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LEAK DETECTION TECHNIQUE IMPROVEMENT
STUDY FOR SPACE VEHICLES

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January 8, 1967

Mr. C. P. McMurray, Purchasing Office, PR-EC
George C. Marshall Space Flight Center
National Aeronautics and Space Administration
Huntsville, Alabama 35812

Dear Mr. McMurray:

Attached is annual report 2-a submitted along with annual report 2-b (under separate cover) in fulfillment of requirement of Part B2, Article 1, of NASA contract number NAS 8-11199. These reports cover a period from April 7, 1965 to January 8, 1967.

As directed in Part B2, Article 1, aforementioned, this report in narrative form covers technical findings, studies performed, evaluation of results obtained, and conclusions and recommendations. The report also includes principles, proceedings, and methods of application that should be generally applicable to the utilization of the results of the study.

Sincerely yours,

R. C. Quisenberry

R. C. Quisenberry, Director
NAS 8-11199

Copy No. _____

Annual Report No. 2-a*

OHIO UNIVERSITY
College of Engineering and Technology

LEAK DETECTION TECHNIQUE IMPROVEMENT STUDY
FOR SPACE VEHICLES

An Investigation and Study

for the

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Contract NAS8-11199

Athens, Ohio
January, 1967

* This report consists primarily of the technical portion of the study. The literature search and automization of information part is presented under separate cover as Annual Report 2-b, dated September 1966.

ABSTRACT

A second generation model of the Halogen Gradient Detector using the dual-flow principle is described herein. After a long and extensive study of the state-of-the-art in leak detection methods, accompanied by considerable experimentation with locally-generated concepts, the hot anode platinum diode modified by the dual-flow principle meets the specifications dictated by the application to missile leak detection best of all competitive concepts. Considerable space is therefore allocated to this method, and to a theoretical and experimental study of the platinum diode.

The helium separation probe was designed, developed, and tested at the request of the Huntsville personnel for use with the mass spectrometer. The superiority of a teflon membrane over heated silica glass for the given specifications is demonstrated.

A 300 page separately bound library search report, along with a prepared set of approximately 3,000 IBM cards for rapid data retrieval of information connected with leak detection, accompany this technical report.

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CHAPTER I

INTRODUCTION

The work described in this report is a continuation of a task begun in January, 1962 under Contract NAS8-2563 and reported in the "First Formal Report" LEAK DETECTION TECHNIQUE IMPROVEMENT STUDY FOR SPACE VEHICLES in September, 1962, and also in the "Second Formal Report," LEAK DETECTION TECHNIQUE IMPROVEMENT STUDY FOR SPACE VEHICLES in June, 1963. In April, 1964, work was continued under contract NAS8-1119, and the first twelve months of research activity were reported in "Interim Report No. 1" LEAK DETECTION TECHNIQUE IMPROVEMENT STUDY FOR SPACE VEHICLES dated April, 1965. The material contained in this report emphasizes primarily the work carried out since the completion of "Interim Report No. 1".

Accompanying this report (under separate binding), is "Annual Report No. 2-b" dated September 1966, which covers an extensive search of the literature published prior to 1962 on the subject of leak detection and related fields. Included is a listing of over 3000 IBM entries and cross references. The report consists mainly of these abstracted articles, categorized into 97 subject classes and subclasses so that any particular type of leak detection information can be found readily. Each is catalogued on both title and author cards. A complete description is included in the aforementioned "Annual Report No. 2-b" which consists of approximately 300 pages.

While updating the literature search, the main task assigned to the Ohio University research group was to seek means of improving existing leak detection techniques and/or develop new approaches based on ideas generated in the course of the investigations. Many widely differing approaches were studied, tested, and reported such as acoustical detectors (including both active and passive methods), radioactive tracers, electromagnetic detectors such as the vibrating capacitor, thermoconductivity, and finally ionization detectors.

With the rather severe specifications dictated by the leak detection technicians regarding sensitivity, portability, fast response, low cost, health physics standards, non-corrosiveness of tracer, and many others, by far the most acceptable approach was that using the ionization detection of freon as a tracer gas. Consequently, a maximum effort has been expended in attempting to improve the existing hardware and to learn more about the ionization process itself. This has resulted in the design and construction of a working model using the time-sharing principle to detect minute freon concentration gradients. Included in this report is the design for a dual flow gradient detector which has demonstrated considerable advantages over the time-sharing model. The major gains consist of faster response, considerably simpler design, and lower production cost.

The radioactive tracer method is probably the most sensitive but must be discarded for the present in view of the rather severe existing health physics specifications. This method may be usable if these specifications are later revised when more extensive knowledge in this area has been obtained.

Acoustical methods have great potential, and if more sensitive detectors are developed, such as the transistor microphone, this approach would likely be competitive with the ionization detectors. It is therefore very important that the state-of-the-art information be updated continually so that breakthroughs in developmental engineering can be utilized at the first opportunity.

Another task attempted by our research group was the development of a suitable helium separation probe which would detect the presence of a helium tracer using an ionization gauge or a mass spectrometer. As described in a later chapter, both glass and teflon were studied and found to be acceptable membranes for separating helium from air. The Ohio University research group was then advised to concentrate on the development of a teflon probe since this material is an effective and fast helium filter at normal environmental temperatures. This probe separator has been developed and tested as reported below.

A leak detector testing facility has been designed and constructed for obtaining quantitative data using various leak detectors. This serves a valuable need in comparing different types of detectors on a rational and quantitative basis. For example, exact concentrations of freon and air can be obtained along with known values of freon concentration gradients. This facility is described in detail later in the report.

The thermal conductivity principle using the hot wire anemometer approach is discussed briefly. Reasons for discarding its use as a competitive leak detector are given. The main objections are its lower sensitivity, in addition to the disturbing effects of variations in flow rate.

CHAPTER II
AN EXPERIMENTAL AND THEORETICAL INVESTIGATION
OF THE HOT-ANODE PLATINUM DIODE

PARAGRAPH 1

INTRODUCTION

A method often employed to detect small leaks in missile plumbing involves the injection of a tracer gas into the air used to pressurize the system. A sensing device which will indicate the presence of the tracer gas is passed over the external walls of the system.

Freon ₁₂ (CCl_2F_2) and Freon ₂₂ (CHClF_2) are halogen-bearing gases that can be used as tracer gases in missile plumbing systems. The General Electric hot-anode platinum diode will respond to the presence of halogen-bearing gases by an increase in current flow from anode to cathode. With the diode at rated operating conditions and in the presence of ordinary air, this interelectrode current is of the order of a few microamperes. If the diode is placed in an atmosphere containing small concentrations of a halogen-bearing gas, this current will significantly increase. This increase is caused by an increase in the number of positive ions that are given off at the surface of the anode.¹

The diode is composed of two concentric right circular platinum cylinders. The anode, the inner cylinder, is electrically heated to a rated temperature of 800°C. A spacing of approximately .025 inches exists between the two electrodes. The anode to cathode voltage is usually 200 volts, but it can vary from 150 to 250 volts without appreciably effecting sensitivity. Figures 1 and 2 show the commercially

available diode and its component parts. Figure 3 is a schematic diagram of the diode circuit. A plot of measured values of anode to cathode current versus parts per million of Freon₂₂ in air, with the diode at rated operating conditions, is shown in Figure 4. The data points for this curve were obtained by measuring the current through the cathode circuit with a Keithley Micro-Microammeter when different Freon₂₂-air mixtures were passed through the diode.

Since the diode responds to a simple absolute level of the tracer gas concentration, precautionary measures must be taken to insure that ambient concentrations of tracer gas will not lead to false leak indications. The Electrical Engineering Department of Ohio University recently developed a time-sharing halogen gradient detector which seems to obviate the effects of high ambient concentrations.² This gradient detector utilizes the standard General Electric diode and a time-sharing input. Air samples are ingested through two probes at slightly different locations (left and right or fore and aft probes). The probes are connected to the diode through a gating valve so that the diode alternately accepts samples from each probe. The rate at which the gating valve switches is determined by the rate at which air samples can be passed through the diode without hampering the resolution of the detector. If the halogen concentrations sampled by both probes are the same, the diode will not exhibit a change in current when the valve switches. However, if one probe accepts a halogen concentration which is stronger than that accepted by the other probe, a change in current will result. This change in current is amplified so that it can be easily detected.



Figure 1. General Electric Halogen Diode.



Figure 2. General Electric Halogen Diode Shown Disassembled.

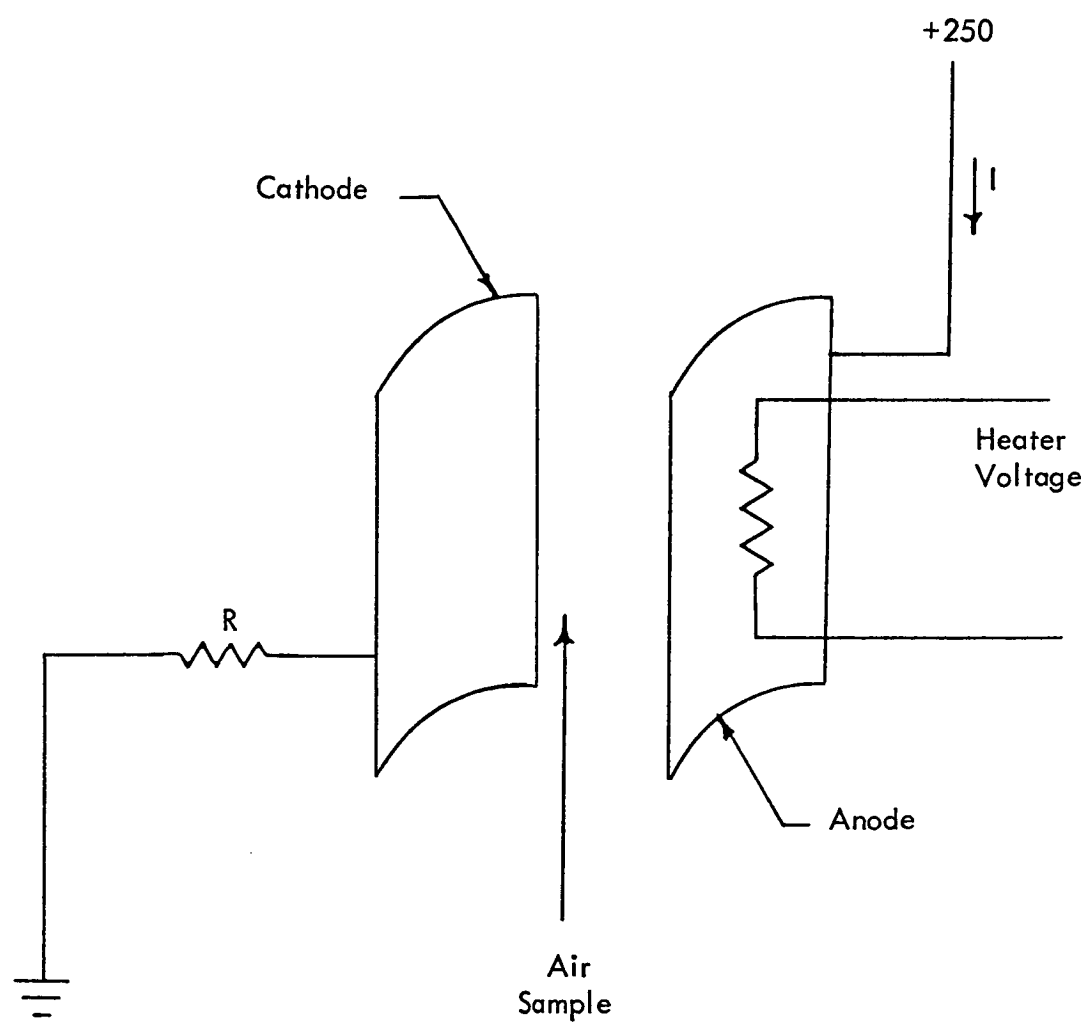


Figure 3. Schematic Diagram of General Electric Platinum Diode Circuit.

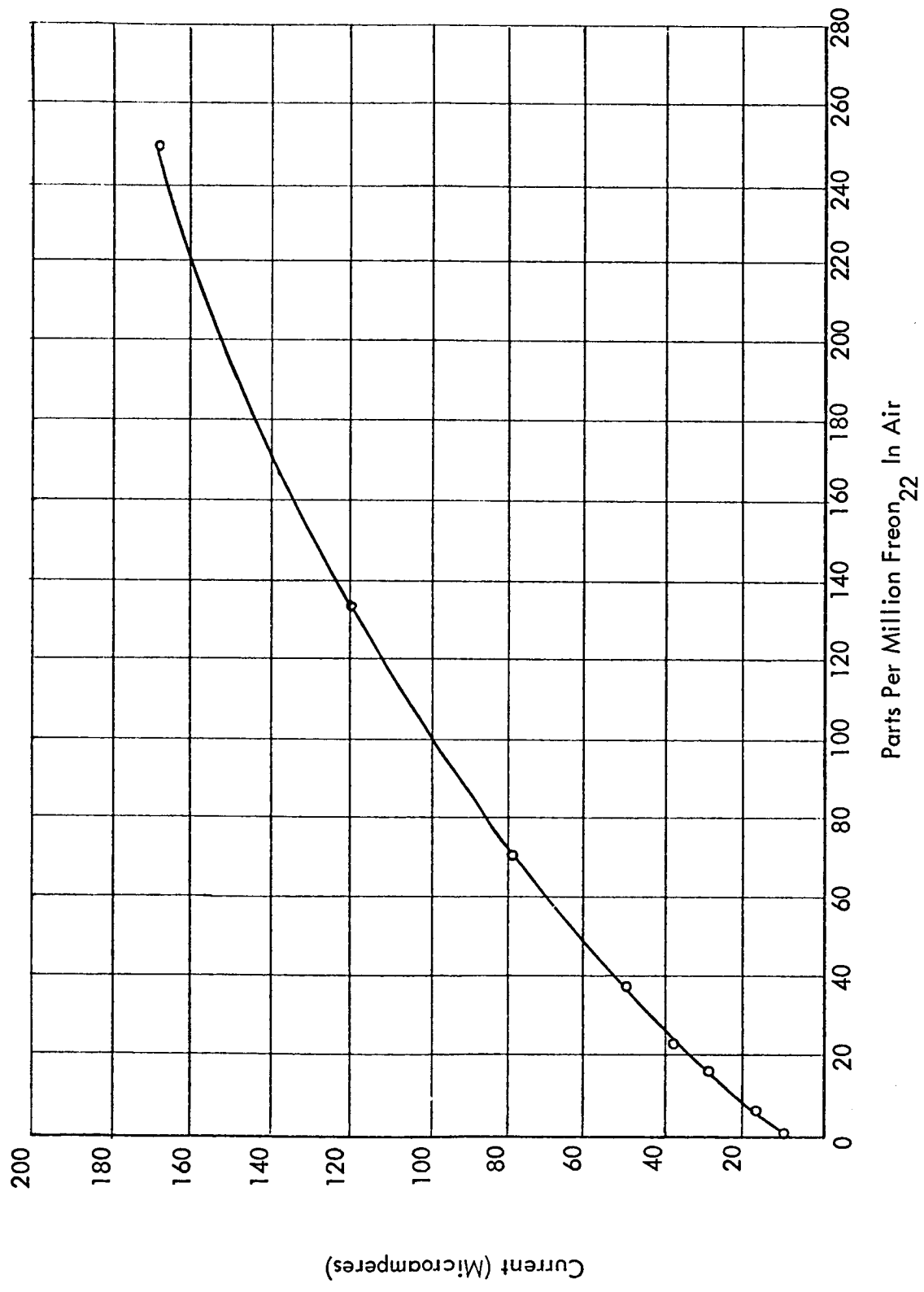


Figure 4. Diode Current versus Parts Per Million of Freon₂₂ in Air For Diode at Rated Operating Conditions

STATEMENT OF THE PROBLEM

The problem presented here is three-fold:

1. To determine a suitable theory of operation which approximates the behavior of the halogen diode.
2. To determine what type of current distribution exists along the axial length of the diode.
3. To investigate the feasibility of decreasing the response time of the time-sharing halogen gradient detector by appropriate modification to the present halogen diode.

The theory of operation is not completely known.⁹ A review of previous investigations on positive ion emission should yield some insight into the phenomenon that takes place when halogen bearing gases are introduced into the diode. The theory that is presented here is based on the Langmuir equation¹⁵ for ion emission and the change in work function that occurs when atoms or molecules of a gas are adsorbed on a metal surface.¹⁸

The second step of this study deals with the current distribution along the axial length of the diode. Information on this current distribution would be valuable if it was desired to alter the present geometry of the diode. Knowledge of this current distribution would also contribute to the theory that is presented. An investigation of a prototype diode should determine this distribution.

The remainder of this study is concerned with the low switching rate of

the gradient detector. The switching rate of the gradient detector has been experimentally found to be restricted to approximately 5 cycles per second. If a gradient of tracer gas concentration exists between the two probes, the resulting ac signal from the diode is limited to this same frequency. The net result is a long response time for the gradient detector.

One possible cause of this low switching rate could be the low flow rate of air (approximately 45 scim*)²⁰ through the diode. If this low flow rate is restricting the switching rate, an increase in the flow rate or a decrease in the interelectrode volume of the diode should enable the switching rate to be increased. Another cause could be that the response of the diode to halogen bearing gases is rather slow. When a Freon-air mixture is ingested by the diode, the interelectrode current will increase. If a significant amount of time must elapse before the current returns to the ambient level, the switching rate would be restricted. An investigation of these possible causes has shown that the factor which restricts the switching rate is the slow decay time of the diode current once halogen bearing gases have passed between the electrodes.

*Scim--The standard cubic inch per minute. The volume flowing in one minute converted to standard conditions; 68 degrees Fahrenheit and 1 atmosphere.

PARAGRAPH 2

POSITIVE ION EMISSION

BACKGROUND

The study of ion emission from metals is by no means new. During the early nineteen hundreds, much scientific work was done to investigate this phenomenon.⁴ Other early work involved the study of the ability of hot metals to retain both positive and negative charges. Atoms or molecules that come in contact with an incandescent metal surface may evaporate as neutral atoms or as positive or negative ions. It is also known that ion emission can occur in the absence of a surrounding gas.

When metals are heated in a vacuum, positive ions, negative ions, and electrons can be emitted depending on the temperature and impurities associated with the metals. At lower temperatures ion emission prevails and at temperature close to melting point of the metal thermionic emission of electrons takes place. Since a chief aim of this study is a theoretical explanation of the ionization process that takes place within the hot-anode platinum diode, the discussion will be restricted to positive ion emission and its peculiarities.

Considering the emission of positive ions from platinum and impurities found in platinum, a number of the behavioral characteristics are known.⁵ When platinum is first heated to a dull red, there is a weak emission of rubidium and caesium ions and a very strong emission of sodium and potassium ions. Regardless

of temperature, it has been found that none of the positive ions emitted were actual ions of platinum atoms.⁶ It is also known that the emission of positive ions falls off with time when platinum is heated in a vacuum and kept at a constant temperature. This decrease in emission usually takes on the form of an exponential with time and can be accounted for in the following manner. The maximum emission of positive ions from platinum is caused by sodium and potassium ions.¹² Since sodium and potassium are impurities in platinum, the formation of ions diminishes as these impurities are exhausted. The impurities below the surface layer reach the surface by diffusion and are then ionized. The initially high rate of emission is caused by the ionization of the impurities directly on the surface of the metal while the steady state level of emission is determined by the rate of diffusion of the impurities to the surface.⁷ These impurities reside in imperfections in the host metal and their diffusion rates are governed by electrostatic interactions between themselves and the atoms of the host surface and the spacing and relative sizes of the foreign and host atoms. If the metal surface is considered on an atomic scale, there can also be many cracks and valleys on the surface in which the impurities may reside. In order to take advantage of these defects in the host metal, various methods have been found to inject alkali metal compounds into a platinum surface.⁸ The ion sources obtained from these "doping" procedures have been found to retain a relatively constant emission capability over a long period of time. The anode of the halogen diode consists of a thin platinum cylinder which surrounds a ceramic covered heater element. The ceramic core is coated with alkali

metal impurities which diffuse through the platinum.⁹

DEVELOPMENT OF THEORY

In order to approach the problem of determining a suitable theory for the ionization process when halogen molecules are introduced into the diode, the simpler problem of determining the ionization process of the diode when placed in a vacuum will be considered. This theory is relatively straightforward and can be explained in the following manner. Considering the fact that the anode of the diode is a combination of host and impurity metals, two systems result which are in chemical equilibrium. For such a case, the Fermi energy, or chemical potential, is continuous across the system.¹⁰ This continuity means that a metallic crystal with impurities can be imagined to consist of nuclei with tightly bound electrons and conduction or valence electrons which are considered to belong to the entire crystal system rather than to any particular atoms. Therefore, the valence electrons of the alkali metal impurities can be considered to be free to move back and forth between the impurities and the host metal. The net result is a new Fermi distribution function for the entire system.

CONTACT IONIZATION

To explain this new Fermi distribution function clearly, it will be necessary to illustrate the host and impurity metals with the energy diagrams found in Figure 5.

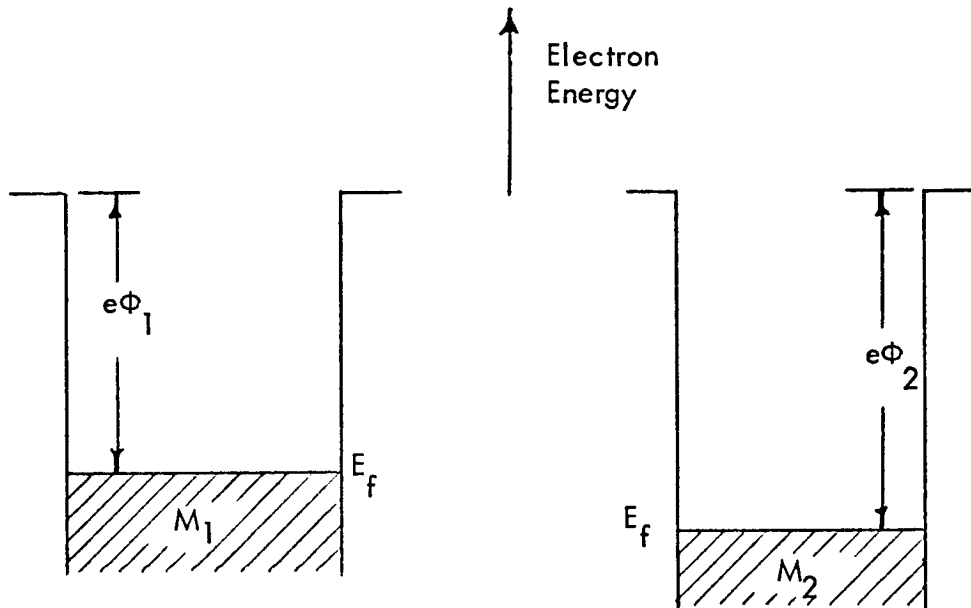


Figure 5 (a). Separate Metals.

where E_f are the Fermi Energy levels and Φ_n are the respective work functions. When the two metals are brought together at a constant temperature, the Fermi levels E_f must line up so that there is no energy gained or lost in transferring an electron from a state at the Fermi level in the impurity metal and a state at the Fermi level in the host metal.

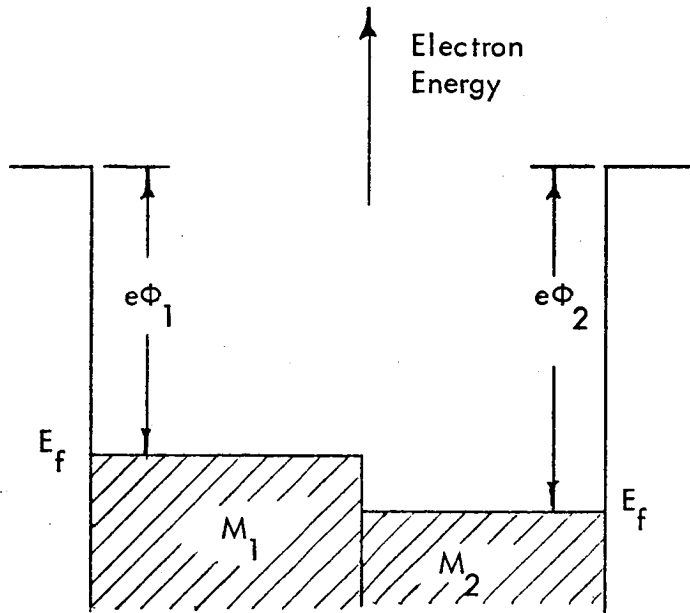


Figure 5 (b). When first brought together.

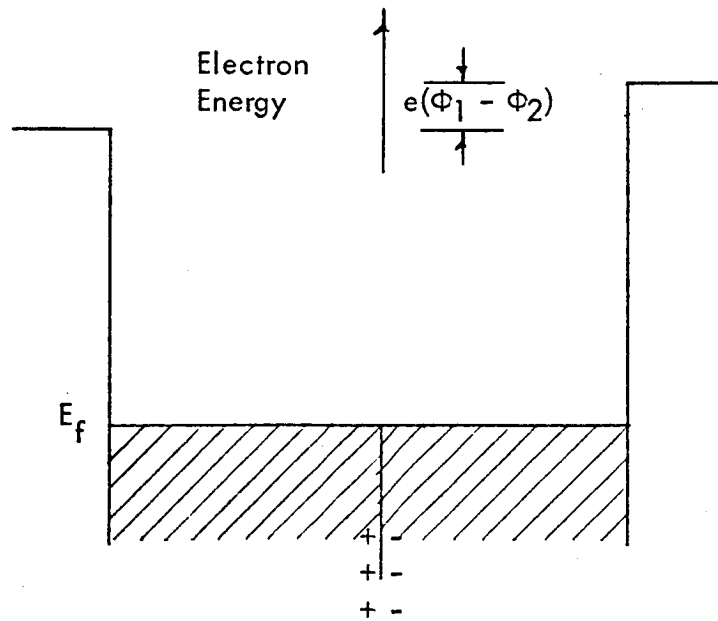


Figure 5 (c). At equilibrium.

A layer of charge is built up between the host and impurity metals which accounts for this lining up of the Fermi energy levels. When the temperature of the system is increased, the impurity atoms on the surface of the host metal begin to gain kinetic energy. As the kinetic energy of the impurity atom increases, the atom tries to leave the surface of the host metal. However, the valence electron of the impurity atom also has a force exerted on it by the platinum surface and it is pulled between the impurity atom and the platinum surface. Since the work function for platinum is 5.32 eV, the electron would have to gain 5.32 eV of energy to leave the surface. However, the ionization potential of an impurity such as potassium is only 4.32 eV. The net result is that the valence electron is lost to the platinum surface and the impurity atom becomes a positive ion. After gaining sufficient energy to overcome image field forces, the positive ion is then discharged from the surface. This phenomenon takes place whenever the work function of the host material is as great or greater than the ionization potential of the impurity atoms.¹¹ The probability that an impurity atom loses its outermost electron to the host material increases as the difference between the work function and ionization potential increases.¹¹ The increase in probability leads to the question of what alkali metal would be the most efficient in the ionization process. If the ionization potentials of the alkali metals are compared to the work function of platinum, it is found

Ionization Potentials

Li - 5.36 ev
 Na - 5.12 ev
 K - 4.32 ev
 Rb - 4.16 ev
 Cs - 3.87 ev

Work Function

Platinum - 5.32 ev

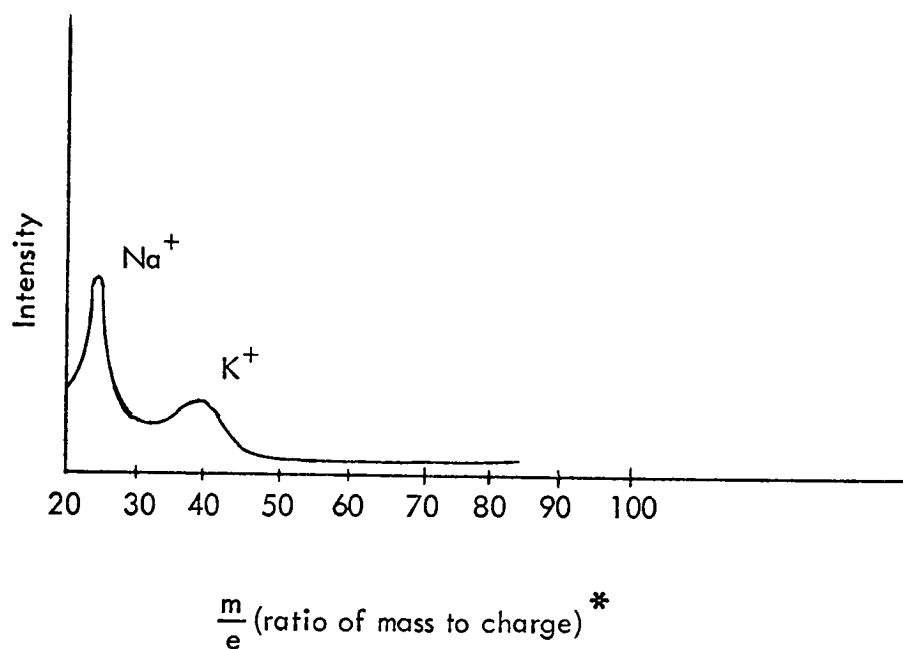
Ionization Potentials

Table 1

that caesium would have the highest probability of being ionized. However, since potassium and sodium are the chief impurities usually found in platinum, the emission of ions of these atoms usually prevails over all others. Of these two impurities, potassium has the lower ionization potential and should be more readily ionized than sodium. The emission of these ions has been supported by experimental observations performed by H.A. Barton¹² who found that most of the ions emitted from platinum were sodium ions with potassium ions comprising a substantial portion of the remainder. Figure 6 is a typical graph of intensity versus mass of the ions that are emitted from platinum.

One conclusion that can be drawn here is that a heavy "doping" of caesium impurities in the anode, coupled with the large amount of sodium and potassium atoms usually found in platinum, would lead to a relatively high value of positive ion emission. However, at low temperatures, approximately 1000°K and below, the impurity atoms tend to build up a coating on the surface of the anode which may consist of several layers of atoms.⁷ For atoms which are directly

in contact with the metal surface, the above theory holds and they become ionized. If a sufficiently large surface coating exists, these ions may not escape as ions but



* mass = Atomic mass number
charge = unit electron charge.

Figure 6. Mass spectrum obtained for ion emission from platinum (Barton¹²).

may be neutralized to atoms as soon as they leave the metal-impurity interface.²²

The fact that the ionization occurs when the work function of the metal surface exceeds the ionization potential of the impurity has already been stated in this section.¹¹ It also has been mentioned that when two systems (impurity atoms and host surface) come in contact with each other, their Fermi levels will line up with

each other causing a new Fermi distribution function for the composite system.¹⁰ If the impurities tend to form a sufficiently deep coating on the platinum surface, the net effect is that the work function of the platinum (5.32 ev) will be replaced by the work function of the specific impurity substance (1.60 ev for potassium and 2.26 ev for sodium). The ions formed directly on the platinum surface will tend to migrate through the remaining impurity surface layers. Since the composite surface now has a work function that is much less than the ionization potential of the impurity atoms, the ion will be neutralized as soon as it dissociates itself from the platinum surface. At higher temperatures, this surface layer is evaporated and simple contact ionization occurs as soon as the impurities diffuse to the surface.

At this stage in the development, it must be pointed out that the diode can be used to detect vapors of the alkali compounds which impinge on the surface of the anode. The effect on the work function of the platinum surface is the same as that of the impurities except that now there are more available atoms that can be ionized. The diode will develop an increase in current whenever the vapor passing between the plates has an ionization potential which is less than the work function of platinum.¹

EFFECT OF FREON

The diode has the capability of detecting a few parts per million of Freon in air. Comparing the ionization potentials of the most prominent molecules

present in a Freon and air mixture, it is found that they all have ionization potentials that are greater than the work function of platinum.¹

$H_2 = 15.42 \text{ ev}$	$O_2 = 12.20 \text{ ev}$
$H_2O = 12.6 \text{ ev}$	$CO_2 = 13.79 \text{ ev}$
$N_2 = 15.50 \text{ ev}$	$CCl_2F_2 = 12.5 \text{ ev}$

TABLE 2. Ionization Potentials

Also, the ionization potential of Freon is of the same magnitude as those of H_2O and O_2 . A phenomenon other than ordinary contact ionization of Freon must be present in order for the diode to possess such sensitivity.

