps)
    24 Hour Timer: Intermatic Time Switch Model T103
    Mercury Relay: Ebert Electronics Type EM-4C and EM-4(2 pole 25 amp.)
    by 110 v.a.c.
    Temperature Controller: Minneapolis-Honeywell Model R7086A
        Potentiometer Controller (thermocouple operated)
    Thermocouple: Type J, Iron-Constantin
    Oven: Litton Model 3 and Model 4
    Volt-Wattmeter: Simpson Model 392: 0-1000 watts
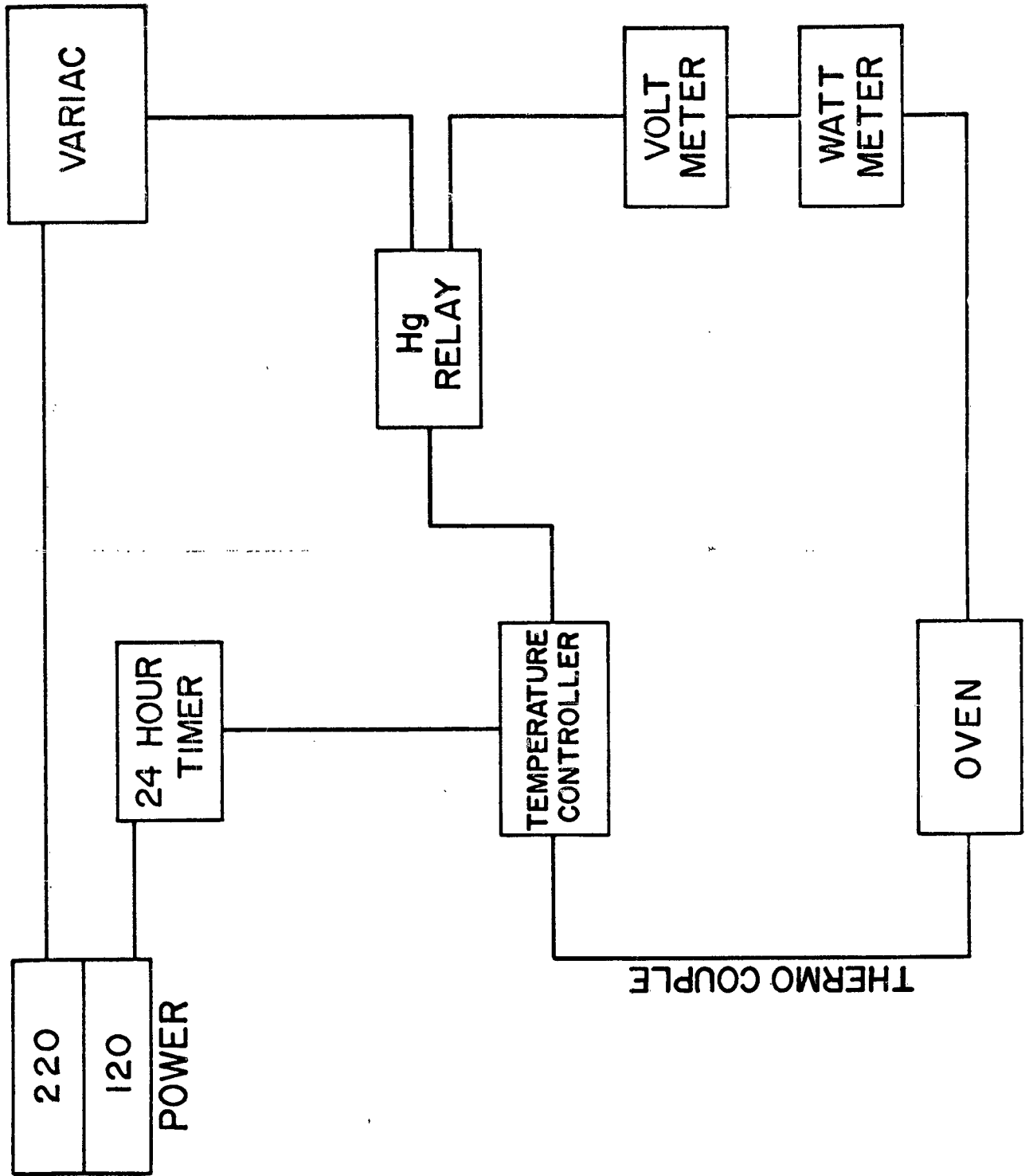   0-5000 watts

Figure 13.   Function diagram of Small Oven Control Circuit.

Aeronautical Research Laboratories,
Wright-Patterson Air Force Base, Ohio.
NATURE OF THE INTERACTION BETWEEN ELEC-
TRONS AND WELL-DEFINED SURFACES: III.
ULTRA-HIGH VACUUM SYSTEM AND SAMPLE
OUTGASSING. IV. GAS SOURCE FOR THE
ULTRA-HIGH VACUUM STUDY OF KNOWN-GAS
LAYERS ON CLEAN SURFACES, by E. L. Kern
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Laboratory, April 1963. 28 pp. incl.
illus. (Project 7022; Task 702201).
(ARL 63-80)

Unclassified Report

The obtaining and maintaining of
atomically clean surfaces is a necessary
basic step in the study of well charact-

(over)

erized surfaces. The initial components
in an ultra-high vacuum system, capable
of maintaining a pressure of $1 \cdot 10^{-9}$ torr
(1 torr $= 1$ mm Hg) to $1 \cdot 10^{-10}$ torr. This
report (III) describes the means of ob-
taining these conditions. A double
pumping arrangement, using a diffusion
pump and a getter pump, with appropriate
vapor trapping, valves and gauges, is
described. Chamber bakeout and high-
frequency heating of metal components is
described. Equipment such as liquid
nitrogen and oven controllers, which
provide around-the-clock maintenance, is
also described.