In order to gain some insight into this phenomenon, it will be necessary to consider a number of known characteristics of the diode. First of all, the presence of the alkali metal impurities is necessary if the diode is to respond to halogen vapors.¹ Alkali ions and not halogen ions are emitted when halogen vapors are introduced into the diode. This fact leads to the conclusion that the halogens have some type of dislodging effect on the positive ions at the surface of the anode. Secondly, the molecules found in ordinary air do not have this same dislodging effect on the positive ions. Finally, when some gases are passed over hot metal surfaces they form a coating on the surfaces which has the effect of increasing or decreasing the work function of the surface. It is believed that this change in work function of the metal surface is the vehicle by which the dislodging effect takes place. To substantiate this theory it will be necessary to consider

the possible effects that Freon has on the diode.

When a halogen vapor is introduced into the diode, the molecules can either coat the anode directly or dissociate into atoms which coat the metal surface. Since it is known that Freon molecules dissociate when passed over metal surfaces at temperatures of 550°C or higher,¹³ the effects of halogen atom coating will be considered. The ionization potentials for fluorine and chlorine are given below.

Ionization Potentials

Chlorine	13.01 ev
Fluorine	17.42 ev

TABLE 3

The high values of these ionization potentials prohibit the possibility of positive ionization of these halogen atoms. However, there is evidence that a strong coating effect exists when chlorine or fluorine are passed over a hot platinum surface.¹⁴ The next step in this investigation will be a study of the effect of the halogen coating on an ion emission equation which approximates the behavior of the diode.

PARAGRAPH 3
EQUATION OF EMISSION

BASIC EQUATION

In order to develop a suitable equation of emission which will correlate with the observed behavior of the hot-anode platinum diode, the initial consideration will be a very simplified case of surface ionization. Suppose that the anode is not contaminated with impurities and that it is placed in a high vacuum. Rather than having the impurity atoms reach the surface by diffusion through the platinum, they will be directed at the anode in the form of a beam of incident atoms. If the impurity atoms impinge on the metal surface in this manner, the degree of ionization can be given by

$$\alpha = \frac{N_+}{N_o} \quad 1)$$

where N_+ is the number of positive ions emitted from the surface per unit area per unit time and N_o is the number of neutral atoms leaving the surface in this same time. The ionization coefficient is then defined as

$$\beta = \frac{N_+}{N} \quad 2)$$

where N is the number of atoms that impinge on the surface per unit area per unit time. If it is assumed that all the incident atoms reach thermal equilibrium at the

surface and none is reflected, it follows that

$$N = N_o + N_+ \quad 3)$$

Expressing the ionization coefficient in terms of the degree of ionization

$$\beta = \frac{N_+}{N} = \frac{N_+}{N_o + N_+} = \frac{\alpha}{1 + \alpha} \quad 4)$$

If the metal surface has a uniform work function over its entire area and if there is no external electric field present, the degree of ionization can be accurately described by the Langmuir equation.¹⁵

$$\alpha = \frac{N_+}{N_o} = \frac{g^+}{g^o} \exp \left[e \frac{(\Phi - I)}{KT} \right] \quad 5)$$

(where $\frac{g^+}{g^o}$ are the corresponding statistical weights of the ionic and atomic states).

e = electronic charge
 Φ = the work function of the metal surface
 I = the ionization potential of impinging atoms
 K = Boltzmann's constant
 T = absolute temperature.

The evaluation of Equation 5 yields some interesting results. The ratio of $\frac{g^+}{g^o}$ is equal to 1/2 for the alkali metal ions since the ion is found in only one state while the atom has two states (parallel or antiparallel spin of the valence electron).¹⁶ If the ionization potential of the impurity is less than the work

function of the metal surface, then the degree of ionization decreases with increasing temperature. This decrease seems strange at first since thermal energy is needed to counteract the image forces formed by the ions at the metal surface. However, referring to Figure 7 which is a plot of the ionization coefficient for caesium atoms on platinum, it can be seen that a certain threshold temperature must be surpassed in order for the equation to hold. For the caesium atoms on platinum in the absence of an electric field, the threshold temperature is in the range from 1000°K to 1200°K. Since this range is approximately the same as the operating range of the diode, this equation of emission must be modified in order to account for this threshold effect.

It is also important to note that this equation does not consider the variation of the work function of the surface with temperature and the fact that the surface may not be uniform but composed of portions which have work functions that differ with each other. Also, this equation was developed with the assumption that the atoms impinging on the surface reached thermal equilibrium before being desorbed.¹⁶ Neglecting the temperature variation and considering the fact that small differences in work function due to different crystalline planes on the metal surface will average out, the average work function of the entire system should be constant for the case described above. The assumption that the atoms impinging on the surface are not reflected and reach thermal equilibrium before being desorbed is reasonable for some systems and is the case for the diode under study since the atoms arrive at the surface by diffusing through the metal.

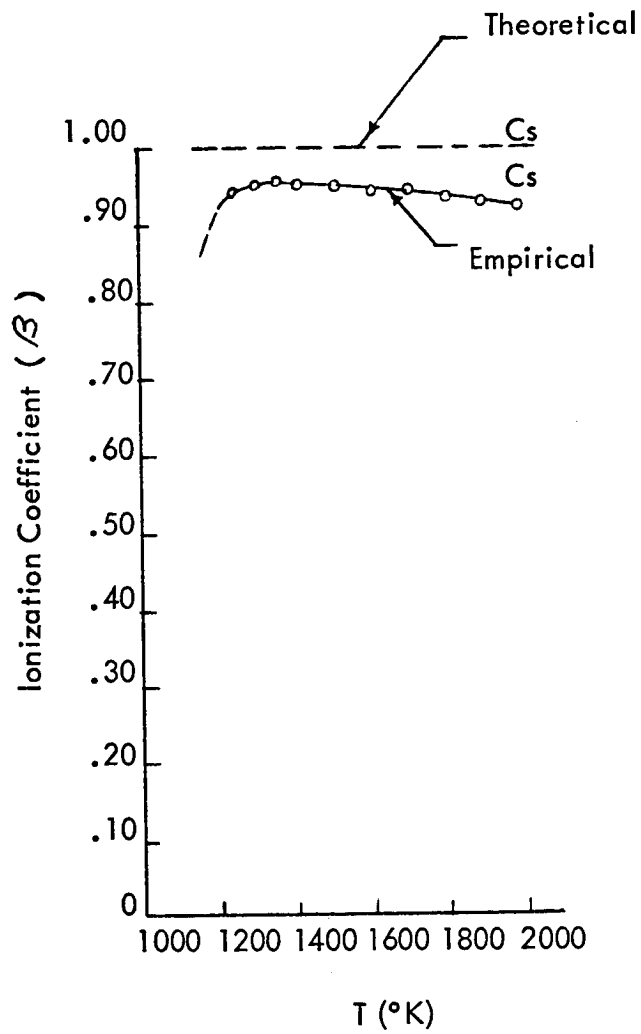


Figure 7. Ionization Coefficient versus Temperature of Surface for Caesium on Platinum. (Datz and Taylor¹⁷).

EFFECT OF ADSORBED SURFACE LAYERS

As mentioned in Paragraph 2, the impurity atoms will cause a surface layer of atoms to form on the platinum at temperatures near the operating range of the diode. In order to consider the effects that this surface layer will have on the emission equation, it will be necessary to investigate the adsorption phenomena that can take place when atoms or molecules impinge on a metal surface.

Essentially, there are two types of adsorption phenomena that can take place. The first of these is known as physical adsorption, or van der Waal's adsorption. It is the result of electrostatic interactions between impinging atoms or molecules and those of the metal surface. If the attraction forces exerted between the foreign atoms and those of the metal surface are strong enough, a surface layer of foreign molecules will be formed. For the case of atoms diffusing through the surface, the possibility of physical adsorption clearly exists. The atoms that tend to reside in the pores will have forces of attraction exerted on them that are much greater than for those which merely lie on a flat surface. Atoms that lie in deeper cracks, caused by imperfections in the surface, have by far the greatest binding forces exerted on them. In fact, the maximum binding forces are found to occur in positions where the adsorbed atom or molecule is surrounded by the maximum number of atoms in the metal lattice.¹⁶

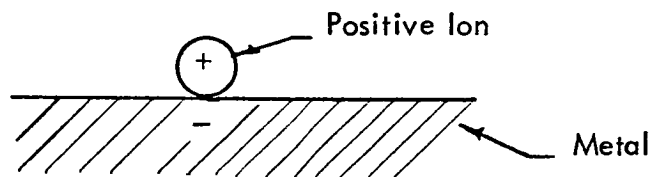
The second type of adsorption process is known as chemical adsorption or chemisorption which is the result of chemical interaction between foreign atoms

and the metal surface. When the adsorbed atom comes in contact with the metal surface, its electron shell may penetrate the electron shell of an atom on the metal surface. The result of this electron penetration is an exchange force or covalent force which weakly binds the atoms to the metal surface. The penetration is more commonly referred to as electron sharing between the two types of atoms. This type of chemisorption is known as weak chemisorption as opposed to strong chemisorption which will now be discussed. Strong chemisorption involves the actual transfer of an electron from one atom to another. If the work function of the metal surface is large compared to the ionization potential of the adsorbed atom, a valence electron of the adsorbed atom may be lost to the metal surface and a positively charged surface layer will result. The other possibility is that the work function of the metal surface is small compared to the ionization potential of the adsorbed atom. For this case, the surface may lose an electron to the adsorbed ion which will result in a negatively charged surface layer. Strong chemisorption results in stronger binding forces than van der Waal's or covalent adsorption and usually requires a much higher temperature for desorption of the accumulated surface layer.

ADSORPTION OF ALKALI METAL IMPURITIES

For the case of alkali metal atoms on hot metal surfaces, it has been found that the adsorbed film is a combination of both strong chemisorption and physical

adsorption.¹⁸ This combination of adsorption processes accounts for the atomic and ionic layers that were discussed in Paragraph 2. The physical adsorption being associated with the atomic layers while the chemisorption process is associated with the ionic layer directly at the surface of the metal. Since physical adsorption is the weaker of the two, the atomic layers will be desorbed at the lower temperatures thereby permitting the emission of positive ions to take place in the neighborhood of the threshold temperature. Suppose that the diode has reached a temperature where the physically adsorbed layers have been desorbed leaving only the strong chemisorbed bond (approximately the operating temperature of the diode). The



layer of positive ions and their image forces will form a dipole layer at the surface with a net result of a positive surface potential. This surface potential can be approximated by the following equation.¹⁸

$$S.P._1 = \frac{\sigma_1 \mu_1 \Theta_1}{\epsilon_0} \quad 6)$$

where ϵ_0 = permittivity of free space

σ_1^0 = the number of available adsorption sites/cm for alkali ions

μ_1 = the dipole moment of an ion-image combination

Θ_1 = the degree of coverage which can vary from zero to one.

Considering the combined system of the adsorbed layer and the metal, the work function will be lowered by the value of the surface potential.¹⁸ The maximum surface potential values for the adsorption of potassium and caesium on platinum have been found to be +3.68 volts for K and +3.76 volts for Cs.¹⁸ Changes in work function caused by adsorbed surface potentials can be applied directly to the Langmuir equation.¹⁵ If the change in work function caused by the surface layer is now taken into consideration, Equation 5 becomes,

$$\alpha = \frac{g^+}{g^0} \exp \left[\frac{e \left(\Phi_{\text{metal}} - \frac{\sigma_1 \mu_1 \Theta_1}{\epsilon_0} - I \right)}{KT} \right] \quad 7)$$

From Equation 7 it can be seen that for platinum (work function = 5.32 ev) and potassium (ionization potential = 4.32 ev) the surface potential need only be greater than 1.0 volts for the degree of ionization to become a fairly small value at the operating temperature of the diode. Also, values of slightly greater than 1.0 ev would indicate that the degree of coverage need not be close to one in order for the surface potential to have an effect on the degree of ionization.

Using an arbitrary value of 3 volts for the surface potential at the operating temperature of the diode and substituting the appropriate values for potassium on platinum, Equation 7 becomes

$$\alpha = 1/2 \exp \left[\frac{1.6 \times 10^{-19} (5.32 \text{ v} - 3.0 \text{ v} - 4.32 \text{ v})}{1.32 \times 10^{-23} \frac{\text{joules}}{\text{°K}} \times 1100 \text{°K}} \right]$$

$$\alpha = 1/2 \exp (-21.1)$$

8)

This small value for the degree of ionization could account for the small amount of ion emission with the absence of halogen at the operating temperature of the diode.

ADSORPTION OF A HALOGEN AND AIR MIXTURE

If a mixture of a halogen and air is now passed over the anode, portions of this gaseous mixture can also be chemisorbed on the surface. Since such a mixture would contain atoms of high electron affinity (the halogen and oxygen), the possibility for an electron to transfer from the surface to the adsorbed halogen or oxygen atoms clearly exists and the net result would be a very strongly bonded negative ion. If a sufficient number of these negative ions are formed, a surface potential opposite to that of the alkali metal ions would result.¹⁸ This surface potential can be approximated as

$$S.P._2 = \frac{\sigma_2 \mu_2 \Theta_2}{\epsilon_0} \quad 9)$$

where the terms have the same meaning as in Equation 6 but now, instead of being values for alkali metal ions they are values for the ion of the predominate electronegative gas. Since chlorine has the highest electron affinity of the

gases, this effect would be predominant.

Taking this second surface potential into account, Equation 7 becomes

$$\alpha = \frac{g^+}{g^0} \exp \left[\frac{e \left(\Phi_{\text{metal}} - \frac{\sigma_1 \mu_1 \Theta_1}{\epsilon_0} + \frac{\sigma_2 \mu_2 \Theta_2}{\epsilon_0} - 1 \right)}{KT} \right] \quad 10)$$

If the values used in Equation 8 are substituted in Equation 10 and an arbitrary value of 2.5 volts is used for the second surface potential, Equation 10 becomes

$$\alpha = 1/2 \exp \left[\frac{1.6 \times 10^{-19} (5.32 \text{ v} - 3.0 \text{ v} + 2.5 \text{ v} - 4.32 \text{ v})}{1.38 \times 10^{-23} \frac{\text{joules}}{\text{°K}} \times 1100 \text{°K}} \right] \quad 11)$$

$$\alpha = 1/2 \exp (+5.27)$$

This large value for degree of ionization could account for the increase in current when Freon-air mixtures are ingested by the diode.

At this point it must be clearly understood that this second surface potential would be a composite surface potential formed by oxygen and halogen atoms. As indicated above, the halogen atoms would have the most effect on this surface potential. If the value of this surface potential is large enough, the exponent in Equation 10 will become positive and the magnitude of the degree of ionization will significantly increase.

The possibility of the high electron affinity of the halogens being the

underlying cause of this increase in emission has also been suggested by W.C. White.¹⁹ However, this theory is merely a hypothesis since it has not yet been proven by experimental fact, but it does agree with the general operating characteristics of the diode.

EFFECT OF THE EXTERNAL ELECTRIC FIELD

The next step is to take into account the effect of an external field on ion emission properties. The purpose of the electric field is to accelerate the positive ions to the cathode once they are emitted. However, the electric field also alters the potential barrier at the anode surface so that the net effect tends to be a decrease in the surface potential.¹⁶

When a positive ion is released from the anode, the force that the surface exerts on the ion can be determined by the method of images. Figure 8 shows the electric field lines for a positive ion and its image charge.

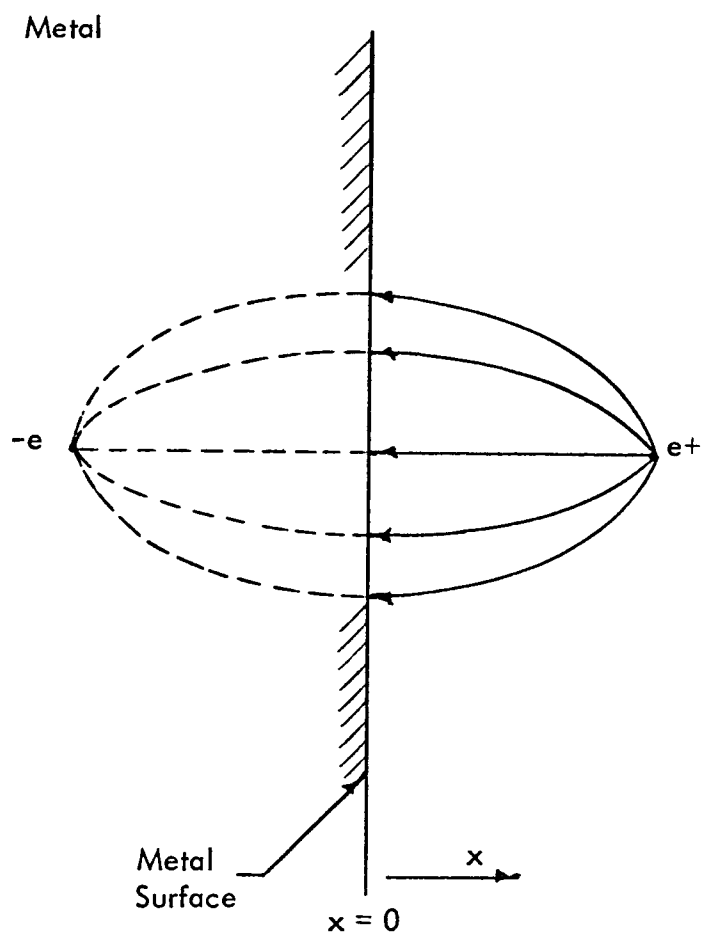


Figure 8. Electric Field Lines for a Positive Ion and Its Image Charge.

Employing classical electrostatic theory to evaluate this problem, the image force exerted on the positive ion at a distance x from the surface of the anode can be given as

$$F = \frac{(-e)(+e)}{4\pi\epsilon_0(2x)^2} = \frac{-e^2}{16\pi\epsilon_0x^2} \quad 12)$$

Where e is unit of electronic charge since the ions are formed by losing only one electron and ϵ_0 is the dielectric constant of vacuum. If we assume that the potential energy of this force is equal to zero when the positive ion is at an infinite distance away from the metal surface, the potential energy can be given as

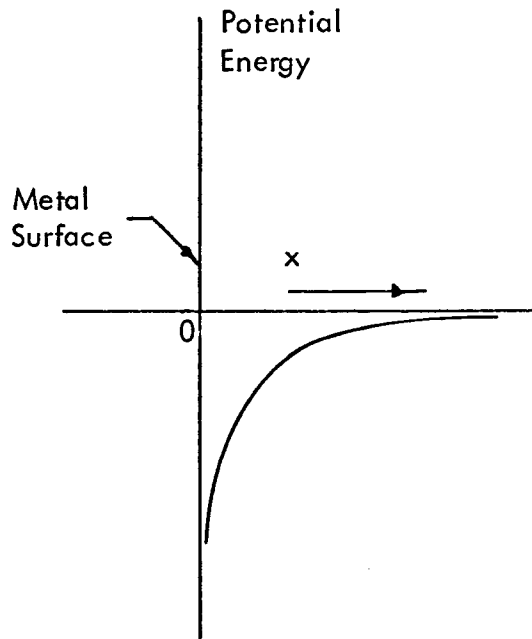
$$P.E._1 = \frac{-e^2}{16\pi\epsilon_0x} \quad 13)$$

The presence of an electric field gives the positive ion an added value of potential energy which can be given as

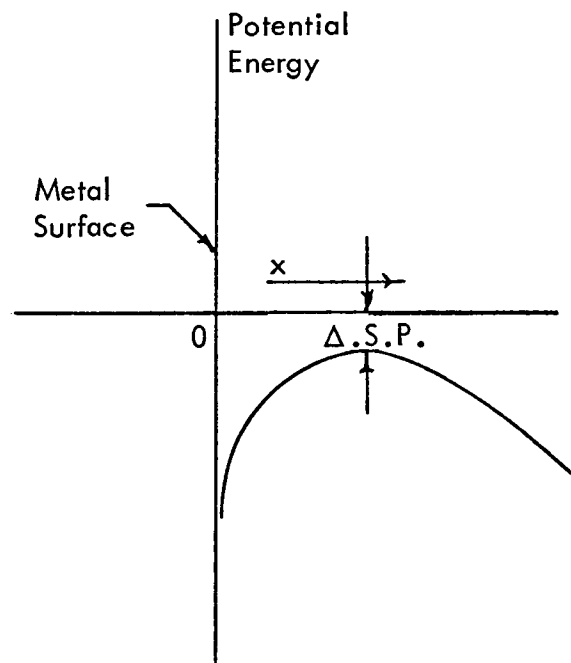
$$P.E._2 = -e \int \mathcal{E} \, dx \quad 14)$$

where \mathcal{E} is the value of the electric field in terms of x . Therefore, the total potential energy of the positive ion can be given as

$$P.E. \text{ total} = P.E._1 + P.E._2 = \frac{-e^2}{16\pi\epsilon_0x} - e \int \mathcal{E} \, dx \quad 15)$$



a) Without Field



b) With Field

Figure 9. Potential Energy of Ion versus Distance from Anode Surface.

In order to locate the point of maximum Potential Energy of the ion, we differentiate P with respect to x and set $\frac{dP}{dx} = 0$.

$$\frac{dP}{dx} = \frac{e^2}{16\pi\epsilon_0 x^2} - e\xi = 0 \quad 16)$$

where ξ for the case of a cylindrical diode is

$$\xi = \frac{\rho_L}{2\pi\epsilon_0(x+a)} \quad 17)$$

where ρ_L is the charge per unit length of the inner cylinder and a is the radius of the inner cylinder.

Equation 16 now becomes

$$\frac{dP}{dx} = \frac{e^2}{\pi\epsilon_0 x^2} - \frac{e\rho_L}{2\pi\epsilon_0(x+a)} = 0 \quad 18)$$

solving for x

$$\frac{e}{8x^2} = \frac{\rho_L}{x+a} \quad 19)$$

or

$$8x^2\rho_L - ex - ea = 0 \quad 20)$$

or

$$x_o = \frac{e \pm \sqrt{e^2 + 32\rho_L ea}}{16\rho_L} \quad 21)$$

since $e^2 \ll 32\rho_L ea$ and $e \ll \sqrt{32\rho_L ea}$

x_o can be approximated by

$$x_o \approx \frac{\sqrt{32\rho_L ea}}{16\rho_L} \quad 22)$$

It is now possible to find the value of Δ S.P. since this is the value of P.E. at $x = x_o$

$$e \Delta \text{ S.P.} = \frac{-e^2 16\rho_L}{16\pi\epsilon_o \sqrt{32\rho_L ea}} - e \int \mathcal{E} dx \quad 23)$$

For the configuration of two concentric cylinders with an inner radius of a , the last term of Equation 23 becomes

$$e \int_a^{a+x_o} \frac{\rho_L}{2\pi\epsilon_o x} dx = \frac{e\rho_L}{2\pi\epsilon_o} \ln \frac{\frac{\sqrt{32\rho_L ea}}{16\rho_L} + a}{a} \quad 24)$$

or

$$e \Delta S.P. = \frac{-e^2 \rho_L}{\pi \epsilon_0 \sqrt{32 \rho_L e a}} - \frac{e \rho_L}{2 \pi \epsilon_0} \ln \frac{\sqrt{32 \rho_L e a}}{16 \rho_L} + a \quad (25)$$

The last term in Equation 25 is negligible compared to the first in the operating range being considered, therefore

$$\Delta S.P. \approx - \frac{e \rho_L}{\pi \epsilon_0 \sqrt{32 \rho_L e a}} \quad (26)$$

Equation 26 is the change in surface potential of the anode due to the external field. The net effect of this change in surface potential will be an enhancement of ion emission. This enhancement can be taken into consideration in the expression for the degree of ionization by adding it to the expression of the work function.

Equation 10 then becomes

$$\alpha = \frac{g^+}{g^0} \exp \left[\frac{e \left(\Phi_{\text{metal}} - \frac{\sigma_1 \mu_1 \Theta_1}{\epsilon_0} + \frac{\sigma_2 \mu_2 \Theta_2}{\epsilon_0} + \frac{e \rho_L}{\pi \epsilon_0 \sqrt{32 \rho_L e a}} - 1 \right)}{KT} \right] \quad (27)$$

The magnitude of the change in work function due to the external electric field can be seen by evaluating Equation 26. The linear charge density for the diode

operating with a potential difference of 250 volts is

$$\rho_L = 1.32 \times 10^{-9} \frac{\text{coul}}{\text{cm}} \quad (28)$$

The inner radius a is

$$a = .318 \text{ cm} \quad (29)$$

Substituting these values into Equation 36

$$\Delta \text{ S.P.} = \frac{(1.6 \times 10^{-19} \text{ coul}) \cdot (1.23 \times 10^{-9} \frac{\text{coul}}{\text{cm}})}{\pi \left(8.85 \times 10^{-14} \frac{\text{coul}}{\text{volt cm}} \sqrt{32 \times 1.23 \times 10^{-9} \frac{\text{coul}}{\text{cm}} \times 1.6 \times 10^{-19} \text{ coul} \times .318 \text{ cm}} \right)} \quad (30)$$

or

$$\Delta \text{ S.P.} = .0194 \text{ volts}$$

Clearly, this change in work function is not as significant as those changes caused by adsorption.

Considering Equation 27, it must be pointed out that α , the degree of ionization, is the ratio of ions to atoms given off at the surface. The correlation of this term with current will require going back to Equation 4. Since β of Equation 4 is the ionization coefficient which is the ratio of ions emitted to the number of atoms arriving, the maximum value that β can have is one. In the case

of the diode at constant temperatures, the rate of diffusion of atoms to the surface will be constant, therefore any increase in current must be the result of an increase in N^+ .

$$\beta = \frac{N^+}{N} = \frac{\alpha}{1 + \alpha} \quad 31)$$

Since β is dependent upon α , as shown in Equation 31, very small values of α will cause β to be small and very large values of α will cause β to approach one. The effect of halogen vapors on ion emission can now clearly be shown. At the operating temperature of the diode and in the absence of the surface potential caused by halogen atoms, α will be a very small values since the exponential in Equation 7 will be negative. Therefore, the current reading will be small. However, when the surface potential effect of the halogen is introduced into Equation 27, α becomes very large causing β and the current to increase. At temperatures below the operating range of the diode, the atomic adsorption layers discussed in Paragraph 2 prohibit ion emission and at temperatures above the operating range of the diode, the chemisorbed surface potentials previously discussed are evaporated and β becomes essentially one. Considering the above observations, it can be said that the theory correlated with the operating characteristics of the diode. However, as mentioned previously, this theory is only a hypothesis since it has not been proven by experimental fact.

PARAGRAPH 4

CURRENT DISTRIBUTION STUDY

The purpose of the experimental work presented in this paragraph is to determine if a non-uniform current distribution exists along the axial length of the diode. Information on this current distribution would prove useful if it were desired to decrease the interelectrode volume of the present diode. Also, such information would add to the existing knowledge on the phenomenon that takes place when halogen bearing gases are ingested by the diode. An experimental diode was built to investigate this current distribution.

THE EXPERIMENTAL DIODE

The test diode utilized the standard General Electric anode as the positive ion emitter. In order to determine if one portion of the anode was more active in the ion emission process than other portions, the cathode was divided into five equal annular cylinders. These cylinders were isolated from each other by mica washers. The material used for the cathode cylinders proved to be very important. Stainless steel was tried but the surface was found to oxidize at the high cathode temperatures. The cylinders that were used in these measurements were stainless steel with platinum plated onto the surface. These platinum plated cylinders were found to be reasonably free from oxidation although there was some evidence of surface contamination after long periods of operation. The difficulties encountered

here indicate that platinum is necessary for reliable diode operation since it does not readily oxidize at high temperatures.

The anode and the segmented cathode were housed in a ceramic outer shell. Figures 10 and 11 show the test diode and its component parts. It must be noted that the physical size of the cathode cylinders proved to be somewhat of a problem. The cathode employed in the standard diode is a very thin annular platinum cylinder (.280 inch inside diameter) especially designed to minimize heat loss from the anode.⁹ The cathode cylinders of the test diode had the same inside diameter but an outside diameter of one half inch. The heat loss caused by this thick cathode required the heater voltage of the anode to be significantly increased over the usual value of 8 volts²⁰ in order to maintain the proper operating temperature. Aluminum plates were attached to both ends of the diode housing. These plates were machined to accommodate the tubing from the air pump.

The schematic diagram of the experimental diode is shown in Figure 12. Figure 13 contains a block diagram of test apparatus. The current through each cathode segment was measured with a Keithley Micro-Microammeter (Model 414). Since this type of meter exhibits a high input impedance in the ranges of interest, resistors were incorporated into the cathode circuits to insure that no potential difference existed between cathodes when measurements were being taken. For example, if the current through one cathode segment was being measured, the other cathode circuits would be connected to ground through resistors of the

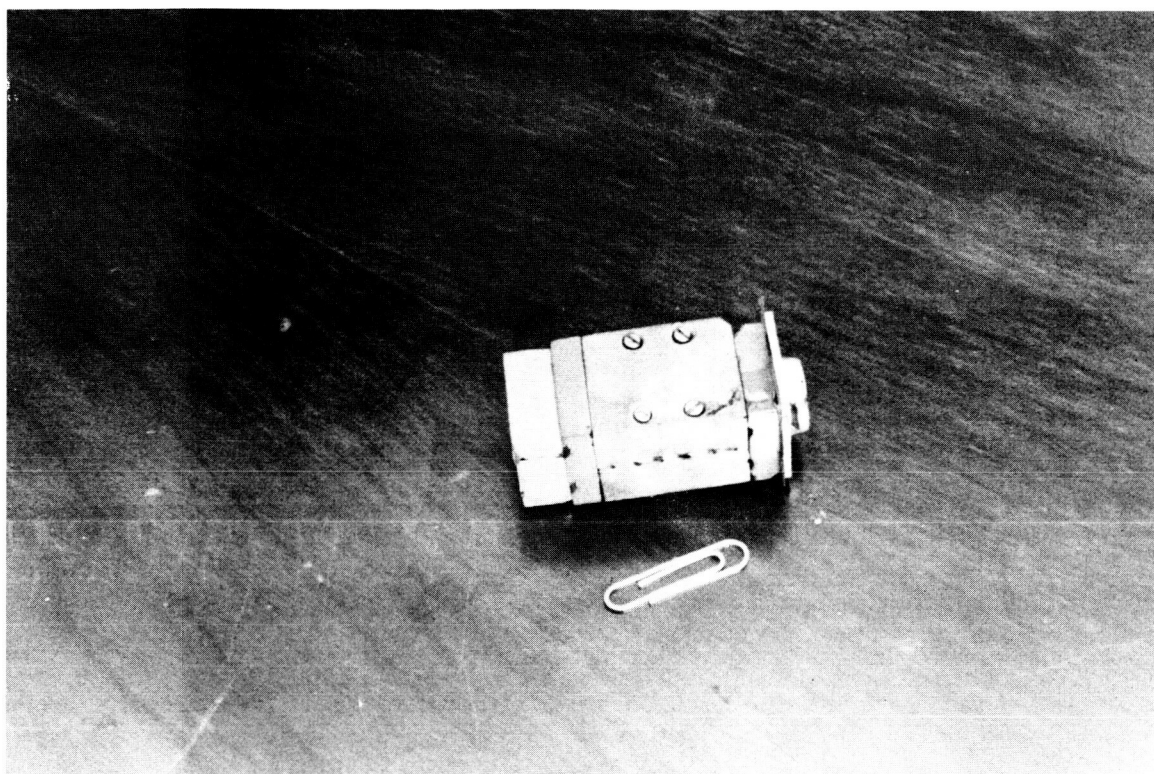


Figure 10. The Experimental Halogen Diode.

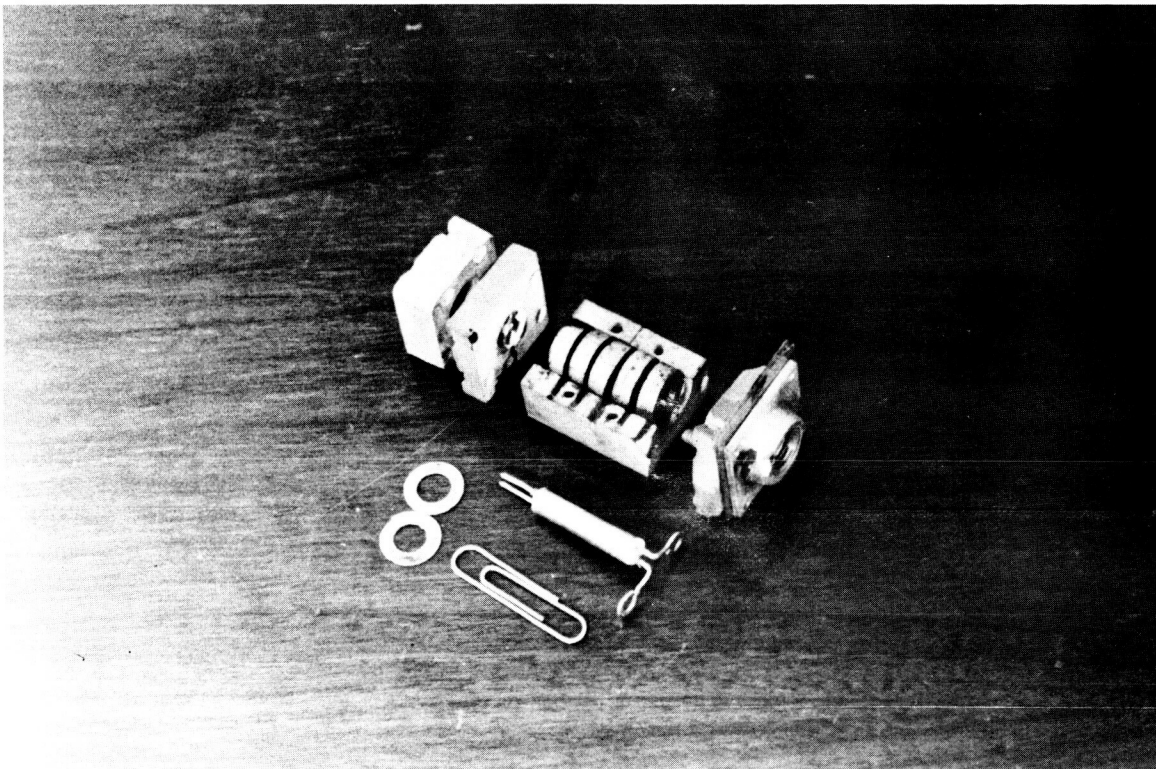


Figure 11. The Experimental Halogen Diode Disassembled

same value as the input impedance of the meter. The input impedance varied with different current scales of the meter which resulted in a number of different resistance values. A rotary switch was used in order to facilitate the switching of the meter from one cathode circuit to another. The heating element of the anode was connected to a variable ac (0 - 16 volts) power supply. The dc voltage was supplied to the anode by a 250 volt battery. Air samples were drawn through the diode by means of an air pump.

The experiment was performed in the following manner. The air pump and filament supply were turned on and the diode was allowed to warm up to rated operating temperature. Rated operating temperature is usually 800°C but the diode can operate at a temperature range of 700°C to 900°C.²¹ After sufficient warm-up time, the air flow was checked with a flow meter and set at 45 scim. Known Freon (CHClF₂)-air concentrations were ingested by the diode at its recommended flow rate and the current measurements were taken.

RESULTS

With air as the ingested sample, the magnitude of the interelectrode current was found to vary with each test run even though an effort was made to keep the diode at constant operating conditions. These variations could have been caused by the accumulation of a background concentration of Freon but the presence of a background concentration is doubtful since precautions were taken

to keep the test area well ventilated. A more probable cause would be a change in anode surface characteristics due to repeated exposures to Freon. Variations of this type are not peculiar to the test diode. W.C. White has also observed changes in halogen diode characteristics after repeated exposures to small concentrations of halogen bearing gases.¹⁹

The results of the current measurements are shown in Figure 14 and Table 4. In Figure 14, the curves were obtained by plotting the magnitude of the change in current when the diode ingested Freon (CHClF_2)-air concentrations versus the diode axial length. These currents are found to peak at approximately the middle of the diode. This non-uniform distribution could be attributed to the possibility that the middle of the anode is at a higher temperature than the ends. The higher temperature would result in the impurity atoms diffusing more rapidly through the platinum and, therefore, more atoms would be available for ionization at the surface.⁷ Also, the kinetic energy of the Freon molecules would increase with temperature. The net result would be an increase in the probability that the Freon molecules would come in contact with the surface of the anode. Another possibility for the non-uniform current distribution would be non-uniform cathode segments. However, the configuration of the segments was changed with no apparent effect on the current distribution.

The sensitivity of the diode is lower than that of the standard diode. For example, a concentration of ten parts per million of Freon₂₂ in air increased

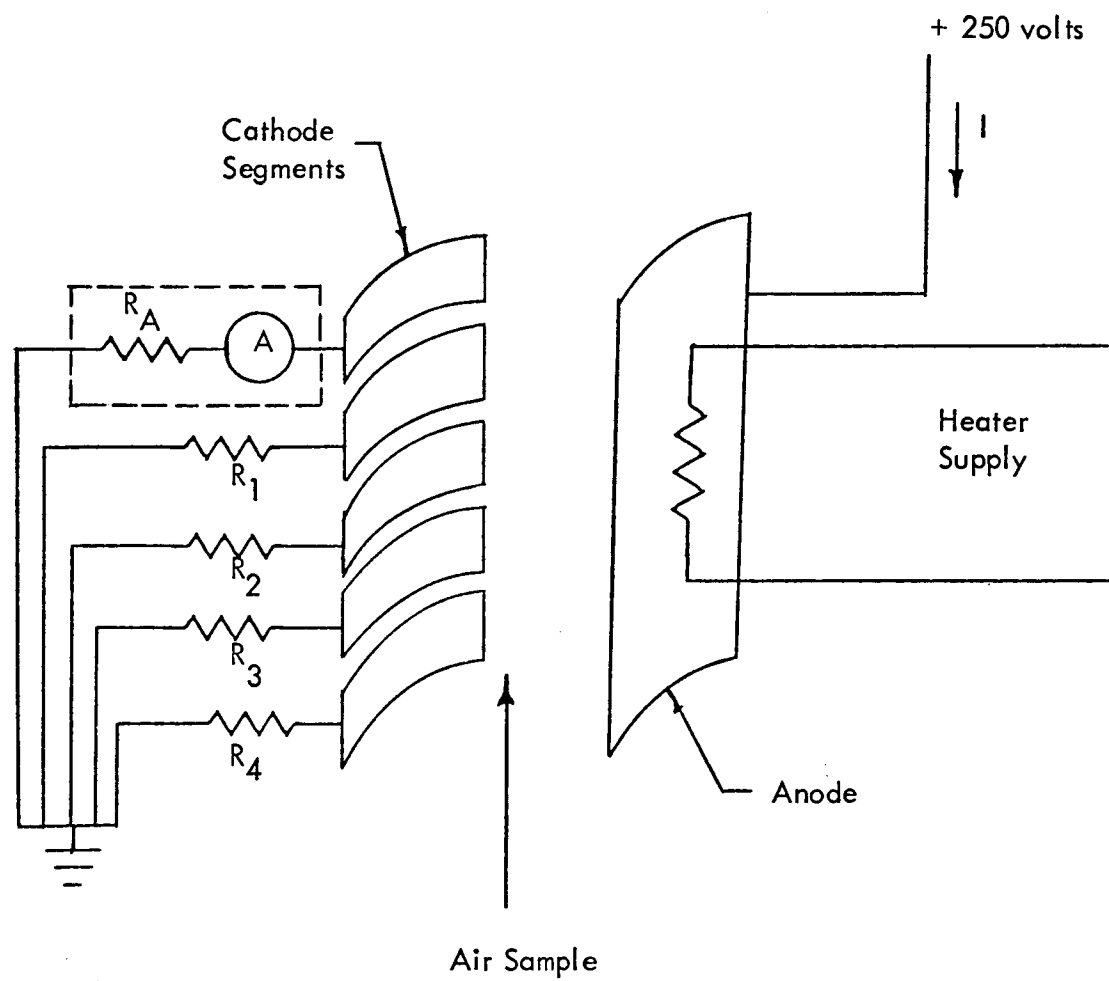


Figure 12. Schematic Diagram of Experimental Diode

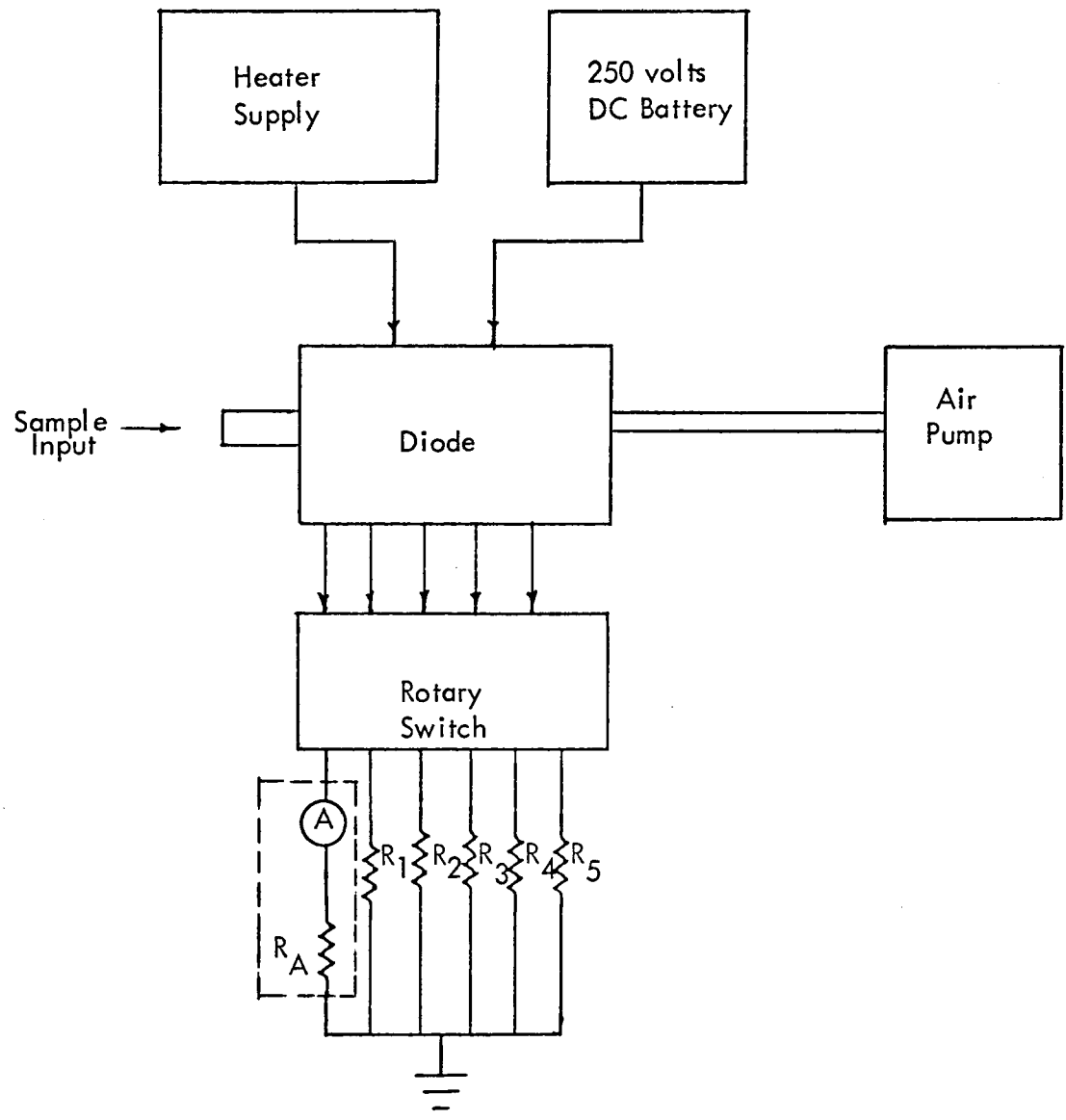
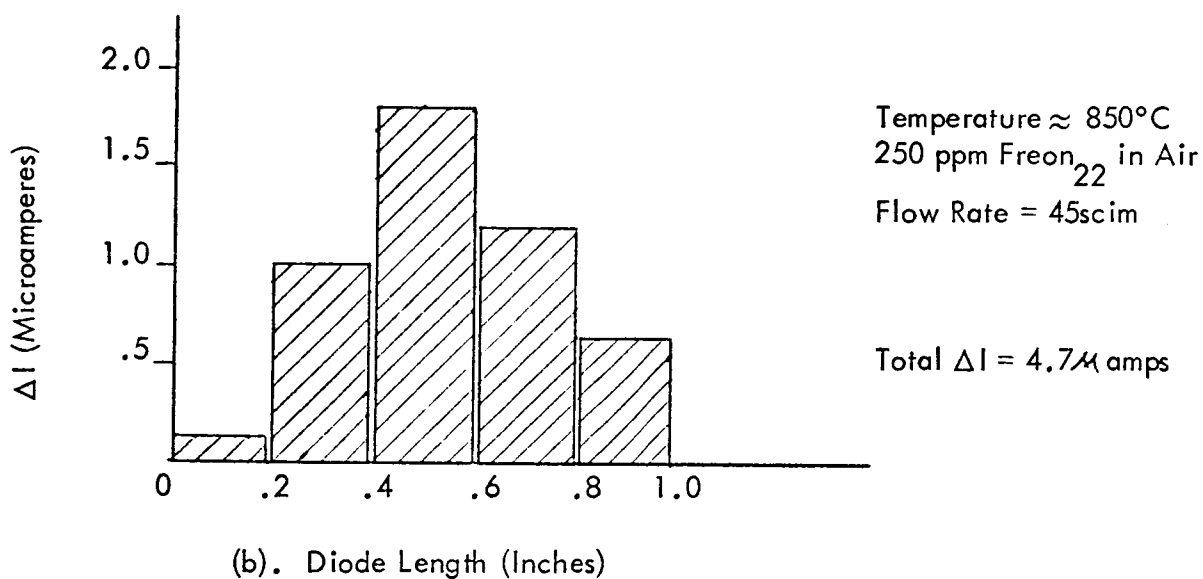
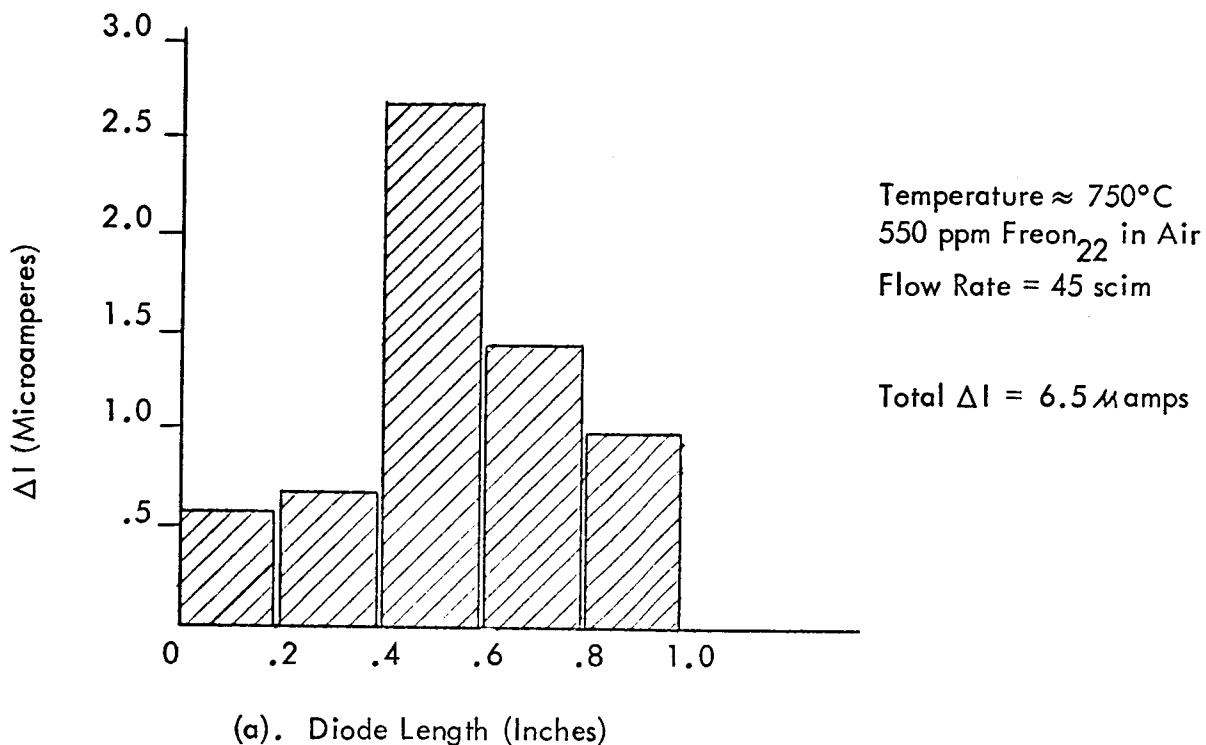


Figure 13. Test Apparatus.



NOTE: ΔI values were obtained by subtracting current when sample is air from current when sample is a Freon-air concentration.

Figure 14. ΔI versus Diode Axial Length.

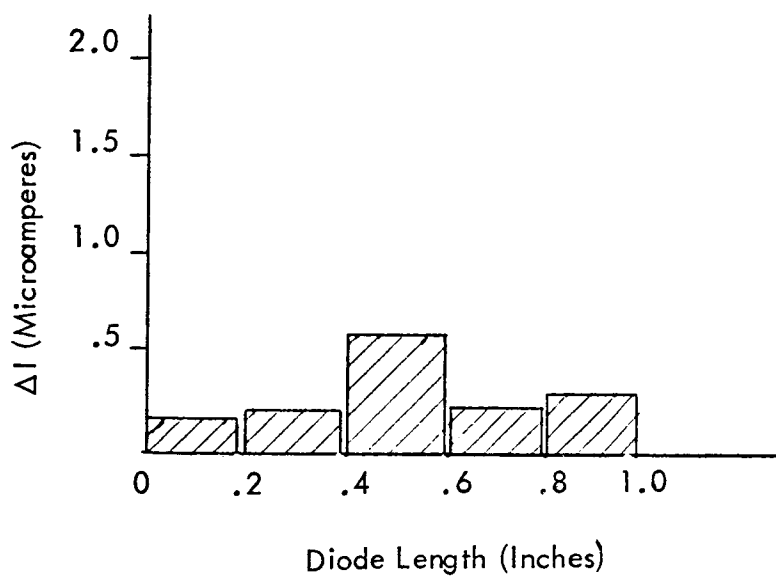
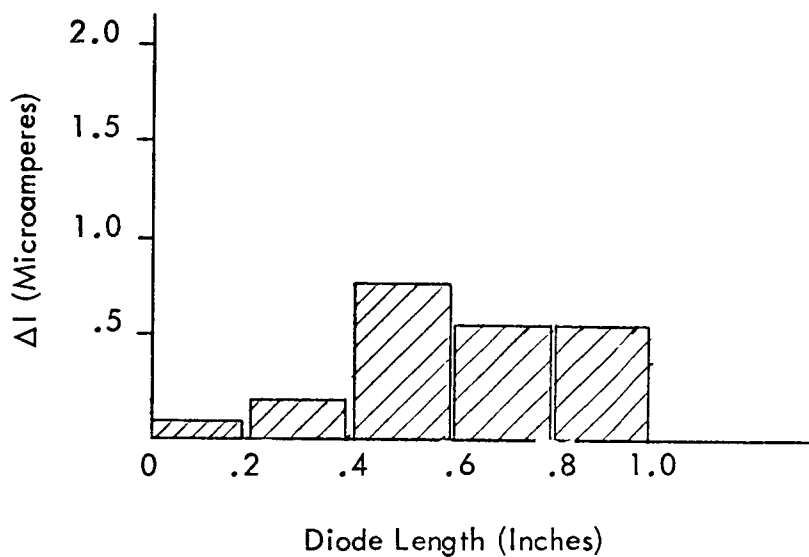


Figure 14 (Continued)

Anode Temperature \approx 850°C

Air Flow = 45 scim

Freon-Air Concentration = 410 parts per million Freon₂₂ in Air

Current in Microamperes

<u>Segments</u>	<u>Air</u>	<u>ΔI</u>	<u>410 ppm Freon₂₂ in Air</u>
1	10.3	4.2	14.5
2	10.4	5.1	15.5
3	9.0	6.0	15.0
4	8.7	3.5	12.2
5	6.4	4.6	11.0
all segments	12.6	6.9	19.5
1 and 2	10.5	4.0	14.5
1 and 3	9.0	3.0	12.0
1 and 4	8.5	2.5	11.0
1 and 5	8.0	2.5	10.5
2 and 3	9.5	6.5	16.0
2 and 4	6.0	5.5	11.5
2 and 5	7.0	4.0	11.0
3 and 4	9.5	3.5	13.0
4 and 5	8.5	4.5	13.0

Segment numbers indicate cathode segments that are being monitored.

The remainder of the cathode circuits are disconnected from ground.

Ionization Currents in Segmented Diode

TABLE 4

the current in the test diode by 1.4 microamperes while for the standard diode the current increase was 10 microamperes. This decrease in sensitivity can be expected since the cathode of the test diode was not made of the same material as the standard cathode. The test apparatus was checked by substituting the standard cathode in place of the segmented cathode. For a known Freon₁₂ leak rate of 3.5×10^{-4} scim, the change in current when the standard cathode was used was 19.5 microamperes while the change in current when the platinum plated cathode was used was 7.5 microamperes. This check indicates that the lack of sensitivity lies chiefly in the material used for the cathode. The presence of contamination of the platinum plated cylinders and the heat loss caused by the thickness of the test cathode are suspected as being the major causes of poor sensitivity. However, neglecting the fact that the sensitivity of the test diode was not up to expectations, the results indicated that there is a non-uniform current distribution along the axial length of the diode. The current measurements given in Table 4 were performed in order to determine the effect that a decrease in cathode length would have on diode sensitivity.

PARAGRAPH 5
SWITCHING RATE STUDY

EFFECT OF DECREASING VOLUME

The low switching rate of the gradient detector could be the result of the low flow rate of air (approximately 45 scim)²⁰ through the diode. If this flow rate restricts the switching rate, then an increase in switching rate could be obtained merely by increasing the flow rate. However, diode sensitivity to halogen bearing gases is known to decrease as the rate of air flow is increased.²⁰ Decreasing the interelectrode volume of the diode would have the same effect as increasing the flow rate. With a smaller diode volume, the volume of the ingested air samples could be decreased. The net result could be an increase in the limit imposed on the switching rate. The question that now must be answered is to what extent does the flow rate of air through the diode restrict the switching rate? A study of the input to the gradient detector should answer this question.

The air samples are ingested through two probes which are at slightly different locations. The diode alternately accepts samples from each probe by means of a gating valve that switches the air intake of the diode from one probe to the other. If the two probes intake identical concentrations of tracer gas, the diode will have the same dc current for each of the samples. However, if one probe intakes a stronger concentration of tracer gas than the other, the diode will

exhibit a change in current for each of the two samples. The frequency of this ac current is the same as the frequency at which the gating valve changes the input to the diode. One cycle occurs during the time it takes to pass air samples from each of the probes through the diode. If it is assumed that the flow rate determines the value of the switching frequency and that the diode is sensing an air sample from the left probe, the valve must maintain the left probe open and the right probe closed until the volume of the ingested sample is equal to the volume between the electrodes. When this sample has passed into the diode, the valve begins to allow the diode to accept a sample from the right probe. Using the recommended flow rate of air and the interelectrode volume of the diode, a value for the switching frequency can be calculated. Since the value for the flow rate is given in scim, the volume of the diode initially will be assumed to be at standard temperature (68°F) and pressure (1 atmosphere). The switching frequency can then be found by

$$1/2 K \frac{\text{Rate of Flow}}{\text{Volume}} = \text{Switching Frequency}$$

where volume = .025 in³

$$\text{Rate of Flow} = 45 \text{ scim}$$

$$K = 1/60 \text{ (Conversion from minutes to seconds)}$$

Then

$$\text{Switching Frequency} = 1/2 \frac{1 \text{ min}}{60 \text{ sec}} \frac{45 \text{ in}^3/\text{min}}{.025 \text{ in}^3} = \frac{15 \text{ cycles}}{\text{seconds}}$$

If the fact that the diode volume is not at standard conditions is taken into consideration, this theoretical limit of 15 cycles per second significantly increases. The difference between this theoretical limit and the experimental frequency of 5 cycles per second indicates that something other than the flow rate is restricting the switching frequency. The next step in this investigation will be to consider the response of the diode to halogen bearing gases.

DIODE RESPONSE

The diode will indicate the presence of halogen-bearing gases by an increase in the interelectrode current flow. If this current does not return to near ambient levels before the gating valve switches, the resolution of the gradient detector will be hampered. Figure 15 contains a diagram of the circuit employed to investigate the response time of the diode when it was momentarily exposed to Freon-air mixtures. The diode was mounted in the standard General Electric detector (Model H2) gun assembly. The current through the cathode circuit was monitored with a Varian Associates Model G14 graphic recorder connected in parallel to the cathode resistor. The heating element of the anode was connected to a variable ac (0-16 volts) power supply. Anode bias voltage was supplied by a dc power supply.

The experiment was performed in the following manner. With the diode at rated operating conditions and the air flow set at 45 scim, the diode input was momentarily exposed to a Freon-air mixture and the response time was plotted

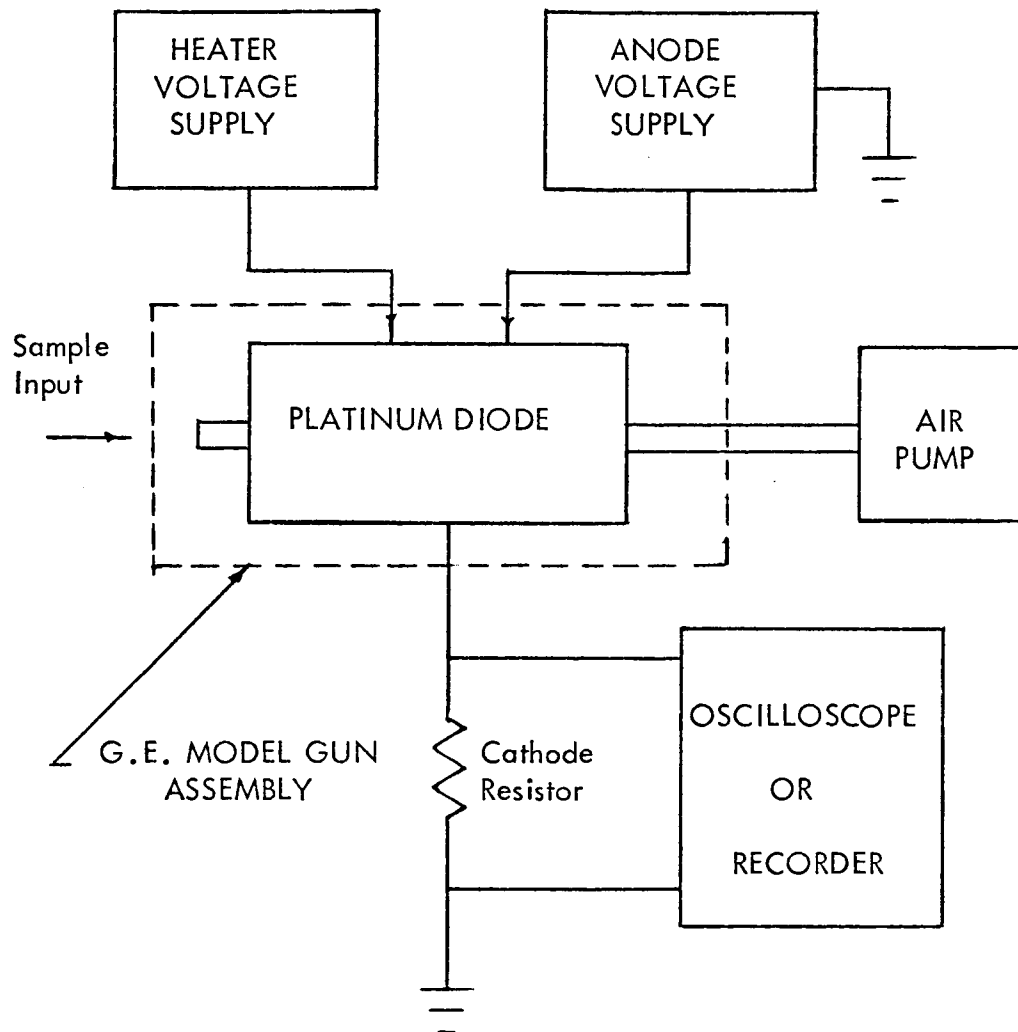
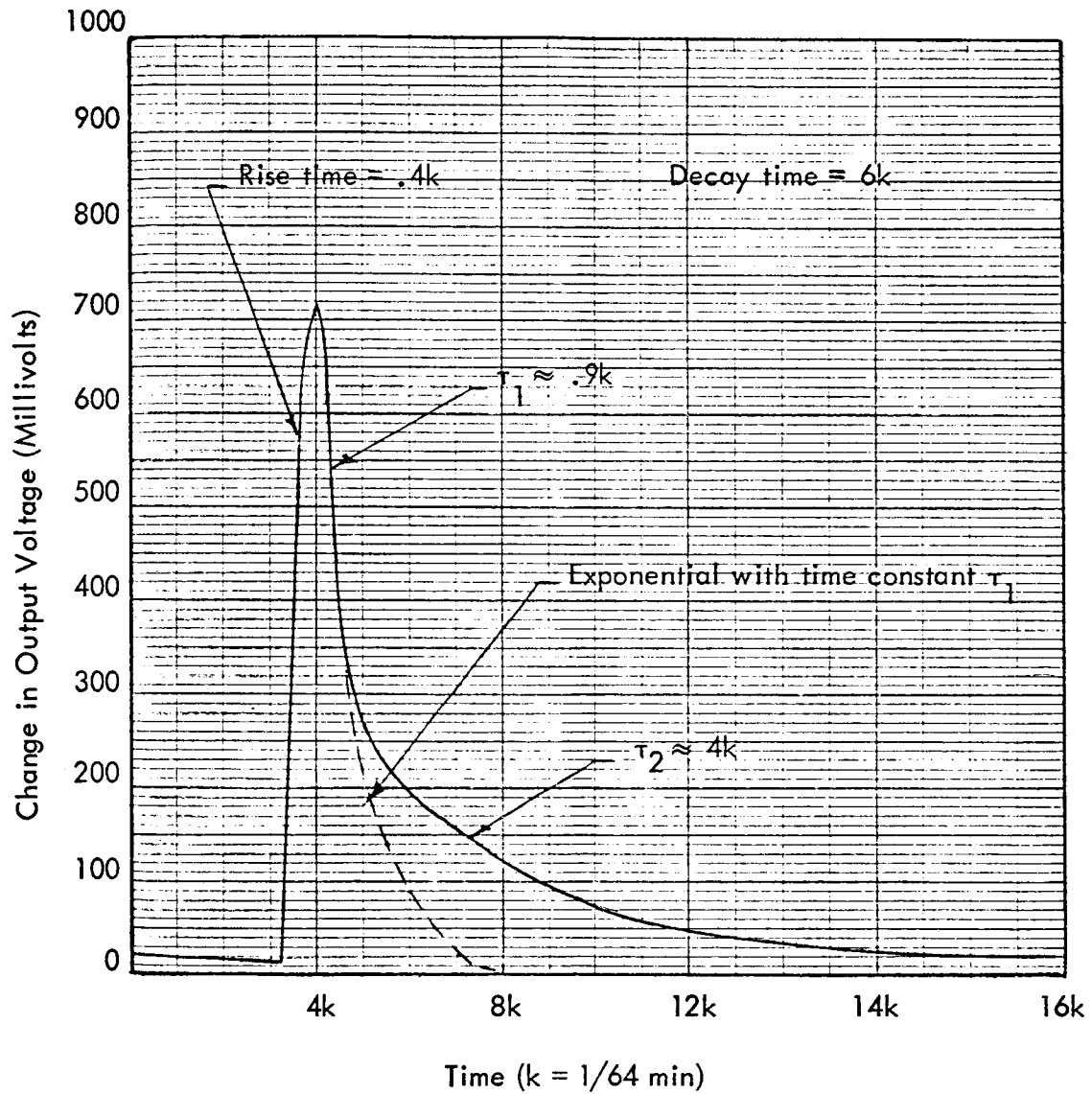


Figure 15. Diagram of Diode Response Test Apparatus.

by the graphic recorder. The nostril from the General Electric gun assembly was removed in order to shorten the exposure time of the diode to the tracer gas. Precautions were taken to keep the output of the Freon-air source well ventilated to insure that a high background level of tracer gas concentration did not exist at the source. Figure 16 (a) contains a typical plot of the change in voltage at the cathode resistor versus time. The rise time is defined as the time for the voltage to go from ten to ninety percent of its maximum while the decay time is defined as the time from ninety percent to ten percent of maximum voltage. The time constant τ_1 was obtained by assuming an exponential for the initial portion of the decay period. At the point where the exponential of time constant τ_1 differed from the experimental value, a new exponential of time constant τ_2 was assumed and found to fit the curve fairly well.

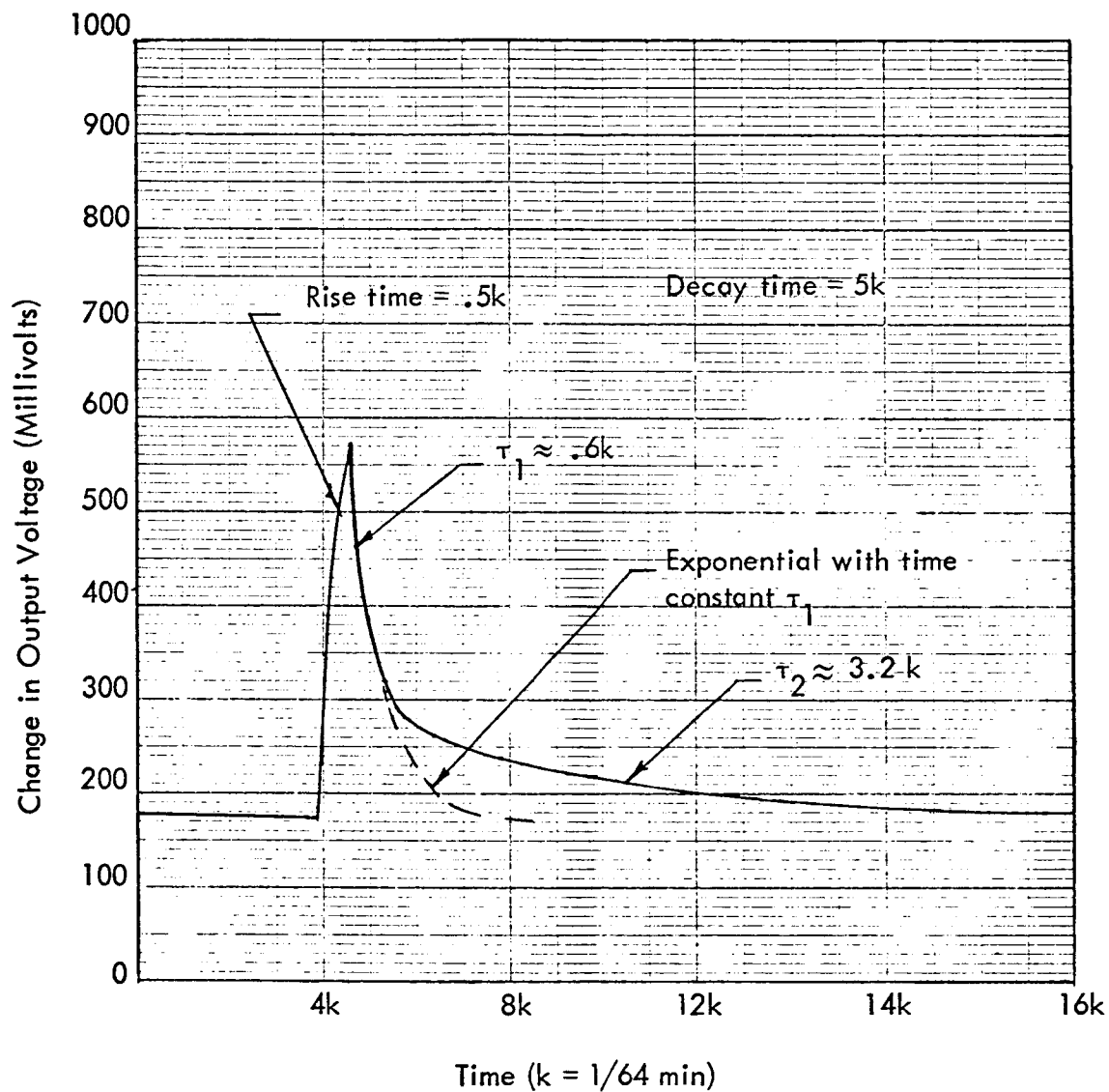
The results obtained here indicate the response time of the diode is the cause of the slow switching rate of the detector. The long decay time (3.8 sec) is probably due to the coating effect of halogen atoms at the surface of the anode. Flow rate calculations indicate that the sample input to the diode should return to ordinary air in a fraction of this total decay time. Halogen atoms or ions (formation of negative halogen ions has been shown to be possible in Paragraph 2) reside at the anode until they gain enough energy to be neutralized and evaporated off. If there is a formation of negative halogen ions, the decay time should be improved by changing to an ac anode voltage. Figures 16 (b) and 16 (c) are plots



72 ppm Freon₂₂ in Air

Anode to Ground Voltage = 200 V dc

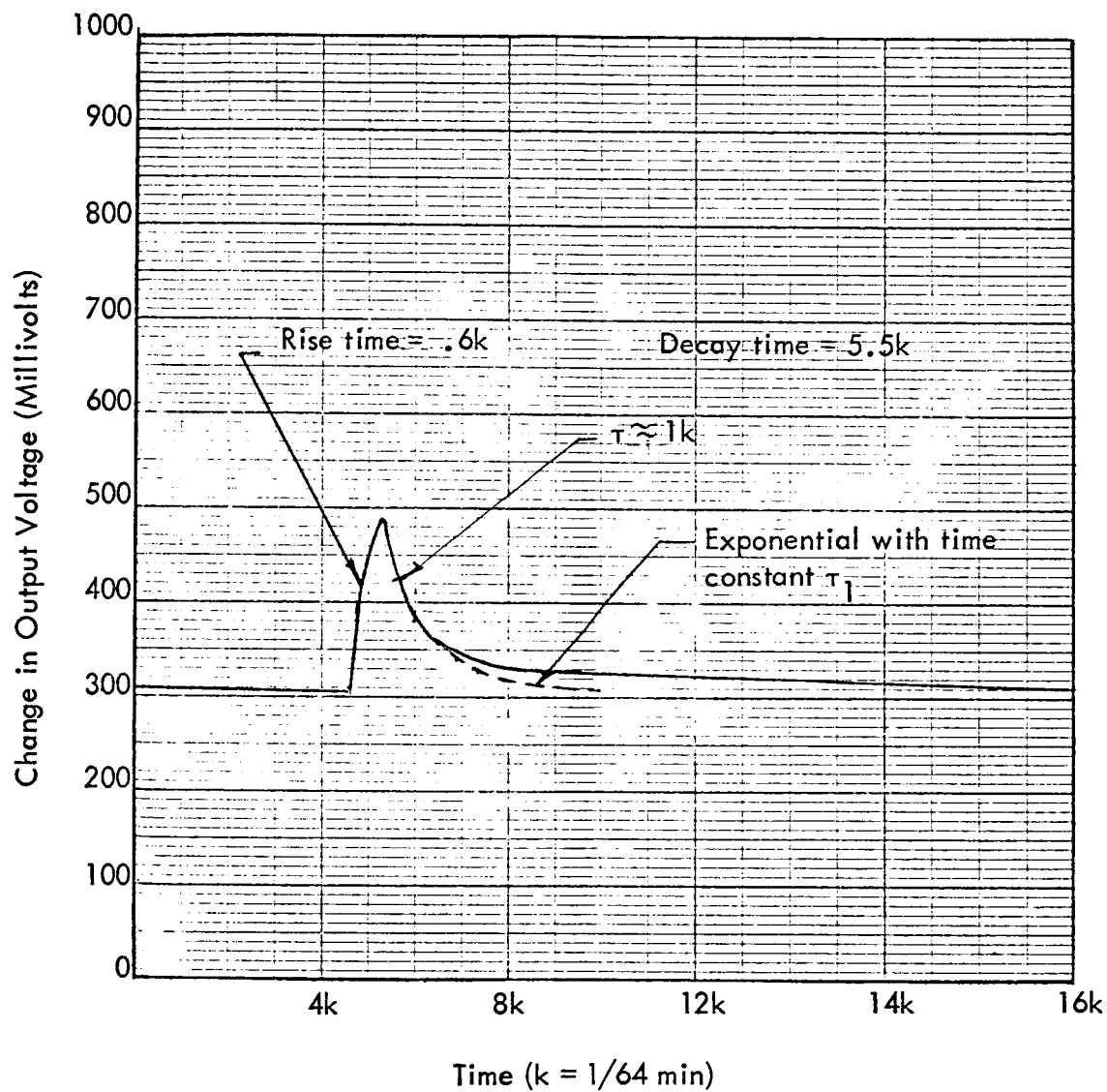
Figure 16 (a). Response of Diode Due to Momentary Exposure to Freon-air Mixture.



72 ppm Freon₂₂ in Air

Anode to Ground Voltage = 250 ac (60 cps) rms

Figure 16 (b). Response of Diode Due to Momentary Exposure to Freon-air Mixture.



72 ppm Freon₂₂ in Air

Anode to Ground Voltage
= 250 V ac (60cps) rms -
150 V dc Bias

Figure 16 (c). Response of Diode Due to Momentary Exposure to Freon-air Mixture.

of the change in voltage versus time for an ac anode voltage and an ac anode voltage with a negative dc bias. These measurements were obtained at the same operating conditions used to obtain the curve in Figure 16 (a). Comparing Figure 16 (a) and Figure 16 (b), it is seen that the decay time constants are slightly smaller with the ac anode voltage than with the dc anode voltage. This improvement in decay time warrants further study. Future experiments should be performed employing ac anode voltages of varying waveforms and frequencies in order to determine if this decay time can be minimized.

PARAGRAPH VI

This study of the ion emission properties of the halogen diode has been motivated by the need for a shorter response time for the time-sharing halogen gradient detector. The positive ion emission at the anode has been explained. These ions are not ions of halogen molecules but are ions of alkali metal impurities that are doped onto the ceramic core of the anode. The alkali metal ions reach the platinum surface by diffusion and are then ionized. Freon molecules dissociate upon contact with the hot anode and cause the alkali atoms to ionize more readily. This increase in ionization has been attributed to a chemisorbed coating which results when halogen atoms are passed over a hot platinum surface.

A suitable equation of emission has been developed which approximates the behavior of the diode. This equation was obtained by making appropriate modifications to the Langmuir equation. The electric field has been found to have little effect on the surface ionization properties of the anode.

The results of the experimental investigation show that a non-uniform current distribution exists when Freon-air mixtures are ingested by the diode. The sensitivity of the experimental diode was less than that of the standard diode but this difference can be attributed to the size and material of the segmented cathode.

The switching rate study has shown that the response time of the time-sharing detector cannot be significantly improved by an increase in the flow rate of air through the diode or a decrease in the diode volume. The restricting factor which limits the switching frequency of the detector has been found to be the slow decay time of the diode current once halogen bearing compounds have passed between the electrodes. The application of an ac voltage to the anode resulted in a slight improvement in this decay time. This slight improvement lends support to the possibility that negative halogen ions may be formed at the anode surface. Future study should be directed to determining the effects that different waveforms and frequencies of alternating voltage have on the diode response to halogen bearing compounds.

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CHAPTER III

ACOUSTICAL LEAK DETECTION BY SONIC INJECTION

Earlier experimental studies¹ performed by the Ohio University research group showed that leaks on both high and low pressure systems have a passive spectral distribution of acoustical energy which peaks at a frequency between 35KCPS and 45KCPS. If an efficient detector is to be realized, it should be designed so that it will respond to this energy peak which is at approximately 40KCPS. The Delcon Ultrasonic Translator is designed in this manner; however, it is plagued by environmental disturbances. Namely, it cannot distinguish consistently between acoustical energy generated by the leak and energy generated by discontinuities in the plumbing system. Also, reflections of acoustical energy from objects which surround the leak frequently cause the Delcon system to give erroneous indications.

In an effort to eliminate these difficulties, an attempt was made to modulate the leak rate by injecting active sinusoidal or pulsed pressure changes. The ultrasonic noise generated by the leak hopefully would then be amplitude modulated and could be sensed and demodulated by a suitable transducer. This type of sound injection should easily lend itself to a method of phase correlation detection somewhat similar to that developed by the American Gas Association.² The net effect of this phase correlation technique should be to eliminate the difficulties encountered by the Delcon Ultrasonic Detector.

Initial experiments consisted of employing two low frequency motor-driven transducers to inject variable frequency pressure changes into the plumbing system.

One transducer consisted of a variable speed diaphragm pump, while the other was a fixed speed piston-type pump. The plumbing system was a 30 ft. length of 3/8 inch copper tubing which connected the leak to the transducer. The leak consisted of a circular orifice which was located in a soundproof chamber. Detection equipment included:

Microphone ... Bruel and Kjaer, Type 2613, Serial Number 89798.

AmplifierBruel and Kjaer, Type 2604, Serial Number 82452.

A comparison of the leak noise generated by both transducers was made with the system at atmospheric pressure. The piston-type transducer yielded an output noise level of 96 db* while the diaphragm type transducer's output level was 84 db. Both transducers were run at 35CPS with a background noise level of 54 db, and a 2-mil diameter leak orifice. Figure 17 shows relative sound levels obtained with varying orifice sizes.

Measurements were also made with the leak removed from the soundproof chamber. In order to reduce background noise and increase directivity, the microphone was mounted at the focal point of a 14 inch parabolic reflector. Figure 18 gives relative sound level readings at varying distances from the leak. Directivity was found to be fairly high. A five degree variation in a direction away from the leak at distances greater than 2 feet yielded sound level readings which were essentially background noise.

*db readings are referenced to 2×10^{-4} microbars

Figure 19 illustrates data obtained using a Bruel and Kjaer, Type 2202 sound level meter with Type 1613 octave filter set. The leak consisted of a 2-mil orifice located outside the soundproof chamber with the system operating at atmospheric pressure. The top curve is a combination of leak and background noise while the bottom curve is the background noise found at different frequencies.

Results of the measurements taken with this relatively restricted plumbing system showed that leak noise generated by variations of pressure within an open system can be detected with some degree of accuracy. Also, the utilization of a parabolic reflector was found to reduce random background noise and increase the directivity of the microphone. This increase in directivity was due to the higher frequency noise components generated at the leak. Directivity of the parabolic reflector microphone combination was lost when a low pass filter was incorporated into the amplifier unit.

Tests similar to those performed on the small system were employed on the compressed air plumbing system of the Engineering Building (four stories). This plumbing system is similar in some respects to that found in missiles. Figure 20 shows relative sound levels obtained with various orifice sizes and with the plumbing system operating at atmospheric pressure.

It should be noted that the sound levels contained in Figure 20 are relatively low. These low energy levels are due to the lack of sufficient output power developed by the available transducers. Most important of all, the modulating effort of the transducers was almost completely ineffective when a pressure of a few psig was

injected into the system. These results clearly indicate that transducers of prohibitive size would be needed to develop sufficient pressure variations in a large pressurized system for suitable detection using presently available hardware.

Carrying the investigation further, the decision was made to build a small-scale pressurized plumbing system which approximated the much larger and more complete missile systems. The system consisted of a small piston type transducer connected to a flange-type leak by means of a 20 ft. length of 3/8 inch copper tubing. The system size relative to the transducer was of a suitable ratio to permit the injected pressure changes to amplitude modulate the ultrasonic noise produced by the leak. For example, with the system at a bias pressure of 15 psig, the transducer injected pressure variations of 5 psig. The leak consisted of a loosened 1/4 inch stainless steel pipe fitting. The leak rate was 25.8 scims. Detection of the noise generated at the leak was accomplished through the use of a microphone-amplifier unit tuned to respond to acoustical signals in the neighborhood of 40CPS. The circuit diagram of this microphone amplifier unit is given in Figure 22. A block diagram of the entire test setup is given in Figure 21.

With the transducer operating at 40CPS, the detector failed to distinguish the modulation of the noise generated by the leak from the 60CPS noise present in the testing area. In other words, the signal-to-noise ratio was too low to permit reliable detection of the modulating signal. The oscilloscope was connected to the output of the tuned amplifier and some modulation of the leak noise was noticeable, but it was of such a small amount that changing the bias pressure of the system to 20 psig and over completely eliminated modulation detection.

Experimental data emphasizes the manifest differences between underground gas line leaks and leaks in a complex pressurized missile plumbing system. The gas pipe has a covering of earth which is a good conductor of sound waves and which contributes to the vibrational stability of the system. Typical missile leaks involve much more complex plumbing geometry, smaller critical leak sizes, and higher pressures.

The three principle mechanisms responsible for leak noise, namely, vortices formed at the sharp inner edge of the orifice and expelled into the air at the outer edge, turbulence caused by the high velocity flow in the orifice itself, and turbulence caused in the surrounding air by the jet moving out into the stable air, apparently react when the system pressure exceeds 15 to 20 psig in such a way that pressure modulation energy does not contribute significantly to total leak noise. It should be emphasized, however, that developments on the transistor microphone and other similar semiconductor transducers could considerably brighten the picture due to the much higher sensitivity and lower source noise which they inherently supply. A breakthrough in this area could well make combined active and passive acoustical systems very effective leak detectors.

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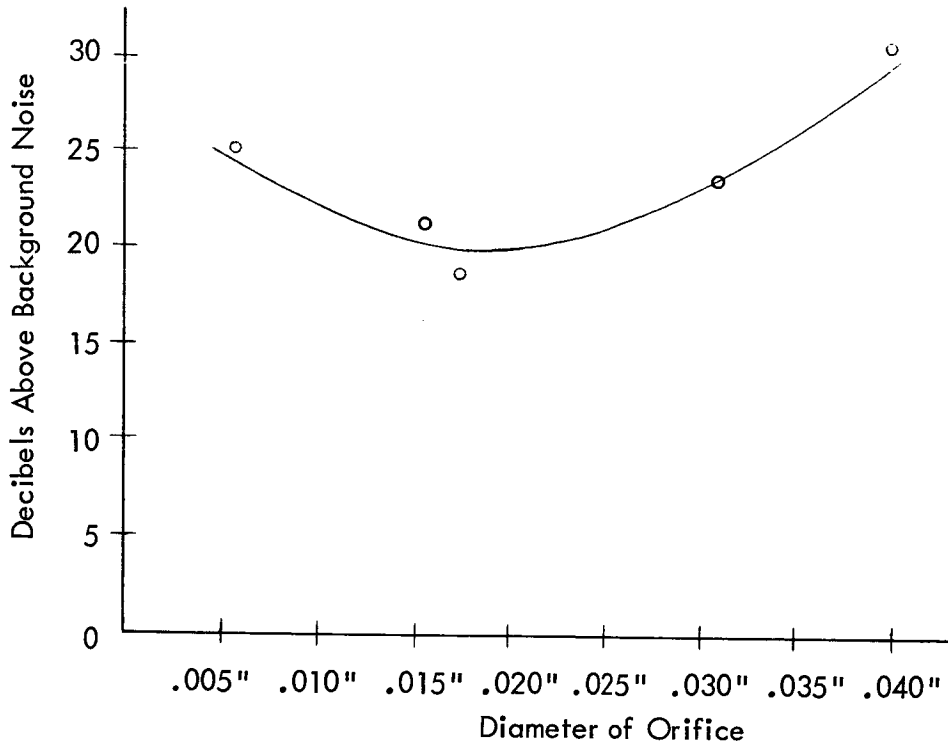


Figure 17
 Injected Pressure Change With Diaphragm
 Transducer At 40 CPS. Microphone Was 8" From Orifice

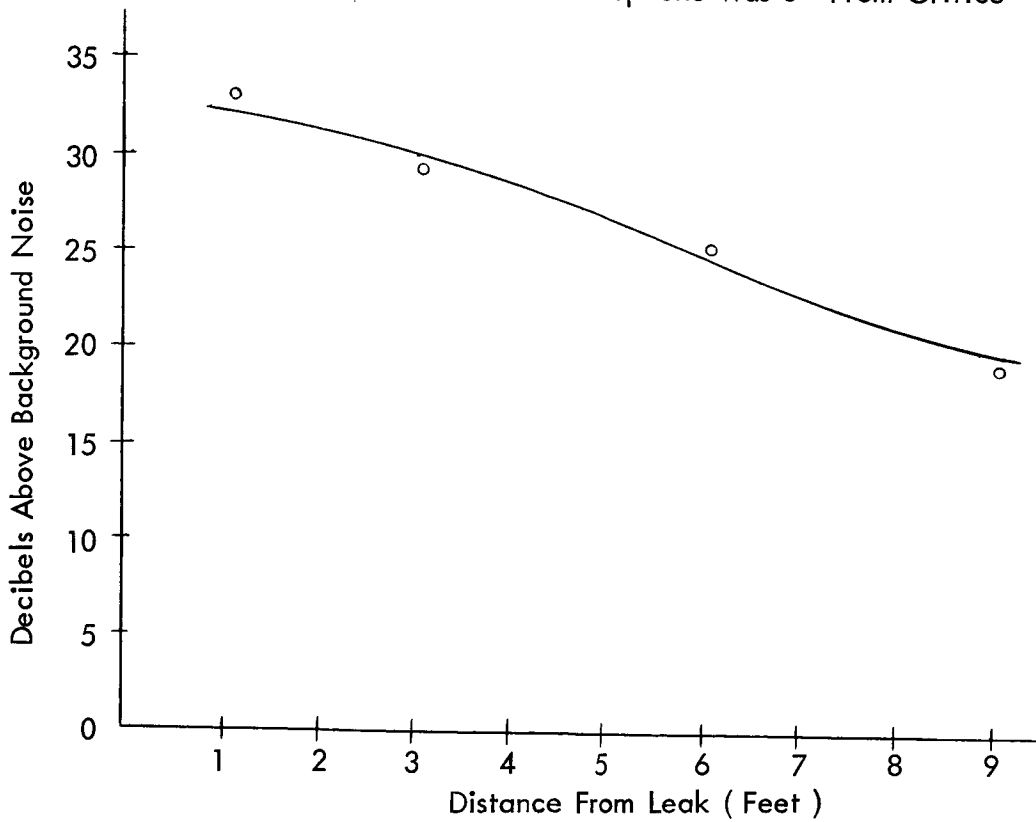
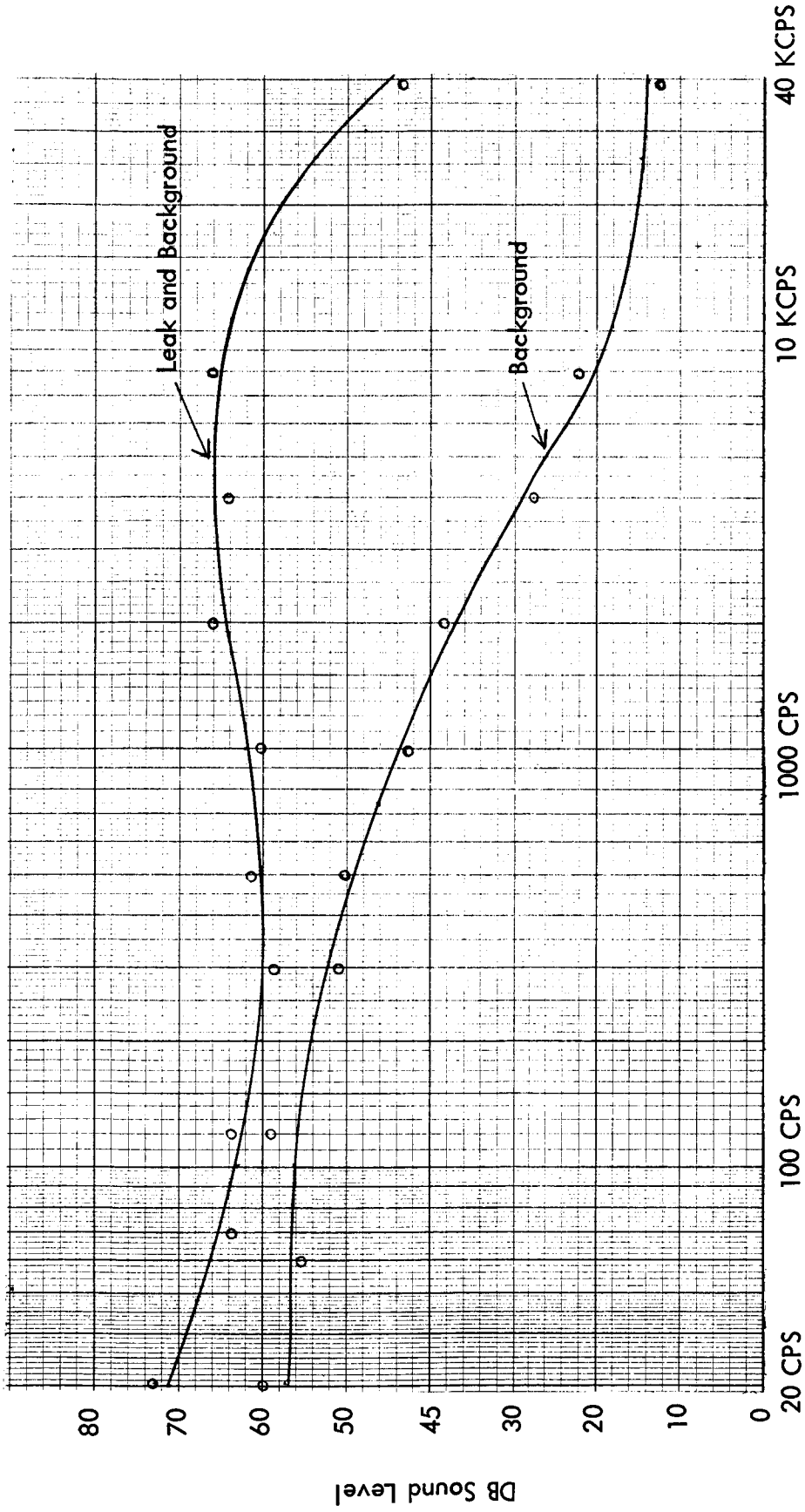


Figure 18

Distances From Leak (Feet) .002 Inch Orifice
 Piston Transducer Operating at 35 CPS



Frequency

Microphone Located 6 Inches From Leak

Pressure Variations Were At 35 CPS

Figure 19

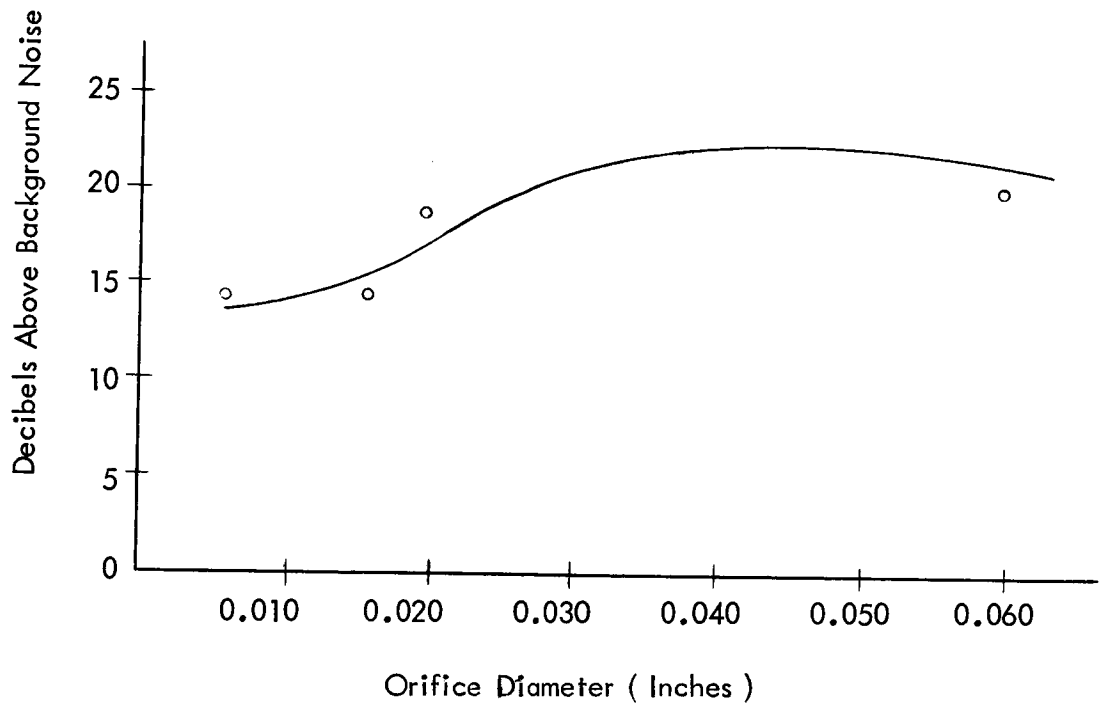


Figure 20

Microphone Located 1 Inch From Leak Piston
Pump Transducer Operating at 40 CPS

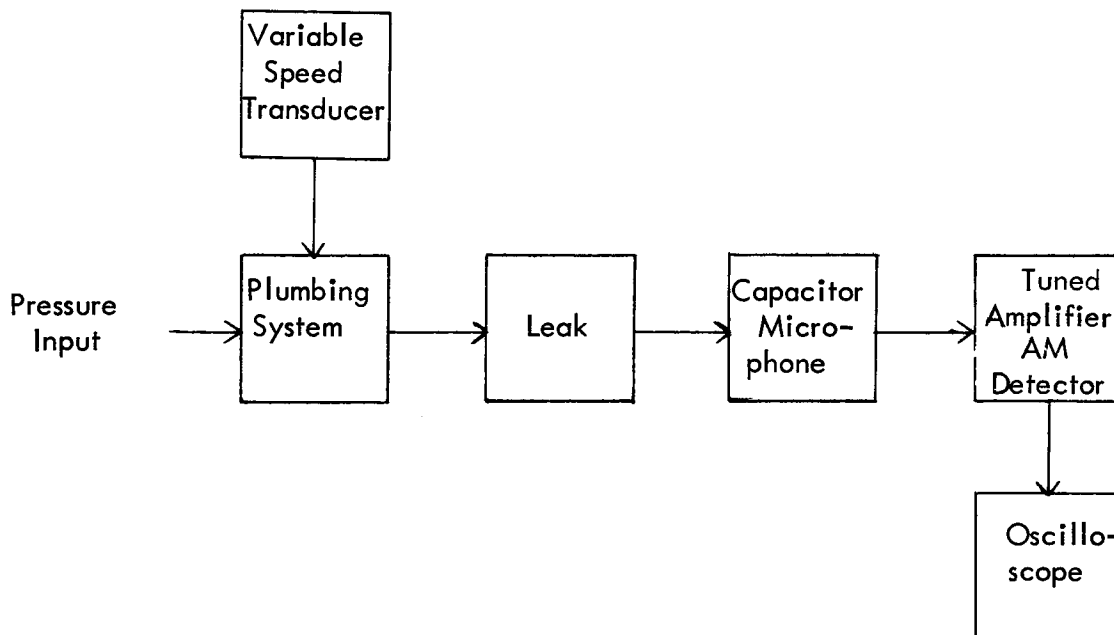
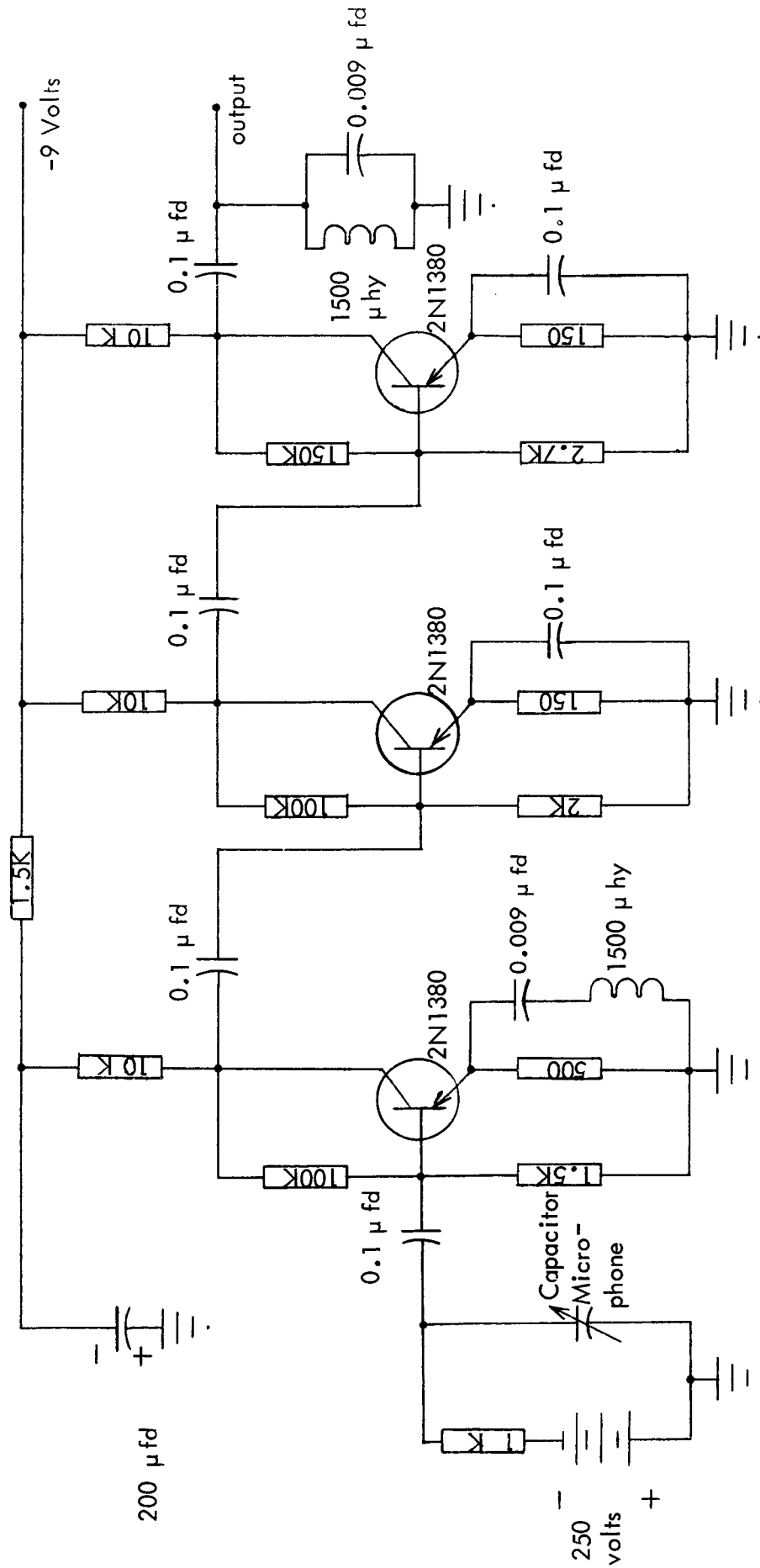


Figure 21

Test Apparatus



Microphone - Amplifier Unit Tuned To Respond To 40 KCPS Signals

Figure 22

CHAPTER IV

LEAK DETECTOR TESTING FACILITY

In pursuing state-of-the-art investigations, the Ohio University research group has made evaluations in as realistic a context as possible. It is very difficult to obtain highly quantitative data since all the variables are primarily statistical in nature, and reliable measurements can be obtained only by repeated use and statistical analyses of sufficient samples of data. This would seem to lead to the conclusion that the best testing laboratory is the missile test floor itself. It does not, however, preclude the usefulness, desirability, or even the necessity of having an auxiliary laboratory in which conditions may be rigorously controlled. It is with this in mind that work has been completed in setting up such a laboratory here at Ohio University.

In addition to the samples of missile plumbing with typical leaks and the calibrated orifice leaks already in use, this laboratory contains a means of supplying freon-air and other gas-air mixtures of precisely controlled and known composition. Available calibrated leak devices, such as the G. E. test leak, supply leaks of known rate but give very little control over background or tracer concentration gradients. In order to obtain reproducible data for determining meaningful parameters of sample-ingesting leak detectors, it is necessary to be able to supply the instrument being tested with reasonably large volumes of known tracer-air mixtures at atmospheric pressure. There must be no interference with the free flow of mixture into the instrument probe and no accidental admixture with ambient air, contaminated with back-

ground or otherwise. For testing of gradient -sensitive instruments there must be two such sources of mixture so related that the difference in concentration is precisely known. It may also be desirable to have a third source for making tests on some instruments to evaluate the effects of ducting instrument exhausts in producing feedback responses.

In the early stages of study of new transducers such as the hot ceramic diode, use was made of a vacuum plate and bell jar. This chamber was adequate for testing the transducer response to different tracer concentrations; however, it was impossible to adjust rates of flow across an electrode surface, or to measure concentration gradient sensitivity, since this involves the difference between sensitivities to almost identical concentrations.

In use, the bell jar works on the very simple and accurate principle of partial pressures. An example will illustrate its operation. It is desired to test a transducer in a mixture of one part freon to a million parts of air. The pressure in the chamber is first reduced to, say, 0.9 atmospheres. Pure freon is then added to bring the pressure back to 1.0 atmospheres. The mixture is now 10% (by volume) freon. If the pressure is now reduced to 0.1 atmospheres and air added to bring it back to 1.0 atmosphere, the concentration will be reduced to 1.0% freon by volume. This process may then be repeated until the desired concentration is reached - in this case four more times. Care must be exercised, of course, to see that thorough mixture of the freon and air is achieved. Even though the freon is considerably heavier than air, there will be no tendency for settling out once complete admixture has been

achieved*. Other final compositions may be had by applying the following formula:

$$\% \text{ Tracer} = 100 (1 - p_1) \prod_{i=2}^n p_i$$

where p_1 is the pressure to which the system is reduced before addition of the freon and the p_i are the pressures to which the mixture is reduced before adding the air. The symbol π indicates that the product of the pressures indicated is to be taken. All pressures are to be expressed in per unit of ambient atmosphere. Obviously it is necessary to be very careful about thorough mixing and to permit the temperature to stabilize before measuring pressure. If it is desired, for any reason, to set up a mixture of a certain percent by weight rather than by volume, the above formula is multiplied by the ratio of molecular weights of tracer and air.

Certain unavoidable errors in the measurement of pressure and temperature make it impossible to use the repeated dilution method for preparing samples of mixture differing by small increments of tracer concentration for use in testing gradient sensitive leak detectors. For this purpose it is necessary to prepare a single sample of the desired background level of concentration, divide it into two samples and then add freon to one or air to the other sample to produce the desired precise difference. The apparatus constructed for doing this is shown in Figure 23.

In this apparatus, freon is measured in the water displacement device A, injected into mixing tank M, where it is diluted with air. A small quantity of this mixture is injected into tanks L and R and again diluted to give exactly equal concen-

* Refer to Second Formal Report, page 260.

tration in L and R. A measured amount of air is then admitted to tank R from apparatus B to give a known small difference in freon concentration between L and R. The samples from L and R then flow to sampling hoods S where they are kept at a slight positive pressure as indicated by the inclined manometer M to prevent atmospheric dilution. Detector probes in the sampling hoods are thus exposed to a precisely known level and difference of freon concentration.

The operation and methods of calculation are illustrated by the following example:

- 1) Flush the entire apparatus with compressed air to remove any traces of freon from previous tests.
- 2) Take in 10 cc of freon to the graduate apparatus A at 1 atmosphere.
- 3) Open V1 and inject the 10 cc of freon into tank M, which is isolated by valve V2.
- 4) Shut V3 and pressurize M to 4 atmospheres by the air inlet at P1. This insures that the entire 10 cc of freon is now in tank M, and none remains in the tubing.
- 5) The parts per million of freon in tank M are now,

$$\text{ppm} = \frac{(P \cdot V)_F}{(P \cdot V)_M} = \frac{1 \times 10}{4 \times 14,400} \times 10^6$$

$$\therefore (\text{ppm})_M = 173 \quad (\text{The volume of M, L, R is 14,400 cc.})$$

- 6) Now open V4 and V5 and charge both L and R from 1 atmosphere to 1.5 atmosphere, from M via V2.

- 7) Shut V2, and pressurize L and R to 4 atmospheres with P2. The concentration in L and R is now

$$\text{ppm}_{L, R} = \text{ppm}_m \cdot \frac{\Delta P_{\text{from M}}}{P_{L, R}} = 173 \cdot \frac{0.5}{4} = 21.5 \text{ ppm}$$

- 8) To obtain a slightly lower ppm in R, shut valves V4 and V5 and admit a measured amount of air from B into R through V6. In this case, with the pressure of B kept at a steady 5 atmospheres by P4, a one inch rise of water is observed as air is bled into R from B. This represents 207 cc of air, from the cross section of tank B.

The change in ppm of R is now computed as:

$$\Delta \text{ ppm} = \text{ppm}_L - \text{ppm}_R = 10^6 \left[\left(\frac{\text{moles freon}}{\text{moles air}} \right)_L - \left(\frac{\text{moles freon}}{\text{moles air}} \right)_R \right]$$

$$\Delta \text{ ppm} = \frac{\text{moles freon}}{\text{moles air}_L} \cdot 10^6 \cdot \left[1 - \frac{1}{1 + \frac{\text{air from B}}{\text{air from L}}} \right]$$

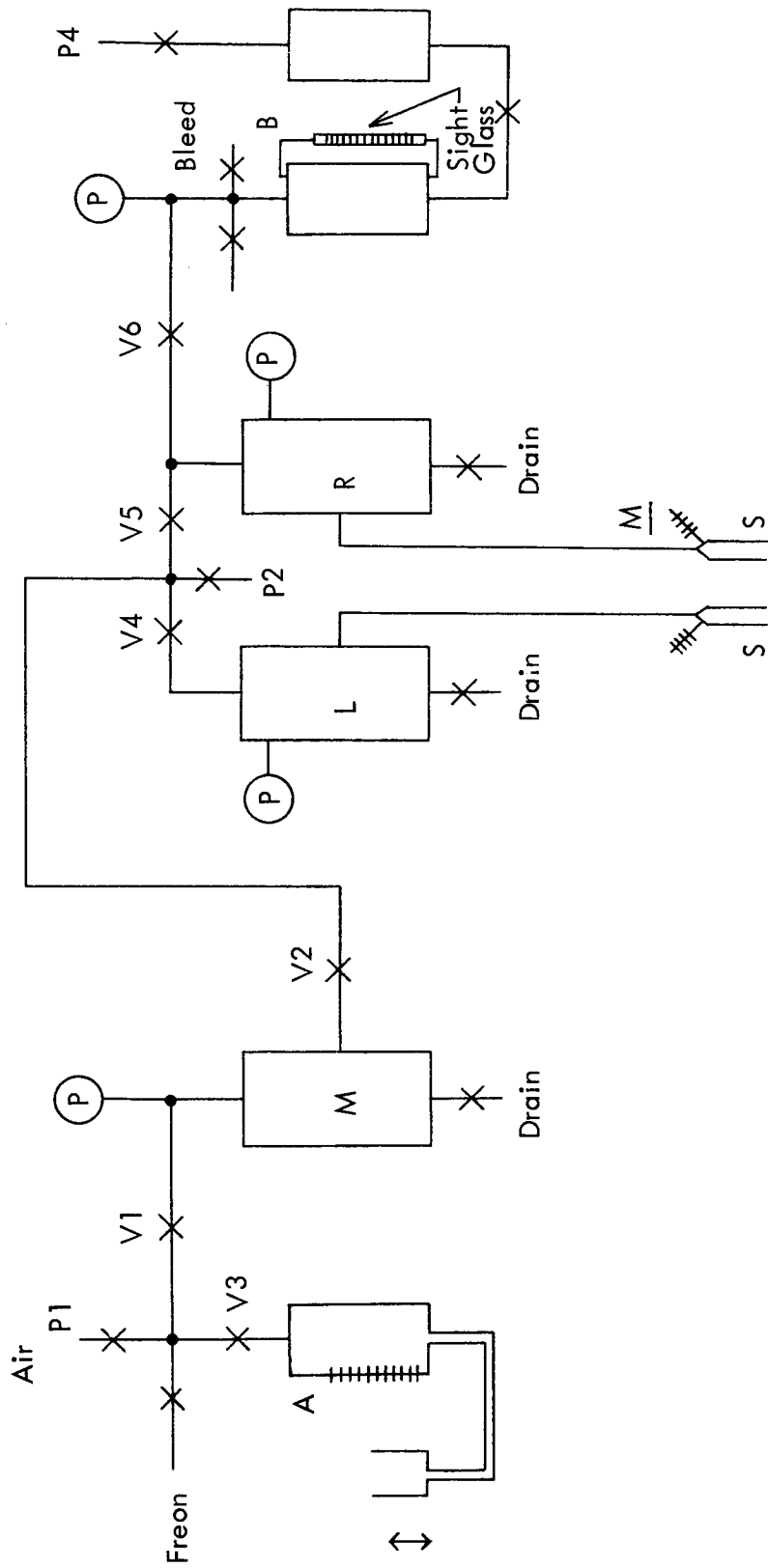
and since the ratio of (air from B) to (air in L) is a number considerably less than 1,

$$\Delta \text{ ppm} = (\text{ppm})_L \cdot \frac{(\text{vol. from P}) \cdot P_B}{(\text{vol. of L}) \cdot P_L}$$

$$= 21.5 \left[\frac{207 \times 5}{14,400 \times 4} \right] = 0.408 \text{ ppm}$$

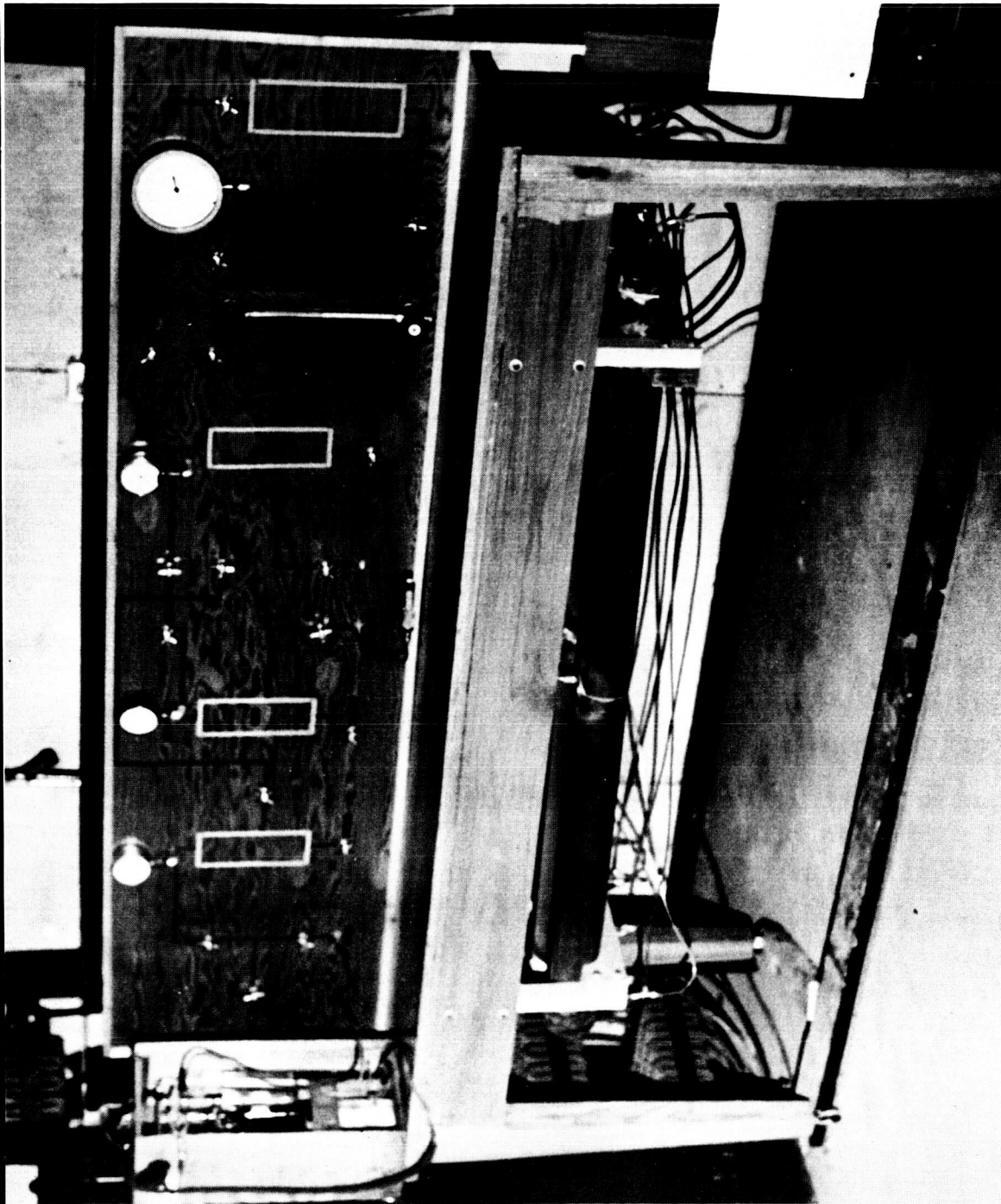
In the example above, the result was 21.5 ppm in the left tank, and (21.5 - 4.08) or about 21.1 ppm in the right tank. This would allow a test of a gradient-sensing detector at this level of concentration and 0.4 parts per million difference.

As can be seen from the example, the accuracy of the absolute level of concentration depends directly on the accuracy of volume and pressure measurements. The accuracy of the percentage difference in concentration, $\frac{\Delta \text{ ppm}}{\text{ppm}}$, depends solely on the accuracy of volume and pressure measurements in apparatus B and tank R. It is not difficult to get a 1% accuracy in these parameters by careful water displacement volume measuring techniques, and by use of a manometer for pressure measurements.



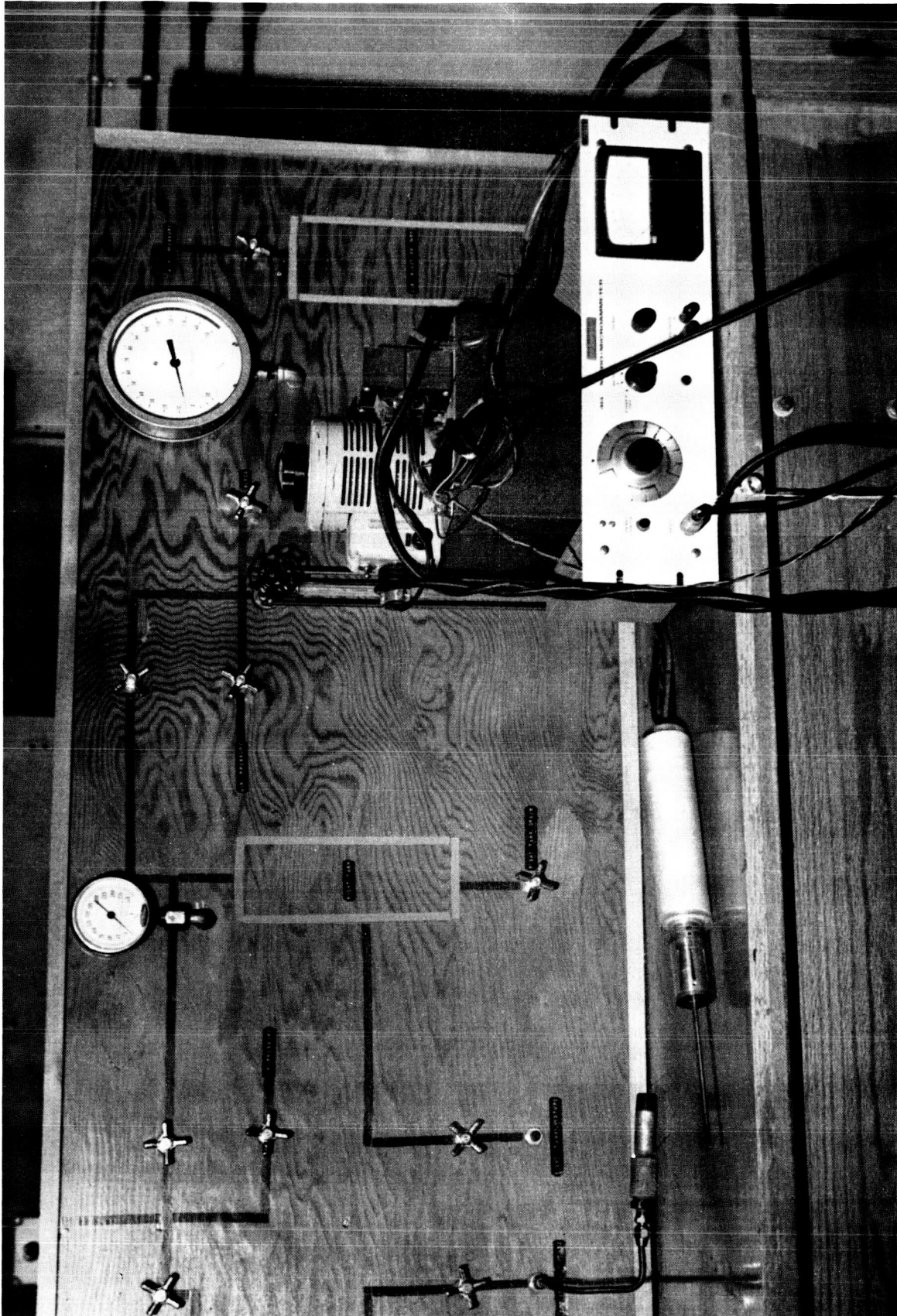
Leak Detector Testing Facility

Figure 23



Leak Detector Testing Facility

Figure 24



Apparatus Being Tested On Leak Detector Facility

Figure 25

CHAPTER V

THE HELIUM SEPARATION PROBE

The idea of using a semi-permeable membrane to improve the concentration of a test gas so that it could be more easily detected first originated with the discovery of an article in the Scientific American³ which discussed the use of a palladium-silver alloy by the Humble Oil Company in concentrating carbon monoxide gas by removal of hydrogen. This process uses the property of the alloy to pass hydrogen at high temperatures (about 700° F.). Other companies use this process to produce ultra-pure hydrogen. It was hoped that if such an approach could be used to concentrate hydrogen, a similar process might be possible for the separation of helium (or other good leak detection tracer gases) from an atmospheric environment.

A search of the literature revealed an article by G.A. Williams⁹ and J. B. Ferguson originally printed in the Journal of the American Chemical Society in 1922. This article dealt with the use of silica glass as a permeable membrane capable of allowing the diffusion of both hydrogen and helium while retarding the flow of almost all other gases normally found in the atmosphere. No detectable leakage of either air or pure nitrogen was found through cylindrical silica glass tubes at pressures up to one atmosphere and temperatures up to 880°F. Tests were made on evacuated glass vessels with one atmosphere external pressure at temperatures in excess of 300°F. Pressure variations were also studied but it was hoped that the simplest system for leak

detection would be one which operated at one atmosphere on an evacuated or semi-evacuated probe unit. According to this article⁹ no apparent diffusion occurred below 300°F but above this temperature the permeability of the glass took on an exponential variation as a function of temperature.

An interesting result of this study was that at 500°F helium has a relative permeability approximately 27 times that of hydrogen. Wall thicknesses of 1 mm. were used in these tests. It was hoped by our group that by decreasing the thickness of the glass, useful response might be expected at room temperature or at least at a substantially lower temperature than 300°F. Unfortunately an article by Leiby and Chen⁴ indicated unusably long permeation time constants.

To test this idea a brass test cell was constructed with a grill work of 0.122 inch holes on the front face. (see Fig. 28 A) A microscope slide cover glass was then fastened to this face plate with DeKhotinski Cement. The area of the exposed glass face surface was about 1/2 square inch and the thickness of the slide cover was approximately 4 mils.

This test probe was connected to a Model M-60, General Electric Mass Spectrometer with a 12-inch length of rubber vacuum tubing. Although a good vacuum seal was achieved, there was no detectable response when helium was discharged in the area surrounding the test cell face.

Since this was not an unsuspected result, in the next attempt several thin wall glass bubbles were blown from a silica glass stock (Fig. 26) The thickness of these bubble walls was about 1 mil. Tests on these were also unsuccessful at room temperature

when connected to the mass spectrometer as previously described.

In the final test, a heating jacket for the thin wall glass bubbles was designed which would give a temperature between 300°F and 400°F. (Fig. 27A & 27B) Due to lack of an experienced glass worker and contract time limitations, these tests were never completed. The teflon membrane described below is clearly superior to silica glass in terms of permeability, time response, and desirable environmental requirements.

During these tests a major effort was made to obtain samples of palladium-silver alloy for similar tests. These were not easily found and were impossible to obtain on short notice due to an understandable reluctance on the part of the industries concerned to supply this material.

Since glass did not appear to hold any immediate promise, the brass test cell was modified (Fig. 28A & 28B) to accommodate plastic film membranes. Tests on rubber, saran, nylon, and polyethylene membranes showed these films to be unsatisfactory for our needs. Literature on the permeability of these materials discovered after the tests were completed agreed with this result. (For detailed information on characteristic response data on these and other plastic membranes see references 1,2,5,6,8, and 10.)

Teflon was the next indicated choice for testing. The first sample obtained for the project was 1/8-inch sheeting which did not show promising properties when tested. Several unsuccessful attempts were made to machine this sheeting into a thin film. Finally, a roll of teflon gummed-tape was located which proved to be

quite satisfactory for our needs.

The teflon tape was of the FEP type with a thickness of 3 mils and a width of 1 inch. The gum was removed from the back and the tape was sealed to the brass test cell by pressure on the edges around the honey-comb grill face plate. (Fig. 28 and 29)

The brass test cell was connected to the General Electric Mass Spectrometer, Model M-60, with an eight inch piece of high-vacuum rubber hose. The vacuum seal was not difficult to obtain if care was taken in positioning the teflon membrane and in tightening the face plate bolts.

The lower helium detection limit on the G.E. ¹¹M-60 is approximately 2×10^{-9} cc/sec and no difficulties were found in reaching the required vacuum with the 3 mil teflon membrane in place on repeated tests. Shutting off the diffusion and fore pumps of the mass spectrometer resulted in a gradual increase in pressure, showing that some air was being drawn through the membrane but with the pumps in operation the lowest pressure reading on the mass spectrometer could easily be maintained. No measurements were made on the diffusion rate of air through the teflon membrane.

The General Electric, Model M-60, Mass Spectrometer is a general purpose vacuum system leak detector. It is tuned for helium as a test gas. No changes were made in this commercial unit when tests were made for the project. This is an older model unit and does not have either high capacity pumps or great sensitivity, however, the tests conducted on the FEP teflon tape showed good response. If newer and more up to date spectrometers were used, the response should be better than the

results quoted in the following section.

Helium gas was introduced at the face of the test cell by means of a small rubber balloon. No response was observed when the gas was released around the rubber hose connections or the sides of the test cell. Helium released on the teflon membrane itself resulted in full scale deflection of the mass spectrometer gauge in both its most sensitive (X 500) and second most sensitive (X 50) setting. No absolute calibration was made of the unit so the actual amount of helium passing through the membrane could not be determined.

No appreciable time delay was observed between the release of helium near the face of the teflon membrane and the response of the spectrometer. Later tests showed that the response time was in the order of 0.2 second. The introduction of a second sheet of teflon tape over the face of the test cell increased the response time by about 0.1 seconds, and decreased the sensitivity by a factor of approximately three. From this test it was concluded that the response was not due to pinholes in the teflon membrane, but rather the result of the permeability of teflon to helium. Literature discovered after these tests were completed bore out this conclusion.⁷

The main industrial interest at present in the permeability of teflon membranes to helium is for the separation of this element from natural gas without going to the expense of cooling all of the fluid to a liquid state and then using a fractionation process for the separation. An article by Stern, Sinclair, Gareis, Vahldieck, and Mohr, published in 1961 justifies our general conclusion that teflon FEP has

desirable permeation properties.⁷

This article⁷ points out that silicone glass is permeable only to helium and leads to very high purity of diffused helium that can be obtained by a single separation process. Unfortunately the rate of helium permeation is low at room temperatures. Since we are interested in short response times, it is essential to have a fast, reasonably good permeability even if minute quantities of other gases also pass through the membrane. Tests related by Stern, et al., show that teflon FEP has just such characteristics.

All permeation is dependent on a pressure differential across a membrane. The actual permeation process of helium through any plastic is a rather complex transport action in which the helium dissolves in the plastic film on the high-pressure side, diffuses through the membrane and evaporates from the low pressure side. Studies of teflon⁷ showed that above 60°C the permeability is independent of the pressure differential across it. This is true not only for helium but also for nitrogen and methane. Below 60°C, helium permeability⁷ was found to depend only on temperature variation but the permeability of other gases show marked influences from not only temperature and pressure differentiation but also on the nature of the diffusing gas and on the thermal history of the membrane under test. These results do not appear to be great enough to influence the use of teflon membranes for helium separation in mass spectrometer detection probes. Figure 31 compares the permeability of helium (and methane) as a function of temperature and pressure differential. It should be noted that at room temperature and atmospheric pressure there is still a

:

factor of 10 difference between these permeabilities. It is not until pressures of 4 or 5 atmospheres are reached that the permeabilities begin to approach one another.

The permeability of helium through teflon increases in an approximately exponential relationship with temperature increase over a wide range. Teflon FEP shows a high stability at temperatures up to 200° C which is not found for most plastic membranes. The permeabilities of several membranes for helium are shown in Figure 32. Teflon FEP is one of the most permeable membranes to helium as seen from the figure. Complete data on silicane rubber⁷ and related substances are not given but these substances do not have the thermal and physical stability and endurance that teflon exhibits.

In comparing the permeation sensitivity of a substance for gas (A) with respect to another gas (B), a separation factor is computed as the ratio of the permeability constants of the two gases at a given temperature.

$$S_{AB} = P_A/P_B$$

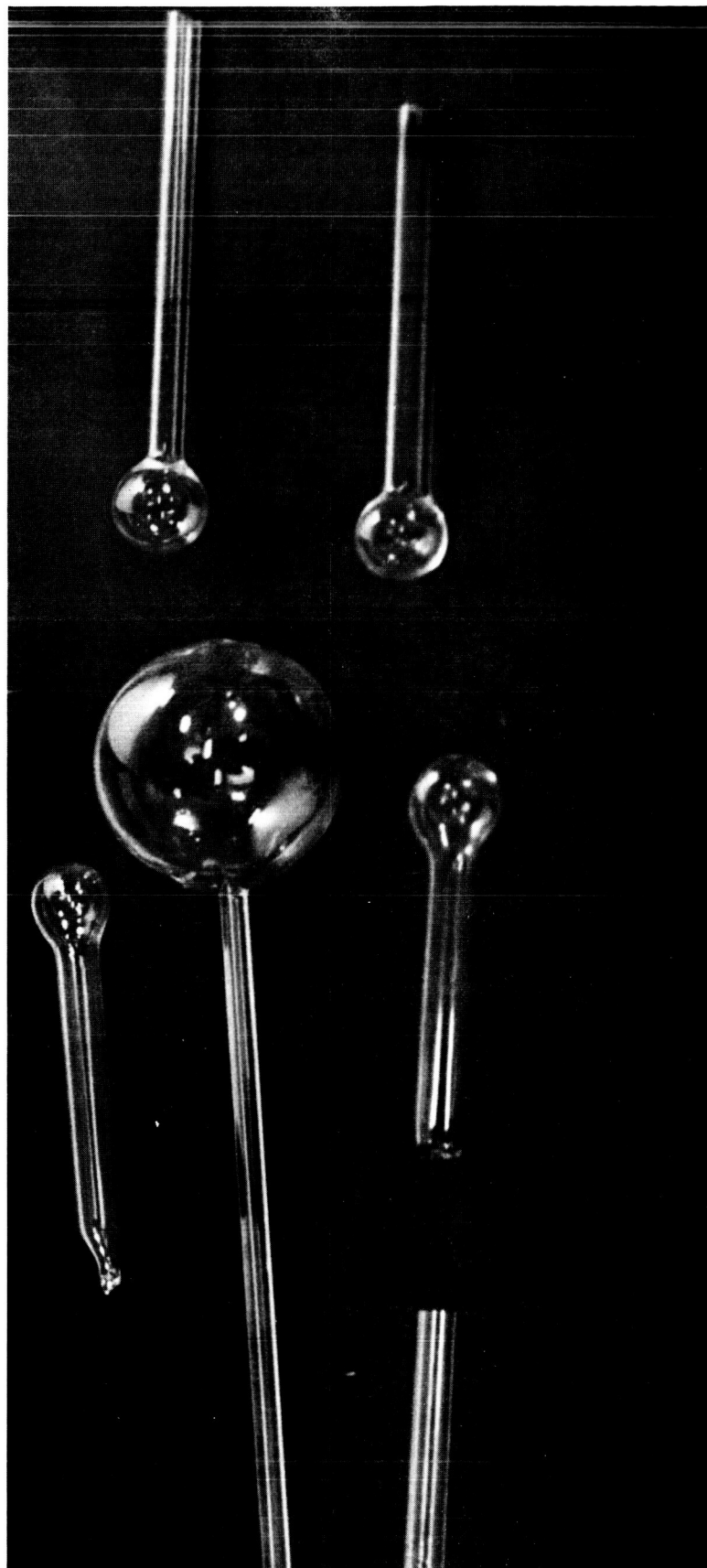
An S greater than one shows a higher penetration sensitivity of a given membrane for gas A than gas B. The separation factors as well as the absolute permeability constants of helium, nitrogen, and methane are shown in Table 5. In each case helium is taken as gas A, so the larger the number the better the separation of the

two gases by a membrane of the given substance. Note that silicone rubber has a very low separation factor for nitrogen and is even better at passing methane than helium. Teflon FEP appears to offer one of the best combinations of separation factor and absolute permeability.

It should be noted that teflon FEP is not the only form of teflon available. Type TFE⁷ has very similar permeation properties to those of type FEP. FEP has the advantage over TFE in that it is a true thermo-plastic substance so that thin films can easily be fabricated. It also has the advantage that it can be heat bonded to itself and to certain metals which might be a great help in the fabrication of production line models of the mass spectrometer probe.

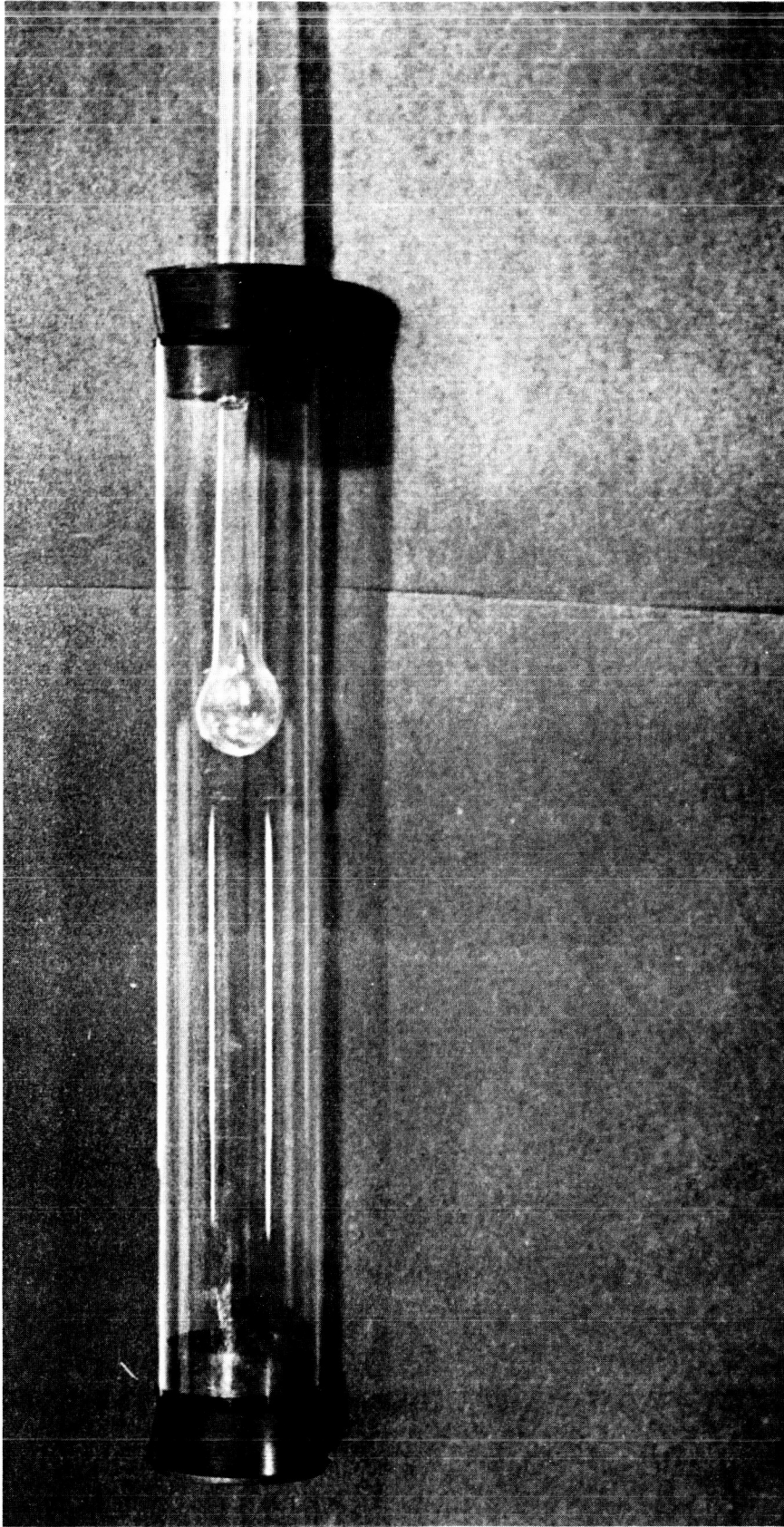
Stern, et al.,⁷ also reports that the lifetime of a teflon membrane exposed to natural gas for periods in excess of 4000 hours showed no signs of deterioration or saturation with helium or other natural gas components. Although no tests of this type were carried out in connection with this contract, it can be assumed that lifetimes of at least this long can be expected from spectrometer probes using teflon films to separate helium from normal air.

In response to a request from the Huntsville personnel, a teflon FEP helium separation probe will be delivered at the conclusion of this contract. This should provide real advantages over the pinhole type probes currently in use.



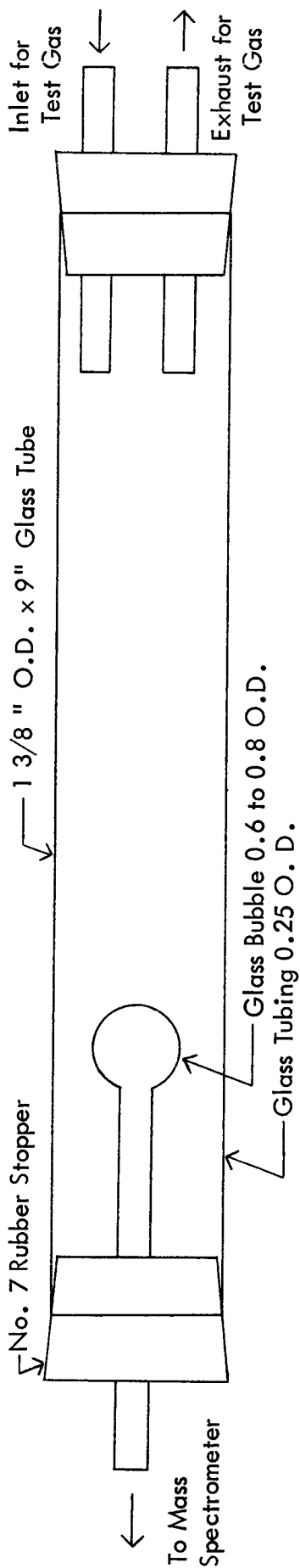
Thin Silica Glass Bulbs Used In The Study of Permeability of Glass Membranes

Figure 26



Glass Test Apparatus Used in Testing Glass Bulb Membranes

Figure 27 A

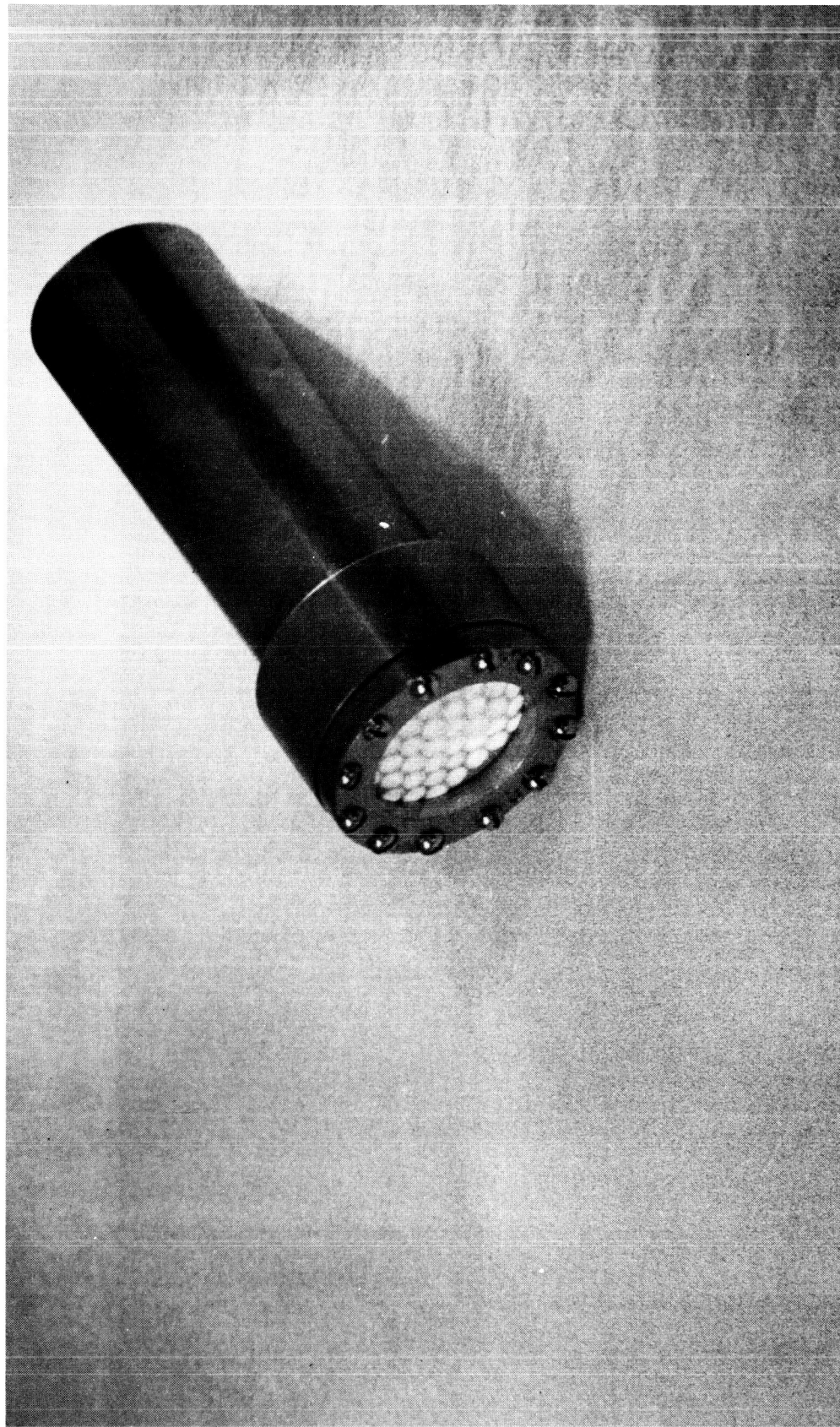


Note: For thin glass bubbles, the test gas was introduced inside the bubble and the large diameter tube was connected to the mass spectrometer.

Glass Bulb Test Cell Drawing

scale 3/4" = 1"

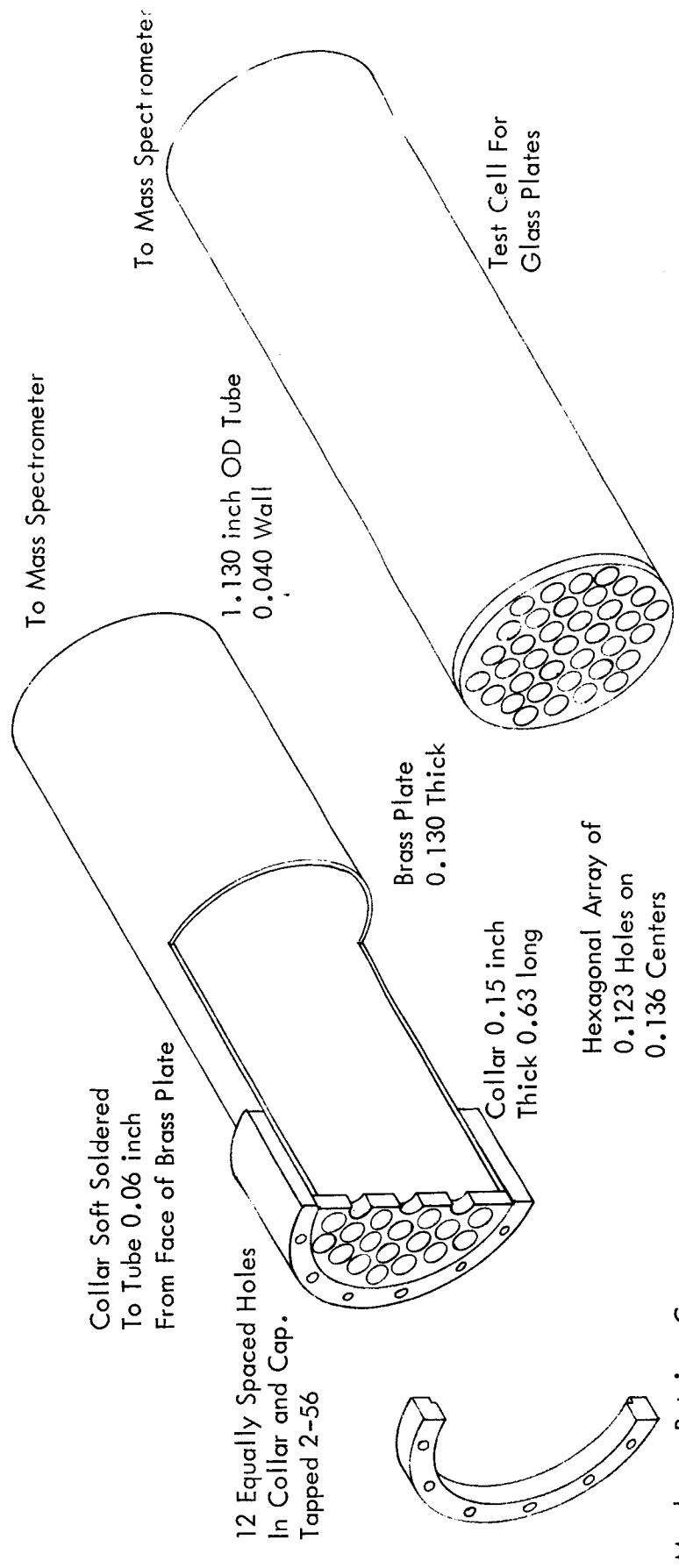
Figure 27 B



Assembled Brass Test Cell With Teflon Membrane

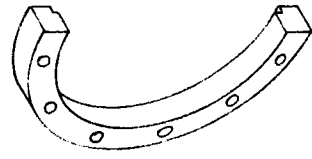
Figure 28 A

Test Cell For Membranes



Collar Soft Soldered To Tube 0.06 inch From Face of Brass Plate

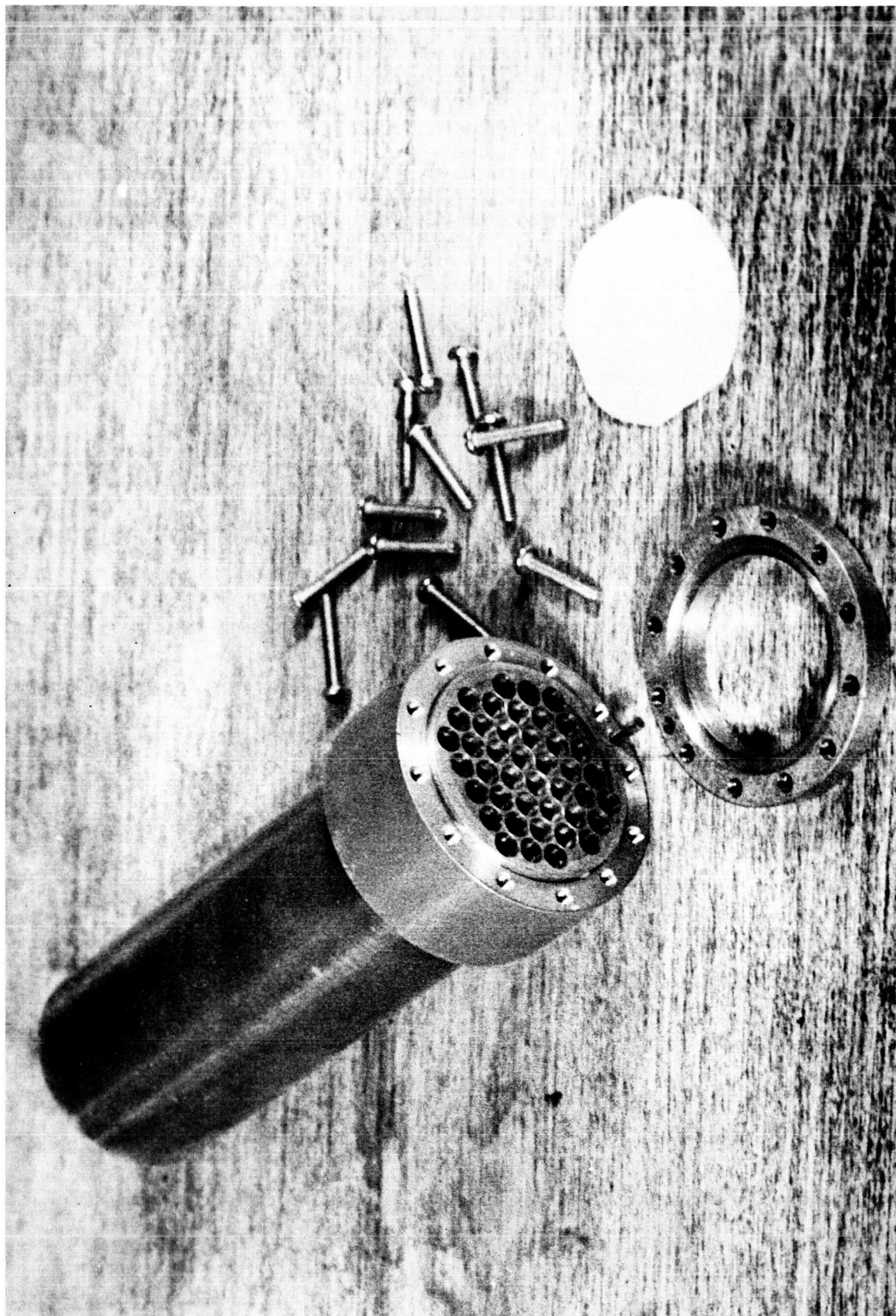
12 Equally Spaced Holes In Collar and Cap. Tapped 2-56



Membrane Retainer Cap 1.0 inch I. D. Grooved Lip 1.15 inch I. D. Cap 0.20 long.

Brass Test Cell Drawing

Figure 28 B



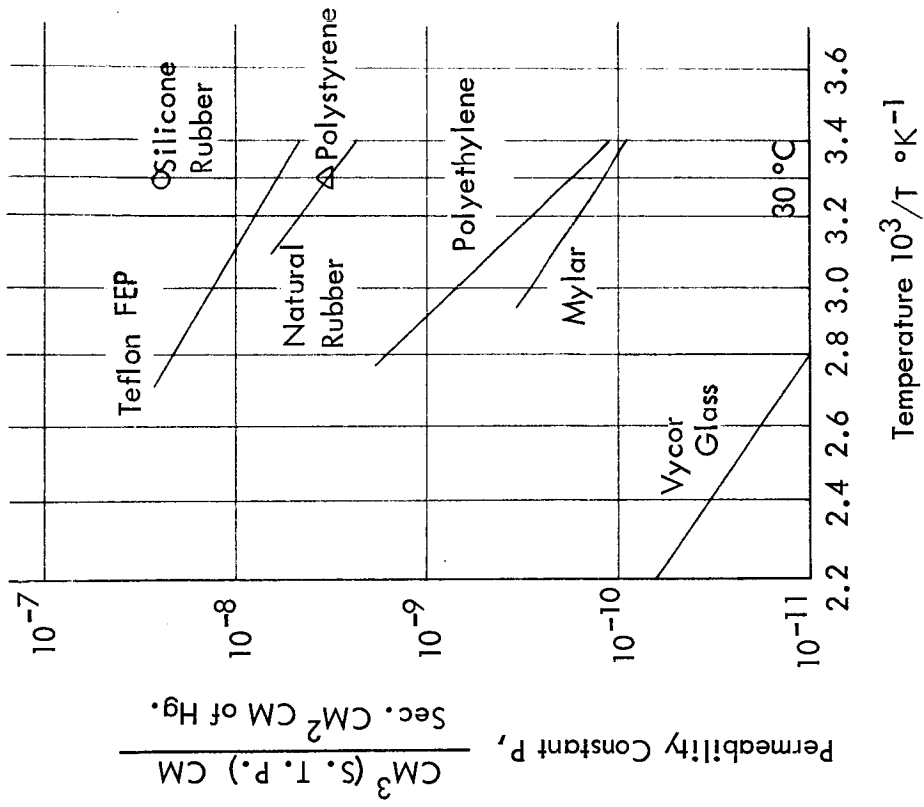
Brass Test Cell With Teflon Membrane Shown Disassembled

Figure 29



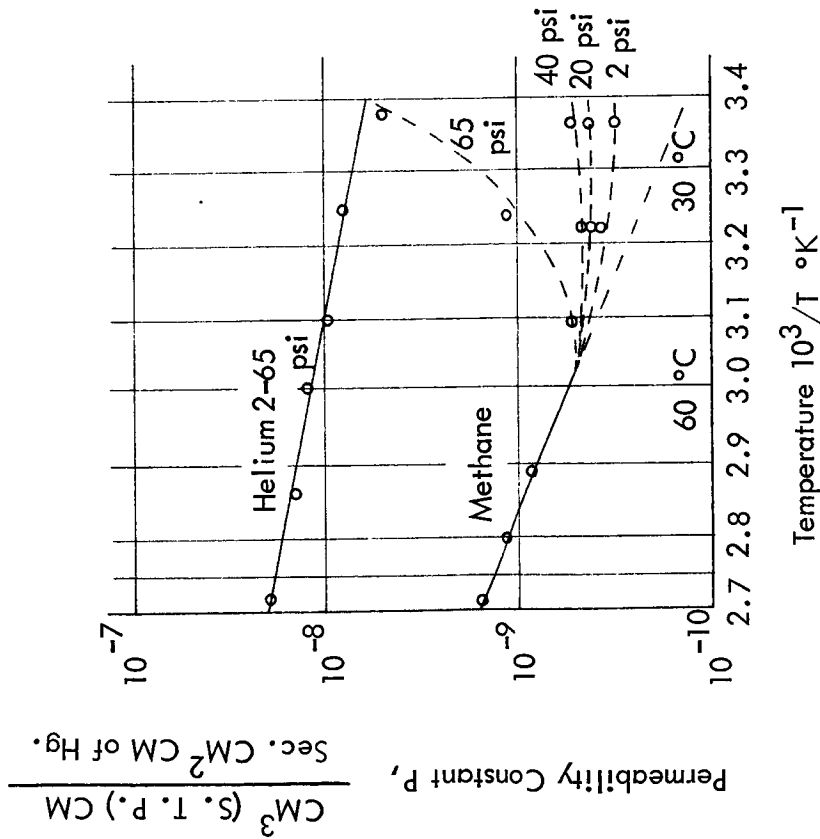
Brass Test Cell With Teflon Membrane Mounted
On General Electric Mass Spectrometer, Model M-60

Figure 30



Permeability of Various Membranes To Helium As A Function of Temperature

Figure 32



Permeability of Teflon FEP to Helium and Methane As A Function of Temperature

Figure 31

Membrane	* Permeability Constant, P $\frac{\text{Cm.}^3 (\text{STP}) \text{Cm.}}{\text{Sec. Cm.}^2 \text{Cm. of Hg}}$					Separation Factor	
	T (°C.)	He	N ₂	CH ₄	S _{He-N₂}	S _{He-CH₄}	
Silicone rubber	30.0	2.3×10^{-9}	1.5×10^{-8}	5.9×10^{-8}	1.5	0.39	
Phenylene silicone rubber	30.0	1.5×10^{-8}	0.40×10^{-8}	2.0×10^{-8}	3.8	0.75	
Nitrile silicone rubber	30.0	0.79×10^{-8}	0.21×10^{-8}	1.0×10^{-8}	3.8	0.79	
Polycarbonate	30.0	6.7×10^{-9}	0.46×10^{-9}	0.36×10^{-9}	15	19	
Teflon FEP	30.0	6.2×10^{-9}	0.25×10^{-9}	0.14×10^{-9}	25	44	
Natural rubber	30.0	3.6×10^{-9}	1.05×10^{-9}		3.4		
Polystyrene	30.0	3.5×10^{-9}	0.22×10^{-9}	0.23×10^{-9}	16	15	
Trithene B	30.0	3.4×10^{-9}	0.012×10^{-9}	0.0084×10^{-9}	280	400	
Ethyl cellulose	30.0	3.1×10^{-9}	0.28×10^{-9}	0.64×10^{-9}	11	4.9	
Ethylene-vinyl acetate	30.0	2.1×10^{-9}	0.28×10^{-9}	1.1×10^{-9}	7.5	1.9	
Viton A	30.0	1.7×10^{-9}	0.031×10^{-9}	0.016×10^{-9}	55	110	
Polyvinyl chloride (plasticized)	30.0	1.4×10^{-9}		0.2×10^{-9}		7	
Polyethylene	30.0	1.0×10^{-9}	0.19×10^{-9}		5.3		
Polyvinyl fluoride	30.0	1.8×10^{-10}	0.019×10^{-10}	0.0065×10^{-10}	95	280	
Mylar	25	1.0×10^{-10}	0.006×10^{-10}	0.006×10^{-10}	170	170	
Saran	25	6.6×10^{-12}	0.018×10^{-12}	0.025×10^{-12}	370	260	
Vycor glass	400	4.8×10^{-10}	0.00063×10^{-10}		7600	Very large	
Vitreosil	30	6.4×10^{-11}			Very large	Very large	
	400	3.2×10^{-9}			Very large	Very large	

* Permeability Constant, P: defined as cubic cm. of gas passed (referenced to Standard Temperature and Pressure) per unit time, per unit area of membrane in square cm., per cm. of Hg. pressure differential across membrane, times thickness of membrane in cm.

Helium Permeability and Separation Factors⁷

Table 5

REFERENCES

1. Brubaker, D. W., Kammermeyer, K., *Ind. Eng. Chem.* 44,1465 (1962)
2. *Ibid.*, 46,733 (1954)
3. International Nickel Company, Inc. N.Y., N.Y. (*Adver. in Sci. Amer.*) (1966)
4. Leiby, C.C., Jr., Chen, C.L., *J. Appl. Phys.* 31,268 (1960)
5. Meares, P., *J. Amer. Chem. Soc.* 76,3415 (1954)
6. Sarge, T. W., *Anal. Chem.* 19,396 (1947)
7. Stern, S.A., Sinclair T.F., Gareis, P. J., Vahldieck, N. P., Mohr, P. H., *Ind. Eng. Chem.* 57,3, 49 (1965)
8. Van Amerongen, G. J., *J. Polymer Sci.* 5,307 (1950)
9. Williams, G. A. and Ferguson, J. B., *J. of Amer. Chem. Soc.*, V XLIV, No. 10 (1922)
10. Weller, S., Steiner, W. A., *J. Appl. Phys.* 21,279 (1950)
11. *Instruction and Operation Manual for General Electric Mass Spectrometer, Model M-60.*

CHAPTER VI

THE THERMAL CONDUCTIVITY PRINCIPLE

In order for a thermal conductivity detector to attain the same degree of sensitivity as the present halogen detector, it must be capable of detecting a change in thermal conductivity resulting from a change of about 1 part per million of the tracer gas in air. Since the ratio of thermal conductivity of the air with tracer to that of standard air, $\frac{K}{K_0}$, is approximately $1 + E\sqrt{\frac{m_0}{m}}$ where E is the mole fraction of tracer, and $\frac{m_0}{m}$ is the ratio of molecular weight of air to tracer, then $\frac{\Delta K}{K_0}$ is $E\sqrt{\frac{m_0}{m}}$, or for air and helium, $\frac{\Delta K}{K} = E\sqrt{29/4}$ or roughly, $3 E$.

Thus the change in thermal conductivity can be expected to be on the same order of magnitude as the parts per million of tracer, and in order to detect a part per million of helium, the thermal detection must be able to sense a change of 1 to 10 parts per million of thermal conductivity. This in turn would require an ability to detect a change of current of the same order of magnitude in a constant current thermal detector. This small change must be separable from changes due to ambient temperature, or much worse, random variations in humidity or other ambient contaminants. It is highly probable that such contaminant variations would impose noise much greater in magnitude than 10 ppm on the signal.

A partial solution to the atmosphere contamination problem, and to the sensitivity problem in general, would be a selective filter between atmosphere and detector. But this method has obvious problems of its own, including discovery of adequate filter-tracer combination, time lag, pressure drop requirement,

contamination and clogging by atmospheric dirt.

In summary, since the thermal conductivity detector depends on variations in properties which are not exclusively the result of the tracer, and since the percentage variation of the property is low relative to the tracer concentration, the thermal conductivity meter does not look very promising as a detector of leaks of the size currently found by the halogen detector.

CHAPTER VII

LITERATURE SEARCH UPDATING

The search of the literature begun under NAS8-2563 has been continued under NAS8-11199 so that a listing of over 3000 IBM entries and cross references on leak detection have now been listed and abstracted. Figure 33 shows the format of the abstract listings. The abstract number is given in the upper right hand corner. The numbers in the left hand corner are first the source reference number, then the last two digits of the year the article was published, and finally the subject classification number. Directly under this are listed the author or authors of the article. The complete source listing and title appear at the upper right center and the abstract and pertinent information on the article appears below this in paragraph form.

These articles have also been categorized into 98 subject classes and subclasses so that any particular type of leak detection information can be found easily. A listing of these classes and subclasses is given in Table 6. In order to facilitate the sorting of these articles, each has been catalogued on both IBM title cards and IBM author cards. (For details on the formats of these cards see Annual Report No. 2-b of Leak Detection Technique Improvement Study For Space Vehicles, September 1966, Pages 12 and 13.)

Subject sorting can be done on three levels by using only the first, the first and second, or all three digits of the subject classification number. The first number is a broad subject classification while the second and third break these down into smaller, more specific subclasses. The computer program which does this sorting has been written so that a complete printout of the useful articles is done by the computer at the time

of the sort. The abstracts of these articles can then be looked up in the text of this report. The section on computer program information gives the details of the program used for the computer sorts.

Articles abstracted in the bibliography updating since September 1966 contained in this chapter are designated by numbers beginning with 10001. Note that the articles abstracted in Annual Report 2-b (Literature search portion) which included all articles abstracted from January 1962 to September 1966 are designated by numbers 00010 through 07295.

378 - 51 - 100

00550

Blears, J.
Leck, J. H.

J. Sci. Instr., Supl. 1 20-8 (1951)

General Principles of Leak Detection

The techniques of leak detection are reviewed, special emphasis being placed on methods for obtaining high sensitivity with simple apparatus. A theoretical analysis of the gas flow problem is supplemented by experimental results obtained by using two diffusion pumps in cascade. The importance of using constrictions and correctly positioning the gage is stressed and the losses of sensitivity due to virtual leaks, leaks in series and by-pass methods are considered. The relative sensitivities of different leak detection methods are tabulated. (auth.)

Deals with mechanical, gage type, leak detectors where pressure differences in the entire system must be measured. No discussion is given to locating individual leaks, only showing if system is leak tight.

FIGURE 33

FORMAT USED FOR ABSTRACT INFORMATION SHOWING CROSS-REFERENCE NUMBER USED IN
IBM CLASSIFICATION AT TOP RIGHT

Computer Program Information

A computer program has been written which will sort through the subject and/or author cards decks and select the cards whose classification numbers correspond to those given by a control card. In this way a printed list of references can be obtained quickly.

The program is written for the IBM system/360 in Fortran IV (level E). The data deck contains a control card followed by the subject and/or author deck. A blank card is placed as the last card of the data deck and is used to stop the program.

The control card contains four numbers in integer format in the first four five column fields. The first number determines how many subdivisions are to be considered. If this number is 2 only the main and first subdivision will be considered. The next three numbers determine the main classification, the first subdivision, and the second subdivision to be listed on the output. If the control card contains the numbers 2,4,3, and blank, those cards whose main classification is 4 and whose first subdivision is 3 would be listed. The second subheading is not given because the first number on the control card (2) indicates that the process will not consider any division beyond the second.

TABLE 6

SUBJECT CLASSIFICATION AND SUBCLASSIFICATION

1. Gas Detection
 11. Thermal Conductivity
 111. Hot Wires
 112. Thermistors
 113. Thermocouples
 114. Thermonic Emission
 115. Thermal Expansion
 116. Thermal Diffusion
 12. Combustion Meters
 121. Hydrogen Detectors
 13. Ionization Detectors
 131. Halogen Guns
 132. Halogen Bridges
 133. Ionization Gauges
 134. Field Emission Devices
 14. Electric, Magnetic, and Electromagnetic Devices
 141. Vibrating Capacitors
 142. Paramagnetic Detectors
 143. Ultraviolet

- 144. Infrared
- 145. Tesla Coils and/or Spark Discharge
- 146. Resistivity and Impedance Detectors
- 147. Electric Conductivity
- 148. Photoelectric Processes
- 15. Analyzers
 - 151. Mass Spectrometers and Omegatrons
 - 152. Miniature Mass Spectrometers
 - 153. Chromatographs
 - 154. Chemical Responders and Reactions
 - 155. Microwave Devices
 - 156. Sonic Analyzers
 - 157. Interferometers
- 16. Olfactory Tracers
 - 161. Mercaptans
 - 162. Other Compounds
- 17. Optical Methods
 - 171. Luminescence
- 18. Flame Testing
- 19. Dyes and Other Color Change Methods
- 2. Radioactive Tracer Methods
 - 21. Radioactive Gases
 - 211. Kr85
 - 212. Radon

- 22. Radioactive Detectors
 - 221. Alpha
 - 222. Beta
 - 223. Gamma
 - 224. Neutron
- 23. Health and Safety
- 3. Liquid Leak Detectors
- 4. Acoustical Detection Systems
 - 41. Passive
 - 412. Sonic Listeners
 - 413. Ultrasonic (translators)
 - 42. Active
 - 421. Sonic Injection
 - 43. Active-Passive
 - 431. Sonic Modulation of Leak Noise
 - 44. Acoustical Transducers
 - 441. Audio Microphones
 - 442. Ultrasonic Microphones
 - 443. Semiconductor Microphones
 - 444. Seismic Devices
 - 445. Directional Horns etc.
 - 45. Related Circuitry
 - 451. Correlation Detectors

- 452. Noise Immunity Circuits
- 453. Doppler Shift Sensitive Devices
- 5. Pneumatic and Hydraulic Devices
 - 51. Manometers
 - 52. Diaphragms
 - 53. Soap Bubbles, and other Bubble Detection Methods
 - 531. Techniques
 - 532. Solutions and Mixtures
 - 54. Pressure Gauges (spring type)
- 6. Gas Dynamics and Gas Properties
 - 61. Gas Dynamics
 - 611. Nozzle Dynamics for Detectors
 - 612. Gas Flow and Diffusion in Closed Pipes
 - 613. Gas Flow from and Around Leaks
 - 614. Supersonic Gas Flow
 - 62. Gas Properties
 - 621. Densities
 - 622. Diffusion Properties
 - 623. Ionization Potentials
 - 624. Other Electromagnetic Properties
 - 63. Permeable Membranes
- 7. Useful Circuitry
 - 71. Transistorization

- 711. Amplifiers
 - 712. Level Sensing Circuits
 - 713. Indicator Circuits
 - 714. Power Supplies
 - 715. Economizers
- 8. Devices Related to Leak Detectors
 - 9. Phenomena of Possible Applicability

TABLE 7

Journal Listings with IBM Identification Number

001	Acta Chemica Scandinavica
002	American Gas Journal
003	American Industrial Hygenic Association Journal
004	Analytical Chemistry
005	Analyst
006	Annual New York Academy of Science
007	Annales De Radioelectricite
008	Applied Scientific Research
009	Applied Spectroscopy
010	Archives Biochemistry and Ciophysics
011	Australian Journal of Physics
012	Australian Journal of Chemistry
041	Biochemical Journal
042	Brennstoff-Chemie
043	British Patent Office
044	British Journal of Applied Physics
045	Bulletin Academy of Polotechnic Science
046	Bulletin Academy Royal Belgique Cl. Science
047	Bulletin of the Institute of Chemical Research, Kyoto U.

- 048 Bulletin Laboratory Chim. Provinciali
- 049 Bulletin of the Society of Chimestry (France)
- 050 Bulletin of Society Science Bretagne
- 051 Boll. Lab. Chim. Provinciali
- 052 British Chemical Engineering
- 053 Bell Laboratory Record

- 081 Cahiers de Physic
- 082 Canadian Journal of Physics
- 083 Canadian Journal of Research
- 084 Canadian Journal of Technology
- 085 Chemical Abstracts
- 086 Chemical Engineering
- 087 Chemical Engineering Progress
- 088 Chemical Metallurgy
- 089 Chemical Technology
- 090 Chemicke Listy
- 091 Chemicky Prumysl
- 092 Chemie
- 093 Chemiker-Zeitung
- 094 Chemistry in Canada
- 095 Chemistry and Industry
- 096 Chimia (Switz)

- 097 Chimestry and Industry (London)
- 098 Chimica et Industria
- 099 Ciencia
- 100 Civil Engineering
- 101 Collection Czechoslov Chemical Communications
- 102 Colloid Chemistry
- 103 Comptes Rendus
- 104 Chemical Metallurgy Engineering
- 105 C. R. Academy of Science (Paris)
- 106 Chemical and Engineering News

- 121 Dechema Monograph
- 122 Disseratation Abstracts
- 123 Doklady Akadamy Nauk, SSSR
- 124 Drung and Cosmetic Industry
- 125 Diesel Power

- 161 Electrical Engineering
- 162 Electronic Industries
- 163 Electronic Technology
- 164 Electronic World
- 165 Electronics
- 166 Electrotech U. Maschinenban
- 167 Elektrotechnik (Berlin)

- 168 Experimentia

- 201 Farm Science and Technology
- 202 Fonderie
- 203 Fluid Handling
- 204 Factory

- 240 Gas
- 241 Gas Age
- 242 Gas Chromatography
- 243 Gas Council Research Commun. (Eng.)
- 244 Gas Journal
- 245 Gas und Wasserfruh
- 246 Gas World
- 247 General Electric Review
- 248 Gas Instruments and Techniques

- 281 Hochvakuum-Technology
- 282 Heating-Piping
- 283 Hydraulics and Pneumatics
- 284 Health Physics

- 320 IBM Journal of Research and Development
- 321 Indian Journal of Physics
- 322 Industrial and Engineering Chemistry

- 324 Industrial Chemist
- 325 Industrial Chemistry Analytical Edition
- 326 Industrial Radio Engineering Proceedings
- 327 Instruments
- 328 Instruments and Control Systems
- 329 Instruments and Experimental Techniques
- 330 International Journal of Air Pollution
- 331 IRE Trans. of Industrial Electronics
- 332 Iron and Steel (England)
- 333 ISA Journal
- 334 Italia
- 335 Instrument Society of America
- 336 Instrument Practice
- 337 Industrial Design
- 338 Industrial Electronics
- 339 Industrial Equipment News
- 340 Iron Age
- 341 International Journal of Applied Radiation

- 361 Japan Society Bulletin
- 362 Jet Propulsion
- 363 Journal of American Water Works Association
- 364 Journal of Applied Physics

- 365 Journal of Basic Engineering
- 366 Journal of Chemical Education
- 367 Journal of Chemical Society
- 369 Journal of Colloid Society
- 370 Journal of Franklin Institute
- 371 Journal of Inst. Electronic Engineers
- 372 Journal of Industrial Fuel
- 373 Journal of Oil and Colour Chemists
- 374 Journal of Optical Society of America
- 375 Journal of Physical Radium
- 376 Journal of Physics Society of Japan
- 377 Journal of Royal Institute of Chemistry
- 378 Journal of Scientific Instruments
- 379 Journal of Scientific Instruments Supply
- 380 Journal of Technical Physics U.S.S.R.
- 381 Journal of Thoracic and Cardiovascular Surgery
- 382 Journal of Applied Chemistry
- 383 Journal of Chromatography
- 384 Journal of Polymer Science
- 385 Journal of the American Chemical Society

- 401 Kerntechik
- 402 Kolloid Zhur

- 403 Kagaku No Ryoiki
- 441 Laboratoire Mediterranéen de Recherches Thermodynamiques (France)
- 442 Laboratory Science
- 443 Le Vide (In French)
- 481 Magyar Kemiai Folyoirat
- 482 Makromolecular Chemi
- 483 Manufacturing Chemist and Pharmaceutical and Fine Chemical Trade Journal
- 484 Metallurgia
- 485 Microtecnic
- 486 Machine Design
- 487 Mechanical Engineering
- 521 National Engineer
- 522 National Nuclear Energy Service
- 523 National Symposium on Vacuum Technology
- 525 Nuclear Engineering
- 526 Nucleonics
- 527 Nuovo Cimento
- 528 National Bureau of Standards Circular
- 529 Nondestructive Testing
- 561 Oil and Gas Journal
- 562 Oyo Butsuri

- 601 Paper Trade Journal
- 602 Petroleum Engineering
- 603 Pharmazie
- 604 Philips Technical Review
- 605 Philosophical Magazine
- 606 Physica
- 607 Physica, 's Grav.
- 608 Physical Methods in Chemical Analysis
- 609 Physical Review
- 610 Physical Society of London Rep. Progress Physics
- 611 Physics Today
- 612 Power
- 613 Process of the Cambridge Philosophical Society
- 614 Process International Phoel. Congress
- 615 Process of the Pennsylvania Academy of Science
- 616 Proceedings of Physical Society
- 617 Product Engineering
- 618 Plaovo Gimento
- 619 Proceedings of Royal Society
- 620 Proceedings in Mechanical Engineering
- 621 Proceedings of American Rocket Society
- 622 Pipeline Industry

- 681 Record of Chemical Progress
- 682 Refrigeration Engineering
- 683 Research Engineering
- 684 Review of Scientific Instruments
- 685 Ricerca Scientifia
- 686 Revista Dei Combustibili
- 687 Review of Institute of French Petrole et Ann Combustible Liquids
- 688 Research

- 721 Science
- 722 Siemens-Zeitschrift
- 723 Slaboproudy Obzor
- 724 Southern Power and Industry
- 725 Suddent Apoth. Ztg.
- 726 Suomen Kemistilehti
- 727 Safety Maintainence

- 761 Termotecnia
- 762 Transaction American Society of Mechanical Engineering
- 763 Transactions Electrochemical Society
- 764 Transactions of the Faraday Society
- 765 Transactions of the Instrument Measure Conference (Stockholm)

- 801 U. S. Patent Department

- 802 Uspekhi Fiz Nauk

- 841 Vacuum
- 842 Vacuum Techniques
- 843 Vacuum Testing Handbook for Columbia Project
- 844 Vacuum - Technology (Brit)
- 845 Vide

- 881 Water and Sewage Works
- 882 Water and Water Engineering
- 883 Welding Journal
- 884 Westinghouse Engineering
- 885 Wiadomosci Chemistry
- 886 World Oil
- 887 Welding

- 961 Zavodskala Laboratoriya
- 962 Zeitschrift Angewandte Physik
- 963 Zeitschrift fur Electrochemie
- 964 Zeitschrift fur Instrumentenkunde
- 965 Zeitschrift fur Naturforschung
- 966 Zeitschrift fur Physik
- 967 Zhurnal Tekhnicheskoi Fiziki

- 980 All Other Unlisted Sources

- 996 U. S. Colleges and Universities

- 997 U. S. Government Contract Reports

- 998 Symposiums and Conferences

- 999 Independent Publications (Private)

329-62-130

10001

Apter, B.I.

Instruments and Experimental
Techniques 3 505-508, May-June,
1963

Katharometer Leak Detector

The industrial leak detector described is designed to detect leakage in systems operating at pressure above atmospheric. The instrument functions with any test gas whose thermal conductivity is different from that of air. Sensitivity to a flow of Freon-12 as the test gas is $2 \cdot 10^{-2}$ liter/sec. Sensitivity to other test gases is determined by their thermal conductivity.

684-62-130

10002

Barrington, A.E.

Rev. Sci. Instr., v33, n10,
1045-46 (1962)

Sensitivity of Ion Pump Leak
Detector

620-58-900

10003

Kronberger, H.

Proc. L. Mech. E. 172 (1958)

Vacuum Techniques in the Atomic
Energy Industry

529-62-144

10004

Brady, J.D.

Nondestructive Testing 332-334
(1962)

Mobile Infrared Gas Analyzer
for Gas Transmission Line Leakage
Surveys.

- | | |
|--|--|
| 621-62-100 | 10005 |
| Brown, P.E. | Proc. American Rocket Society (1962) |
| Feistman, M.L. | Multi-Point Leak Detection System-
for Space Vehicle Applications. |
| 333-58-900 | 10006 |
| Clancy, W.H. | I.S.A.J. V5, n4,60 (1958)
Detector Warns of Pipeline Leaks |
| 485-63-200 | 10007 |
| Courtois, T.R.
Gasnier, M. | Microtecnic 17 (1,2)
Feb. , 1963 pp. 27-33; April, 1963
pp. 67-83

Leak Detection by Means of
Radioelements |
| <p>Feb. Importance of rapid localazation of leaks in underground pipe systems. Three detection methods are indicated; selection of tracer for detecting leaks in liquid and gaseous media; influence of soil properties on leak detection.</p> <p>Apr. Saclay experimental pipe line; examples of application of leak detection, indicating problems encountered and solutions found; leaks in sealed containers and in mechanical units. 29 refs.</p> | |
| 688-56-900 | 10008 |
| Cross, S.H.
Steckelmacher, W. | Research 124-131 (1956)

Leak Detection by Vacuum Techniques |
| 684-35-110
133 150 | 10009 |
| Cuykendall, T.R. | Rev. Sci. Instr. v6 371-72 (1935)

Use of Pirani Gauges in Finding
Vacuum Leaks |

- 684-37-900
Kuper J.B.H.
10010
Rev. Sci. Instr. v6 n4 131 (1937)
A Vacuum Gauge for Leak Hunting
- 996-33-110
Daynes, H.A.
10011
Cambridge Univ. Press, Cambridge
England (1933)
Gas Analysis by Measurement of
Thermal Conductivity
- 378-60-140
900
10012
Dowling, D.J.
J. Sci. Instr. v37 n4 147 (1960)
Electronic Leak Detector
- 980-64-900
Dreuyder, B.Z.
10013
Energetik (USSR) n3 15-16 (1964)
In Russian
An Electronic Vacuum Gauge
- 980-34-999
Farkas, A.
Melville, H.W.
10014
MacMillian, London (1939)
Experimental Methods in Gas Reactions
- 998 - 151
Gilmour, A.S.
10015
IEEE Transactions on Aerospace-
Support Conference Procedures
Radio-Frequency Mass Spectrometers
and their Applications in Space.

980-49-900

10016

Guthrie, A.
Wakerling R.K.

McGraw-Hill 190-240 (1949)

Vacuum Equipment and Techniques

841-63-900

10017

Lee T.H.
Kurtz, D.R.
Veras, D.J.

Vacuum 13 (5) May, 1963 167-172

Problems in Detecting Leaks with
Long Time Constants in Long-life
Vacuum Devices with Sealed Glass
Envelopes.

Techniques for the detection of leaks at approximately 10^{-13} cubic centimeters per second at standard temperature and pressure. Leaks with intolerably long time constants of days and even weeks were observed; inadequacy of leak checking with helium alone; calculation of allowable bagging time which, if exceeded, would allow enough helium to diffuse into glass and raise envelopes pressure above tolerable level; methods of detection and calibration are presented.

841 - 800

10018

Huber, W.K.

Vacuum v13 399-412

Partial Pressure Measurements in
High and Ultra-High Vacuum Systems.

328-62-900

10019

Hutchens, W.C.

Instruments and Control Systems
v25 n4 107-109 (1962)

Leak Test Specifications

980-62-900

10020

Jenkins, R.O.

Instr. Elec. Engrs. Proc.
v109 ptA(supp) n3 176-183 (1962)Leak Testing during Manufacture
of Sealed-Off Vacuum Devices.

335-63-151

10021

Michel, D.

Proc. Instrum. Soc. Amer. 16 (2)
1963 5 pagesMethods of Applying the Mass
Spectrometer Leak Detector

Applications of device for testing sealed devices with exceptionally low leak rates are discussed. In order to obtain satisfactory results, realistic and sensible leakage limits should be specified. Product designers and specification writers should be familiar with various problems which can effect testing, and endeavor to eliminate such problems by proper design and specifications.

335-63-151

10022

Michel, D.E.
Smith, G.C.J. Instrum. Soc. Amer. 10 (11)
Nov., 1963 55-60Leak Detection using a Mass
Spectrometer Leak Detector.

Methods and principles of mass spectrometer leak detectors are discussed, especially those for testing extremely small leaks in enclosures used in many earth-bound manufacturing and research techniques and in aerospace equipment; methods are described for construction of fixtures required to adapt component to be tested to base plate of instrument.

203-63-300

Probert, S.D.
 Thomas, T.
 Warman, D.

10023

Fluid Handling 163, 164 260-262
 315-318 1963

Leak Technology, Leak Detection

Over pressure methods of leak detection and leak-tight construction are discussed. (15 ref.)

997- 412

Reid, J.M.
 Hogan, D.P.
 Micheal, P.L.

10024

A.G.A. Research Project PB-36

A New Approach to Pinpointing
 Gas Leaks with Sonics

240-63-100

131

Reynolds, S.L.

10025

Gas 39 (7) 1963 67-69

For Leak Detection, A Gun Proves
 Fastest in the West

Features of "Piston-Grip" detector that has search scale and 2 measuring scales. The search scale has a sensitivity of 20-100 ppm. The detector unit weighs less than 3 lb. The power required is 5-8 volts DC, 2 watts. The instrument is especially applicable on leakage survey of services only and on surveying sections of transmission line where terrain is too rugged for infrared units in 3 wheel drive vehicles.

335-63-151 10026
 Smith , G.C. ISA-Proc. Preprint 16.1.63 (1963)
Study of Methods of Constructing
 Fixtures of Testing Low Leak Rate
 Using Mass Spectrometer Leak
 Detector

It is shown that, when using Mass Spectrometer Leak Detector and tracer gas it detects, acceptable and sensible leak rates must be known or defined before study of constructing fixtures can be undertaken; careful design of fixtures with special attention to sealing materials and gaskets, is necessary to assure competent and reliable testing.

841-63-155 10027
 Stolz, W.A. Vacuum v13 n6 223-227, 1963
Microwave Tube Processing Using
 Ion-Getter Pumps

841-62-110 10028
 Steckelmacher, W. Vacuum 12 (3) May-June, 1962
 Tinsley, D.M. 153-159
Thermal Conductivity Leak
 Detectors suitable for Testing
 Equipment by Overpressure of
 Vacuum.

Detector depending on the difference in thermoconductivity between various test gases and air have been developed. Leakage is detected by electrical means and the apparatus is simple and easy to maintain. (12 references.)

- | | |
|--|--|
| 204-60-900 | 10029 |
| Steckelmacher, W. | Instrument Practice (1960) |
| | The Measurement of Pressure in Industrial High Vacuum Systems. |
| 841-63-151
100 | 10030 |
| Tasman, H.A.
Boerboom, A.J.H.
Kistemaker, J. | Vacuum v13 n2 (1963) |
| | Vacuum Techniques in Conjunction with Mass Spectrometry |
| 604-64-141 | 10031 |
| VanNie, A.G.
Zaalbergvanzeist | Philips Technical Review v25 n4 (1963-1964) |
| | A Vibrating Capacitor Driven by a High Frequency Electric Field. |
| 997-64-210 | 10032 |
| Stutzman, G.R. | U.S. 3,126,734 (64) |
| | High Speed High Sensitivity Leak Detectors |

Radioactive gas detection system for tanks and other closed pressurized systems.

997-65-412 10033
 Long, F. V. U.S. 3,170,152 (65)
 Pipeline Leak Detection Device

Two transducer systems of "hearing" leaks producing their own sound.

997-50-100 10034
 151
 Jacobs, R. B. U. S. 2,504,530 (50)
 Vacuum Leak Detection Method

Mass spectrometer vacuum using probe gas.

997-49-151 10035
 Davidson, J. U. S. 2,706,398 (49)
 Leak Detection

Mass spectrometer probe for leak detection.

997-60-130 10036
 140
 Palmer, J. F. U. S. 2,947,166 (60)
 Turner, R. B. Vapour Leak Detection

Detect moist air with sensitive element in the form of a hygroscopic salt between two electrodes to which a potential is applied. Current breakdown is proportional to the moisture content of the air passing the probe.

- | | |
|--------------|---|
| 997-63-130 | 10037 |
| Briggs, W.E. | U.S. 3,085,198 (63) |
| | Ionization Gauge Type used on
Vacuum Systems |
| 997-64-111 | 10038 |
| Minter, C.C. | U.S. 3,159,998 (64) |
| | Vacuum Gauge and Leak Detector |

A circuit containing a current-heated temperature-sensitive wire mounted as close as possible to but not actually in the stream of flowing air.

- | | |
|-------------------|---|
| 378-66-140 | 10039 |
| Allenden, D. | J. Sci. Instrum. 38 (12) Dec.,
1966 512-513 |
| | Diode Leakage Detector Control
Unit. |
| 998-61-100
900 | 10040 |
| King, J.G. | Transactions of the 8th. National
Vacuum Symposium and the 2nd
International Congress on Vacuum
Science and Technology 1961. |
| | Production Leak Testing of Large
Pressure Vessels. |

Used on vessels from 1/4 to 30 cubic feet in the leakage range of 1×10^{-2} to 1×10^{-8} atmosphere cubic centimeters per second.

162-44-143

10041

Electronic Industries 3 (3) Mar.,
1944 108 - 109

P E Tube Detection

Illustrations and operating details of an ultra-violet photometer designed to analyze for dangerous concentrations of harmful gases, device consists of mercury and ultra-violet generator and quart condensing lenses and prisms. Two large gas tubes connect to the analyzing chamber which contains balanced phototubes and amplifiers.

204-61-100
900

10042

Hutchens, W. C.

Factory 119 (2) Dec, 1961 81-87

Leak Detection

General techniques are discussed.

980-62-900

10043

Muller, K.

Arch. Tech. Messen (316) May,
1962 R45-R 49

Leak Detectors and their Application

Tables to assist in selection of proper leak detector for the job.

617-61-130

10044

Mitchell, E.
Johnson, V. I.

Product Engineering 65 (9)
Sept., 1961 54-55

Positive Condenser Leak Detection

A method using high differential pressure and freon gas to detect leaks in steam condensers.

842-63-131

10045

Meurer, E.

Vacuum Tech. 12 (2) Nov., 1963
41-44Improving the Halogen Leak-
Detection Procedure

The sensitivity and life of the measurement cell can be improved by introducing oxygen of air into the system to produce an oxidizing atmosphere.

980-62-540

10046

Bewiloga, L.
Lippold, H.Aper. Tech. der Phys. 10 (5)
1962 373-374A Simple Instrument for Testing
liquid-gas Containers

Excessive pressure of escaping gas through the coupling tube is measured with a spring pressure-gauge. (in German)

328-62-144

10047

Instrum. and Control Systems 35 (5)
1962 119-122

Capacitive Pressure Sensors

Capacitance-type pressure pickups generally employ a metal diaphragm separating two volumes. Stationary metal plates are positioned on each side of the diaphragm. A pressure difference will force the diaphragm toward one or the other of the stationary metal plates. This deflection of the diaphragm changes the capacitive coupling between it and the two plates; coupling with one plate increases while that with the other plate decreases. An a. c. signal across the plates can be used to sense the changes in capacitance.

043-59-153

10048

Dalton, J. L.

Brit. 811,744 1959 Esso Res.
and Engr.

Chromatographic Analysis of Bases

The procedure of Brit. 762,008 is improved by using as the first mixture to be adsorbed, a fraction obtained from the chromatographic sep. of a mixture of the gases being analyzed.

006-49-140

10049

McGehee, C.

Ann, N.Y. Acad. Sci. 72 1959
714-719

The discharge in helium may be excited at atm. pressure, is relatively insensitive to ambient pressure and flow changes, and is only slightly more sensitive to temperature changes when used with a standard d. c. recorder.

329-64-110

10050

Aleksandrovich, E. G. V.
Sokovishin, V. A.
Sazanov, A. I.Instrum. And Exp. Tech.
5 pp. 939-942 Sept. - Oct., 1964Portable General-Purpose
Katharometer Leak Detector

Leak detector, based in comparison of thermal conductivity of gas leaking from vessel being inspected with that of air, is intended for pressure systems containing gas with thermal conductivity different from that of air. Leakage of Hydrogen of about 250 cm³/yr can be detected.

997 - 110

10051

U. S. Gov't. Contract
AD - 606 404 Div. 30
Naval Research Lab
Washington, D. C.

Temperature comparison and improvement of the NRL thermister bridge gas leak detector.

337-62-412

10052

Industrial Design 9 (3) Mar.,
1962 94 Delcon Corp. Palo
Alto, California

Sonic Leak Detector

Detects leaks in pressure and malfunctioning friction joints by listening to production equipment. Translates inaudible sound at frequencies from 35 ke to 45 ke into the audible frequency range. It does not pick up ambient audible noise of any kind so can operate in the deafening din of a tool shop or near a whining jet engine. It works on flashlight or mercury cell batteries.

980-62-900

10053

Lange, F.
Lippold, H.

Exper. Tech. der Phys. 10 (5)
1962 375-379

A Simple Absorption Leak Detector

Uses absorption of silicon gelatin at liquid nitrogen temperatures.

529-62-144
150

10054

Brody, J. D.

Non-destructive Testing 20 (5)
Sept.-Oct. 1962 332-334

Mobile Infrared Gas Analyzer for Gas transmission Line Leakage Surveys

486-62-421

10055

Anonymous

Mach. Design 34 (30) Dec. 20,
1962 14

A device developed by the Institute of Gas Technology U. S. A. injects a constant time sound signal into a pipe. The sound issues from the leak at the same time as the escaping gas.

980-64-410

10056

Mindner, D.
Wolf, G.Tech. Dig. Proba. 6 (1) Jan.,
1964 36-39

Pipe Leak Detector RS - 1

The noise due to leaking water pipes is detected. The instrument was developed at the Vibration Engineering and Acoustics Works in Dresden.

044-64-531

10057

Ratcliffe, R. T.

Brit. J. Appl. Physics. V15 No.1
79-83 '64

Location, Measurement, and
Assessment of Shape of Leak
by bubble emission

Method of estimating leak size by measuring pressure drag after bubbles are emitted for a given time. This takes into account nonuniformity of leak cross section, method can also be used to obtain indication of shape of leak. Apparatus using these principles can be readily adapted for remote operation behind shielding as is thus suitable for investigations in radioactive or other dangerous materials.

998-63-900

10058

Lovelock, R. T.

Proc. Symp. Soc. Envirom. Engrs.
(1) 1963 47-50

Measurement of Gas Leakage in
Sealed Components.

378-64-900

10059

Pacey, D. J.

J. Sci. Inst. Jan., 1964

A Self-extracting Search Gas Probe
for the Location of Leaks in
Vacuum Apparatus

This device avoids contamination of the test area by search gas.

841-64-531

10060

Biram, J.
Burrows, G.Vacuum 14 (6) June, 1964
221-226

Bubble Tests for Gas Tightness

Leak rates of 10^{-7} torr per second under vacuum can be detected with capillaries of 1 mm bore. The liquid should have low surface tension and the gas low velocity and molecular weight.

845-63-150

10061

Chatel, J. L.
Moreau, J.Vide 18 pp 580-584 Nov.-Dec.,
1963The MC 10 Leak Detector Cell:
Helitest 2002

After the necessity of equipping leak detectors with low - pressure analytical cells is shown, the MC 10 analysis cell is described. Its principal characteristics such as sturdiness, simplicity, and interchangeability of constituent elements are then discussed. The performance, together with an indication of the principal control parameters, is given. (in French)

162-62-413

10062

Electronic Ind. 21:116 Sept., 1962

Air Leaks Detected by Ultrasonics

338-63-900

10063

Ind. Electronics 1:25 Feb., 1963

Leak Detectors

882-53-412

10064

Water and Water Eng. 57 (687)
May, 1953 197-199

Demonstration of Aqua-Visual
Apparatus for Leak Detection
at Sheffield

Leak detector consisting of microphones and amplifiers, records sounds produced by underground leaks; tests and results are described.

561-62-412

10065

Oil and Gas J. 60:71 Dec., 1962

Sonic Device Pinpoints Gas
Leaks from Surface

487-63-412

10066

Mech. Eng. 85 : 59 April, 1963

Sonic Leak Finder for Escaping Gas.

282-63-412

10067

Heating - Piping 35:42-43
April 1963

Sonic Device - Detects Pipeline
Leaks

612-64-413

10068

Evans, J. T.

Power 108:162 May, 1964

Today We Quickly Find Tiny Leaks
Using an Eltrasonic Device

- 164-64-413 10069
Elect. World 162:106 May, 1964
Ultrasonic Detector is Versatile
Testing Tool
- 283-64-413 10070
Hydraulics and Pneumatics
17:240 1964
Ultrasonic Instrument Locates
Leaky Valves
- 727-64-413 10071
Safety Maint, 128:55-56 Aug.,
1964
Ultrasonic Method Detects Leaks
in Pressure and Vacuum Systems
- 887-65-413 10072
Welding Eng. 50:72 Feb., 1965
Ultrasonic Detector Cuts Costs 15%
- 486-65-113 10073
Machine Design 37:42 April, 1965
Thermocouples Trigger Damp-Gas
Detector

- 684-65-100
151
Reynolds, F.L. 10074
R. Sci. Inst. 36:1260-1261
Aug. 1965
Helium Leak Detector Probe
- 883-65-413 10075
Welding J. 44:111 112 Feb., 1965
Portable Ultrasonic Device
Used to Check Air Conditioning and
Air Pressure Systems
- 164-65-900 10076
Elect. World 164:128
Aug., 1965
Translator - detector Locates Piping
Leaks
- 283-65-413 10077
Hydraulics and Pneumatics 18:113-
114 Nov., 1965
Ultrasonics Finds Leaks Rapidly
- 980-59-900 10078
Wilson, G.
Leaks in Gas Distribution
Systems 1959 67 pages

Detection, repair, and prevention of leaks.

997- -900

10079

U. S. Gov' t. Contract
AEDC-TDR-64-237, N6S-10469
01-1S

Leak Detection Systems for
Aerospace Systems Enviromental
Chamber

997- -623
624

10080

U. S. Gov' t. Contract
AD-610 590 Div. 4

Investigation of Gas Ionization
Phenomena at Optical and IR
Frequencies

997- -620

10081

U. S. Gov' t. Contract
AD-610 565 Div. 4

Investigation of the Mechanism
Associated with Gas Breakdown
under Intense Optical Illumination

980-39-900

10082

Z. ges. Schiess-u Sprengstoff
34, 1939 pp. 337-339

Alarm Devices for Indicating the
Presence of Dangerous Gases and
Vapors

384-22-100
900

10083

Williams, G. A.
Ferguson, J. B.

J. of Am. Chem. Soc. 44 (10)
Oct., 1922

The Diffusion of Hydrogen and
Helium through Silica Glass
and Other Glasses

A study of the permeability of silica glass and other glasses to hydrogen and helium and a function of pressure and temperature.

998-63-630

10084

Crabtree, J. M.
Glueckauf, E.

Faraday Soc. 59 pp 2639-2654
Nov., 1963
Atomic Energy Research Establishment
Harwell, Berks, Eng.

Structural Analysis of Ion Semi-
permeable Membranes by Co-ion
Uptake and Diffusion Studies

Three different strongly basic exchangers with similar co-ion uptake behavior have very different co-ion diffusion characteristics. Not only are there differences in the order of magnitude of the co-ion permeabilities P at the same external electrolyte concentration c , but also marked differences in the shape of the P - c curves, which can be interpreted in terms of the internal micro-structure of the (inhomogeneous) membrane. Other structural details can be elucidated from the fact that polyvalent co-ions are more strongly excluded from local regions of high counter-ion concentration than are monovalent co-ions, and so leak to different P - c relations. Furthermore there was observed, especially for polyvalent ions, a great difference between the mechanisms of steady-state through-diffusion and of transient diffusion out of a saturated membrane, which also permits interpretation in terms of internal structure. The details obtained by the mathematical analysis of the diffusion data agree with, and go considerably beyond, what can be ascertained by electron microscopy.

997-63-510

10085

U.S. Gov't Contract AT(11-1)-292
Test Evaluation, 1963 13 pgs.

Reactor Plant Containers

A procedure for testing the integrity of reactor plant containers for leak tightness is described, along with the equipment used. The container leak rate was determined from an inclined differential water manometer.

801-63-200

10086

Smith, Richard R.
Echo, Maurice, W.
Doe, Charles B.

U.S. Pat. 3,116,211 Dec., 1963

Methods and Apparatus for
Examining Fuel elements for
Leakage.

A process and a device for the continuous monitoring of fuel elements while in use in a liquid-metal-cooled, argon-blanketed nuclear reactor are presented. A fraction of the argon gas is withdrawn, contacted with a negative electrical charge for attraction of any alkali metal formed from argon by neutron reaction, and recycled into the reactor. The electrical charge is introduced into water, and the water is examined for radioactive alkali metals.

801-63-630

10087

Moutaud, Gilberte
Parisot, Jean

U.S. Pat. 3,102,826 Sept., 1963
Commissariat a l'Energie
Atomique

Porous Membranes and Methods of
Manufacturing these Membranes

In the method described for manufacturing porous membranes, polytetrafluoroethylene powder is thoroughly mixed with absolute alcohol in excess to form a paste. The excess alcohol is removed until only about 40% by weight remains. The paste is then applied on a metal wire fabric and the wetting substance is eliminated by drying to a suitable temperature. The paste may be treated to remove impurities before it is applied to the metal wire fabric.

106-57-144

10088

150

Chem. and Eng. News 35, 70,
and 75 (11) 1957

Infrared Analyzer

043-63-630

10089

Vupillat, Michelle
Depaule, Serge

Brit. 938,127 Oct., 1963
Commissariat a l' Energie Atomique

Appliance for Separating Fluids
by Diffusion

The appliance described consists of a fluid-tight casing containing a plurality of diffusion elements in the form of sleeves of elongated cross section arranged in side-by-side parallel relation with their larger sides facing one another and separated by spacing means. A primary inlet, an outlet for fluid being treated, and a secondary outlet for the fraction of fluid diffused are provided. The opposite open ends of the interior of the sleeves communicate with the primary inlet and outlet, and the spaces between the facing walls of the sleeves communicate with the secondary outlet. The sleeves may be made entirely of a porous material or they may be made of metal with perforations covered by a porous material. Preferably the diffusion elements are so shaped that projecting parts of the diffusion walls themselves provide the spacing means. Struts may be placed between the diffusing walls to prevent possible deformation. The appliance is suitable for separating isotopes of uranium.

401-60-200

10090

Kerntechnik 2 p 148 1960

Apparatus for the Automatic
Warning of Supernormal Radioactive
Radiation (In German)

401-60-220

10091

Kerntechnik 2 p 201-202 1960

An Apparatus for the Continuous
Monitoring of Beta and Gamma
Activity of the Air and Off-
gases at various Sites

052-63-144
150

10092

Anonymous

Brit. Chem. Eng. 8 1963 638

Leak Detectors and Heat Exchangers

Permanently installed infrared gas analyzers give the first indications of a leak by detecting the presence of water vapor in the CO₂ coolant passing through the heat exchange. The header tanks of the heat exchanger are then checked for CO₂.

842- -151

10093

Kirchner, F.
Benninghoven, H.

Vacuum Tech.

Double - focusing Mass Spectrometer with a Center Tube Detector as a very Sensitive Leak Detector

The arrangement of the instrument includes the ion source, a 0.2 mm slit, a 60° sector magnet field 5.5 cm in radius, a second slit, a 60° sector electrostatic field 7.4 cm in radius, a 0.2 ml slit, and an I of similar sounter tube as a detector. The smallest detectable leak is 2.2×10^{-4} cc atm/sec.

684-64-200

10094

Pleasanton, F.
Johnson, C. H.Rev. Sci. Instrum. 35 (1) 1964
97-102

Systems for Continuous Generation and fast Transfer of Radioactive Gases

248-64-150

10095

Fuhrmann, H.

Gas Instrum. and Tech. 8 (6)
1964 340-347

Measuring Traces of Gases with Automatic Analyzer

A review of automatic analyzers with nine additional references.

997- -140

10096

U. S. Gov' t. Contract
NASA - CR - 63281

Transducer to Measure Hot
Gas Leak-mass Flow Leakage

997 - -210

10097

U. S. Gov' t. Contract
EUR - 164S - F
N6S - 15072 05-12

Radioactive Isotopes for
Gaseous Leak Detectors

997- -624

10098

U. S. Gov' t. Contract
AD-605 908 Div. 9
Foreign Tech. Div.
A.F. Systems Comm.

Electrical effects accompanying
flow of gas in shock tube

Process of diffusion developing in front of thermal ionized gas can lead to large products of electric potential along the direction of the prop.

997- -620

10099

U. S. Gov' t. Contract
AD-605 498 Div. 9
Foreign Tech. Div.
A.F. Systems Comm.

Effects related to the multi-
component nature of flowing gas
mixtures

Flow part on impenetrable surface of a mixture of two extraneous gases, on two-atom gas that can dissociate into atoms.

997 - -620

10100

U. S. Gov' t. Contract
AD-426 155 Div. 8
Lear Siegler, Inc.
Santa Monica, California

Development of experimental
gas discharge display

997- -620

10101

U. S. Gov' t. Contract
AD-426 267 Div. 25
Microwave Research Inst.
Poly. Inst. of Brooklyn, N. Y.

Investigation of Gaseous Electronics

Study of various methods for the analysis of the approximately uniform plasma
in a pulsed discharge. (Basically hydrogen)

997- -623

10102

U. S. Gov' t. Contract
AD-424 845 Div. 25
RCA Victor Co., Ltd.
Montreal, Quebec

Generation of Negative Ions
in a Gas Discharge

997- -620

10103

U. S. Gov' t. Contract
AD-423 900 Div. 25
Microwave Assoc., Inc.
Burlington, Mass.

Investigations of high power Gaseous
electronics

Clean-up and thermal recovery of inert gases at the interface between a high
power microwave discharge and a quartz interface.

997- -510

10104

U.S. Gov't Contract
AD-420 004 Div 30
IIT Research Inst.
Chicago, Ill.

Research and development of an
automatic Barone monitor

Sensitivity 10 pts/billion

997- -620

10105

U.S. Gov't Contract
AD-863 Div. 25
Elect. Eng. Research Lab
L. of Texas
Austin, Texas

General Disp. relations for a
partially ionized gas

Computer analysis from Boltzmann's equation using the Perturbation theory.

997- -900

10106

U.S. Gov't Contract
AD-421 498 Div. 20

The Plasma Test particle problem

997- -900

10107

U.S. Gov't Contract
AD-601 133 Div 25
Engineering Center
U. of S. Calif.
Los Angeles, Calif.

Numerical analysis of some nonlinear
problems in partially ionized gases

The theoretical study of harmonics arising from nonlinear dissipation is the
principle subject.

997- -620

10108

U.S. Gov't Contract
AD-600 798 Div 25
Electro-Optical Systems, Inc.
Pasadena, Calif.

Basic research on gas flows
through electric arcs

Extensive measurement in a "cascade" type arc research apparatus. (hydrogen, helium, ammonia, argon)

997- -620

10109

U.S. Gov't Contract
AD-601 048 Div 25

Heating and Ionization of a Gas
Stream by repetitive, suppressed
breakdown discharges

997- -620

10110

U.S. Gov't Contract
AD-600 542 Div 25
Electron Physics Lab
U. of Michigan
Ann Arbor, Mich.

Enhancement of Plasma -
Density of an arc discharge

Detailed study of plasma density in electron beams.

997- -114

10111

U.S. Gov't Contract
AD-610 620 Div 3

Design and Construction of
SPIRIT (short path infra-red
instrumentation)

521-62-110

10112

Nat'l. Engineer, May 1962 26
General Electric. Co.

Audible Alarm Leak Detectors

Bulletin GEA - 7576 illustrates positive halogen leak detection and operation, design, and specification.

043-47-900

10113

British 586, 530 Mar., 1947
Standard Development Co.

Apparatus Responsive to the Pressure
of Noncondensable Gases in Vapor

The device is actuated by the pressure differential existing between 2 closed containers, one containing a volatile liquid and its vapor not subject to contamination, and the second containing the identical liquid and vapor which is liable to contamination by noncondensable gases. Such apparatus may be used to control automatic vents which remove air from NH₃ refrigeration systems.

328-62-120

10114

Instruments and Control Systems
35 (7) 1962 208 General Monitors,
Inc., California

Combustable Gas Detector

New model H200 combustable gas detector permits continuous monitoring of hazardous conditions in any area where hydrogen and other explosive or flammable hydrocarbons are manufactured, stored, or handled. Sensitivity range is adjustable from 5 % of minimum explosion limit. It uses a catalytic reactor in an AC driven balanced bridge which drives relay circuit through a solid state AC amplifier.

980-59-153

10115

Belg. 577, 359 1959 Centre national
de recherches metallurgiques

Gas Chromatography

The sensitivity of the measurements is improved by keeping the detection cell unit at 10° in a thermostat.

339-44-900

10116

Industr. Equipm. News 12 1944 98

Indicating and Recording Electronic
High-vacuum Gauge

163-62-140
900

10117

Electronic Tech. 39 (5) 1962
202 J. Langaham Thompson Ltd.

Vacuum Transducers and Indicators

Models of the Hastings-Raydist range of pressure transducers and indicators for the measurement of degree of vacuum are now available in the UNK. through J. Langaham Thompson. Three self-contained mains-operated models cover the ranges 0 to 0.1 mm Hg, 0 to 1 mm Hg, and 0 to 20 mm Hg. The transducers are rugged devices using the Hastings thermopile element which is self-compensating for changes of temperature. Either nickel-plated or Pyrex glass transducers are available. The indicators are mirror-scale 4 in. instruments with the complete transistorized power unit and measuring circuitry built into the meter case. The unit plugs straight into the 230V, 50 c/s mains and no further auxiliary units are necessary.

125-54-143

10118

Diesel Power 32 (10) Oct., 1954
48-49

Detecting Fuel Leaks with "Black
Light"

Use of Magnaflux ZB Black Light on Southern Railway for finding locomotive fuel line leaks; device is high voltage mercury arc fluorescence of diesel fuel oil as contrasted with that of lubricating oil.

980-39-900

10119

Kibler, A. L.

Z. ges. Schiess - u. Sprengstoffw
34 337-339 1939 Chemische-
technische Reichsanstalt

Alarm Devices for indicating the
Presence of dangerous Gases and
Vapors

Upper and lower explosive limits are given for 18 combustible gases in mixtures with air, and dangerous and lethal concentrations are given for 16 poisonous gases. The requirements which a satisfactory alarm device should meet are listed. All com. devices tested by the Chemisch-technisch Reichsanstalt and by certain other testing stations are named and the principle upon which they operate are described briefly.

997- -510

10120

U. S. Gov't. Contract
AD-601 510 Div. 30
Foreign Tech. Div.
A. F. Systems Comm.
Wright Patterson A.F.B., Ohio

Simple Capillary Compression
Manometers for Measuring Low
Absolute Pressures

340-64-151

10121

Iron Age V 193 (5) 98-99, 1964
Foxboro Co.
Foxboro, Mass.

Mass Spectrometer checks welds

Verifies difficult welding operations with mass spectrometer leak detector.

043- -153

10122

Brit. 811,744 Esso Res. and Eng.
Co.

Chromatographic Analysis of Gases

980-58-630

10123

Duriau, Y.
Gadal, M.
Pecqueur, P.

Belg. 567,135 Oct., 1958
12 pgs.

Improved Devices for separating
Gases by Diffusion

A device is described for the separation of gases of slightly differing molecular weights by diffusion through at least one porous wall. The device is characterized by the fact that it comprises at least one bundle of porous diffusion tubes mounted on a support in parallel, and connecting an inlet collector of gas to be diffused with an outlet collector for the evacuation of residual gas. The tube bundle is located in a jacket delimiting a space insulated in a tight manner from the inlet and outlet collectors, the space serving for the collection of the fraction of gas diffused through the porous tubes.

609-41-900

10124

Olsen, L.O.

Phys. Rev. V 60 739-742, 1941

Quenching and Depolarization of
Mercury Resonance Radiation by
the Rare Gases

The quenching and depolarization of Hg 2 537A resonance radiation by He, Ne, Ar, and Kr was studied experimentally, the results being displayed in graphical and tabular form. Probable numbers of quenching and depolarizing collisions and cross-sections for quenching and depolarization are calculated and presented in a table.

612-58-900

10125

Power V 102 130, 1958
Winton Products Co., Inc.
Charlotte, North Carolina

997- -620

10126

Grass, Eugene P.,

U. S. Gov' t. Contract
AD-419 958 Div. 25
Brandeis University
Walthorn, Mass.

Theoretical Studies of Ionized
Gases

284-65-900

10127

Flyger, H.

Health Phys. VII 223-224, 1965

A Helium Leak Detector method
for Respirator Control

An accurate and fast method for estimating unpredictable leakage from desk work.

980-63-200

10128

Bjorkman, S. N. F.

Swed. 182,540 Pat. 1963

Indication of Leakage from a
Pipeline with Radioactive
Isotopes

The medium in the pipeline is made radioactive and the position of the leak is detected by passing a detector through a channel adjacent to the pipeline.

997- -700

10129

U. S. Gov' t. Contract
AD-601 999 Div. 6
Raytheon Co.
Portsmouth, R. I.

Signal-to-noise ratio adoptive
detection of statistical
signals in noise

The form and properties were studied of a detector designed to operate in an environment in which little statistical information about either the signal or noise field is or can be available. An adoptive detector based on the theory of nonparametric statistics has been designed. This uses signals taken from two distinct receivers, only one of which may contain signals from a target.

341-60-200

10130

Cassem, B.
Burnhum, D.

Intern. J. of Appl. Radiation
and Isotopes V9 54-9 1960

A method of leak testing
hermetically sealed components
utilizing radioactive gas

A leak rate formula based on Poiseuille's law is developed and the limitations in measurement imposed by the range of validity of this formula and of the random nature of radioactive decay is discussed. Means for differentiating between gas (^{85}Kr) which has leaked inside the part being tested and gas absorbed on its surface is presented. A logarithmic rate unit called the leak index is proposed. A brief description of industrial equipment for accomplishing leak rate measurements along with precautions to assure full safety in its operation.

622-65-133

10131

Lewis, R.D.

Pipeline Ind. V. 22 No. 2 37-40 '65

Flame ionization technique speeds
leak surveys

Flame ionizations techniques of gas leak detection has greater sensitivity than IR detectors 0.1 ppm of methane in air and can cover up to 10 MPH; other advantages of flame ionization are stability, speed of response, smaller sample volume required, no need for signal amplification, shock and vibration proof, and convenient multi-range sensitivity switching.

998-63-200

10132

Taylor, R.F.
Wall, G.P.

Progress in Nuclear Energy Sec 4
Tech. Eng. and Safety V5 307-46 '63

Development of production process
for radio-krypton recovery by
fractional absorption

980-60-151
900

10133

Olfe, D.B.

Guggenheim Jet Propulsion Center
California 1960

Mean Beam Length Calculations
for Radiation from Non-Transparent
Gases

Mean beam lengths are studied for the following types of band structure: (1) bands composed of non-overlapping collision-broadened lines, (2) bands which may be approximated by constant average absorption coefficients acting over effective band widths, (3) bands composed of randomly distributed, collision-broadened lines. The temperature and pressure dependence of the mean beam length is studied for these types of band structures and expressions for the mean beam length are obtained for some simple enclosure geometrics.

443-50-613
900

10134

Ortel
Azan

Le Vide 5 914-915 1950

Variable Leaks

Metallic variable leaks are described regulating flows of 0.5-36,000 cc/hr at atmospheric pressure. Pressures of the order of 10^{-5} mm Hg are regulated within 1-5% dependent on the type of construction.

329-63-900

Aleksandrovich, E. G.
Sokovishin, V. A.
Sazanov, A. I.

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Instruments and Experimental
Techniques No. 5 939-42 , 1963

Portable General-Purpose Katharo-
meter Leak Detector

684-39-110

Amdur, I.
Pearlman, H.

10136

Rev. Sci. Instr. Vol. 10 p 174
1939

A New Type Vacuum Thermoelement

684-58-900

Ames, I.
Christenson, R. L.
Teale, J.

10137

Rev. Sci. Instrum. Vol. 29 P 736
1958

526-58-200

Anonymous

10138

Nucleonics Vol. 16 86-91, 1958

Applied Radiation

999- -413

Anger Associates

10139

Ultrasonic Translator for Detection
and Location of Air and Gas Leaks
Model 114, 13126 Michigan Ave.
Dearborn, Michigan

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980-55-200

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J. Sci. Res. Inst. Tokyo, Vol 49
144 (1955)

Effects of Various Geometrical
Factors on Gamma-Ray Counting

980-53-900

Aikman, O.S.

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Ind. Labs. Vol 4, No. 10 114-7 (1953)

Protective Coatings Minimize Effects
of Corrosion on Selenium Rectifiers

093-57-150

Aldebert, Franz

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Chemiker Ztg. Vol 81,596 (1957)

Device for Qualitative Gas Analysis

684-53-130

Alers, G.A.
Jacobs, J.A.
Malmberg, P.R.

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Rev. Sci. Instr. Vol 24, 399-400
(1953)

Increased Sensitivity of Leak
Detection with Hydrogen

980-55-900

Allen, F.G.

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Some Notes on High Vacuum
Apparatus and techniques, AD
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Dec. 1955

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Particle Counter |
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| Anonymous | Nucleonics 82, July 1956 |
| | Bell Labs Tests with Cs 134 |
| 980- -900 | 10152 |
| Anonymous | A Dry Vacuum Test for Air Leaks
in CAD Cartridge, AD 51200 |
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| Anonymous | Iron Age, Vol. 163, 60-1, 1949 |
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| Anonymous | Aviation Week, November 1956 |
| | New Leak Detector Developed
by Boeing |
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Radiation Effects in Stride |

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Radioisotopes |
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Nucleonics, Vol 12 No. 3 (1954)

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Symposium for 1954

526-56-700

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Anon.

Nucleonics, Vol 14 No. 4 (1956)

Report on Scintillation Counting
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980-60-200

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Atomic Energy, Chapter 1, Atomic
Energy Commission, Part 20,
Standards for Protection Against
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221 (1957) Russell Mfg. Co.
South Farms, Middletown, Conn.

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Semiconductor Devices

526-56-200

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Anonymous

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87, 1956109 min A⁴¹ seeks Gas Storage Leak

801- -100

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Halide Detector, U. S. Patent
2,779,666

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Gas Chromatography

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Metal-to-Metal Contact

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P-N Junctions by Impurity Intro-
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609-45-700

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Distribution of Mass Transported
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Germanium Crystal by the Forming
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609-52-151

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164, 1952

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526-59-700

10173

Babcock
Davis
Ruby
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116-22, 1959

Coated Semiconductor is Tiny
Neutron Detector

609-47-700

10174

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Surface States and Rectification
at a Metal-Semiconductor Contact

997-52-100

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III, 1952

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Leak Detection Using Krypton-85

721-58-200

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Dosimetry of Radioisotopes

609-38-151

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A New Mass Spectrometer with
Improved Focusing Properties

980-51-151

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Application of the Mass Spectro-
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An Ionization Gauge Circuit

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A Mass-Spectrometer Probe
Method for the Study of
Gas Discharges

801 - 39 -800

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Gas-Leak Detector for
Refrigerating Apparatus, Fr.
Patent 848,087 (1939)

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Design of Grid Ionization
Chambers

526-49-200

Burt, B.

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Absolute Beta Counting

998-55-144

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Infrared Properties of Selenium

526-56-200

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A New Pulse-Amplitude
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Particle and Quantum Counters.

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Leak Detection by Vacuum
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528-43-200

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New Portable Bench-Type Mass
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Germanium Diodes Sealed in Glass

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Battery, J. F. | J. Appl. Phys. 99-102 (1954)

Some Problems in the Diffusion
of Minority Carriers in Semi-
conductor |
| 980-06-900 | 10378 |
| Voege, W. | Physik. Zeitschr. Vol. 7 498-500
(1906)

A New Vacuum Meter |
| 004-58-130 | 10379 |
| Walisch, W.
Ashworth, M. R. F. | Anal. Chim. Acta. Vol. 18
632-7 (1958)

Polarovoltic Method for Halide
Ion Determination in Dilute
Solutions |
| 980-07-900 | 10380 |
| Warburg, E.
Leithauser, G.
Johansen, E. | Ann. d. Physik Vol. 24 26-42
(1907)

The Vacuum Bolometer |
| 684-34-100 | 10381 |
| Webster, D. L. | Rev. Sci. Instr. Vol. 5 42 (1934)

Vacuum Leak-Hunting with
Carbon Dioxide |

684-52-133

10382

Weinreich, O. A.
Blecher, H.

Rev. Sci. Instr. Vol. 23 56 (1952)

Ionization Gauge with Thoria-
Coated Cathode

605-50-200

10383

West, D.
Rothwell, P.

Phil. Mag. Series VII Vol. 41
873 (1950)

Proportional Counters in a
Magnetic Field

378-50-200

10384

Whiting, F. B.
Thomas, D. G. A.
Marsh, J. B.

J. Sci. Instr. Vol. 27 157-60
(1950)

A Portable Battery-Operated
Radiation Monitor

684-55-151

10385

Wiley, W. C.
McLaren, I. H.

Rev. Sci. Instr. Vol. 26, No. 12
1150-7 (1955)

Time-of-Flight Mass Spectrometer
with Improved Resolution

684-61-700

10386

Wiley, W. C.
Goodrich, G. W.

Rev. Sci. Instr. Vol. 32, No. 7
846-9 (1961)

Resistance Strip Magnetic
Electron Multiplier

364-52-151

10387

Zemany, P. D.

J. Appl. Phys. Vol. 23 924 (1952)

Free Molecular Flow in Sample
Inlet to Mass Spectrometer

801-62-900

10388

Zebroski, E. L.

U. S. Patent 3,070,532
Dec., 1962 (filed May, 1958)Nuclear Fuel Element Leak
Detector

Radioactivity of the effluent coolant from each fuel assembly or channel in a reactor is monitored for the purpose of detecting and identifying leaking fuel elements.

997-62-132

10389

Stine, P. T.
Koncen, R. E.U. S. Government Contract
AD-277410 May, 1962Operational Performance of the
NRL Thermistor-Bridge Gas Leak
Detector and its Sensitivity to
Various Gases

The thermistor-bridge gas leak detector is a portable electronic device useful for location of leaks from pressurized systems containing any gas having a thermal conductivity different from that of the ambient air.

801-63-150

10390

Guenther, K. G.

U. S. Patent 3,075,076
Jan., 1963 (filed Dec., 1958)

Gas-Analyzing Method and Apparatus

A method for performing gas analysis in vacuum with the aid of a mass spectrometer is described.

801-63-410

10391

Wilson, W. A.
John, G. S.

U. S. Patent 3,084,259
Apr., 1963 (filed Aug., 1959)

Method of Determining Pipeline
Leakage

A radioactive tracer method is described for testing for leaks in a section of conduit.

043-63-630

10392

British Patent 918,187
Feb., 1963

Improvements in or Relating to
the Production of Membrane
Filters

A process is given for the production of membrane filters with improved mechanical properties and chemical inertness.

980-61-153

10393

Rath, G.

Instruments and Measurements
Academic Press Publishers,
pp. 498-506 (1961)

Production Gas Chromatography

A production gas chromatograph, which maintained the efficiency of the columns by parallel hookup, was designed.

980-61-151

10394

von Ubisch, H.

Instruments and Measurements
Academic Press Publishers,
p. 245-252 (1961)

Mass Spectrometry

The operation and components of mass spectrometers are summarized. Types of ion sources are briefly discussed.

801-62-900

10395

Jacobs, I. M.

U. S. Patent 3,069,339
Dec., 1962 (filed Aug., 1958)

Nuclear Fuel Element Leak Detector

An apparatus is described for detecting and locating leaking fuel elements in a reactor core by monitoring the radioactivity of the effluent coolant from each fuel assembly or channel.

980-62-900

10396

Brown, P. E.
Feistman, M. L.American Rocket Society
Preprint No. ARS 2640-62 1962Multi-point Leak Detection
System for Space Vehicle
Applications

A technique is described which enables continuous leak detection at many locations throughout a space vehicle with the leakage data made available in a format permitting its transmission via a telemetry link from the vehicle.

980-62-200

10397

Berry, P. F.
Cameron, J. F.United Kingdom Atomic Energy
Authority Nov., 1962
Wantage Research Lab.
Wantage, Berks, Eng.Measurement of Leaks in
Hermetically Sealed Containers
Using Kr^{85}

Two methods of using Kr^{85} for leak detection are described. A comparison is made with the helium mass spectrometer and it is concluded that the Kr^{85} method is cheaper, simpler, more reliable, and in some cases more sensitive.

997-57-900

10398

Colvin, Donald W.

U. S. Government Contract
AT(07-2)-1 Jan., 1957
DuPont de Nemours (E.I.) and Co.
Savannah River Lab.
Aiken, S. C.

A Simple Leak Detector for Tritium

An ionization chamber of the integrating type was built that could detect a tritium leak rate of 10^{-13} cc per second within a few minutes, after a gas-collecting period of 16 hr. Electronic circuitry was avoided by using a quartz fiber voltmeter to indicate the rate of discharge of the chamber.

801-62-900

10399

Scherbatskoy, Serge A.

U. S. Patent 3,060,315
Oct., 1962

Pipe-Line Exploration

Two radiometric methods are outlined for locating leaks in buried oil pipelines.

684-62-130

10400

Barrington, A. E.

Rev. Sci. Instr. 33 pp. 1045-
1046 Oct., 1962

Sensitivity of an Ion Pump
Leak Detector

An analysis of the parameters governing the performance of a sputterion pump leak detector using oxygen or helium as the probe gas is presented.

043-62-130

10401

Daly, Norman R.

Br. Pat. 907,511 Oct., 1962

Method of Detecting Ions

An ion detector for mass spectrometers as designed for suppressing unwanted background signals and for distinguishing between ions of the same mass and charge, eg., H_2^+ and D^+ ions. In the ion detector, the ion beam is passed through an aperture and allowed to strike a thin foil so that the desired ions liberate secondary electrons from the back of the foil, which are then detected.

997- -200

10402

ORNL-3578 pp 78-95
Oak Ridge National Lab., Tenn.

Process Instrumentation
Development

Tank-level telemetering from 24 intermediate level waste tanks to Building 3105 for the Operations Division was updated to provide greater accuracy with less maintenance and to utilize a minimum of leased telephone lines for the system. Instrumentation was developed to permit continuous monitoring of the Cr^{6+} concentration in a Darex dissolver effluent stream. Both ultraviolet absorption and oxidation potential techniques were investigated.

727-64-413

10403

Safety Maint. 127 (5) May,
1964, pp. 45-46

Ultrasonics Hunt Oxygen Leaks

Hospital uses of new portable 9 lb. transistorized instrument of ultrasonic system to locate leaks in central oxygen, nitrous oxide systems. The system is used by the U.S. Army Hospital's medical equipment maintenance branch and is reported to locate leaks of one-thousandths of an inch.

997-64-630

10404

U.S. Gov't Contract
N600 (19)-59530
Fifth quarterly progress report
Feb, 1964
Southern Research Institute
Birmingham, Alabama

Mechanical and Physical properties of TZM Molybdenum Alloy Sheet and of Tungsten Sheet

A program to determine preliminary design data for tungsten and TZM molybdenum sheet is reviewed.

329-64-200

10405

Afrosimov, V.V.
Kalinkevich, I.F.
Serenkov, I.T.

Instr. and Exp. Tech. 1
Jan.-Feb., 1964 pp. 150-153

Automatic Stabilization of Beam
of Fast Atomic Particles

Method for stabilizing intensity and direction of beams of fast ions of atoms in devices of mass spectrometer type is proposed; method is based on direct control of beam intensity in chamber of device for experiments with beam.

329-63-151

10406

Batrakov, B.P.
Kobzev, P.M.

Instruments and Experimental
Techniques 4 July-Aug., 1963
pp. 701-703

Omegatron for High-Vacuum

Omegatron is described, whose basic electrodes are metallic grids, to increase efficiency for evacuating regions enclosed by these electrodes; tests show that "transparent" omegatron has substantially lower noise level than other omegatrons describes in literature; omegatron described was designed for both qualitative and quantitative measurements.

980-64-110

10407

Becker, H.M.
Wagener, H.J.

Atomwirtschaft, 9 pp 224-28
Jan., 1964

Theory and Practice of determining Leak Rates of Reactor Safety Containments

To determine the leak rate of a large container it is necessary to measure the total pressure in the container, the mean absolute air temperature, and the mean specific water vapor content. A measuring process was developed in which a metal wire is used as a resistance thermometer. Because of the difficulties accompanying the use of a free, stretched wire in a large container, a hemp rope was used as a carrier for the wire which was laid between the strands. The theory and practice of this process are described in terms of the practical example provided by the tightness testing of the reactor chamber of the DIDO reactor (FRJ-2) in the Julich Nuclear Research Center. (in German)

335-62-144

10408

Beebe, C.H.
Liston, M.D.

ISA Proc. Preprint 40 (2)
1962 6 pages

Development and Performance
of Monobeam, Ratio, Nondisper-
sive Infrared Analyzer

System that overcomes certain defects of existing instrumentation, employs single source and sample cell, with tandem arrangement of selective detectors.

329-63-151

10409

Belov, N.S.
Bronstein, A.M.
Ozerov, L.N.
Rafal'skii, A.E.

Instr. and Exp. Tech. 4
July-Aug., 1963 pp. 707-709

Spectrometer Having Ion Separation
According to Time of Flight

Multiplier described, that focuses electrons in perpendicularly combined electric and magnetic fields. It is used in recording system for small current pulses of high speed mass spectrometer.

980-64-133

10410

Bergsnov-Hansen, B.
Endow, N.
Pasternak, R.A.

J. Vacuum Sci. Technol. 1
pp. 7-9- Sept.-Oct., 1964

Ion Gauge Calibration at
Low Pressures Using a Leak
Detector

A simple method for ion gauge calibration is presented.

329-64-900

10411

Bilfyukevich, A. L.

Instr. and Exp. Tech.
2 pp. 185-187 Mar.-Apr., 1964

Low-temperature Resonator For
EPR Studies at 0.8 cm Wavelength

A low-temperature resonator with H_{01} type waves is described. The object to be investigated can be oriented in both horizontal and vertical planes without rotation of the magnet. To increase the precision of the measurements the instrument contains a tuning plunger resonator in direct proximity with the investigated object.

842-64-151

10412

Brunnee, C.
Delgmann, L.
Kronberger, K.

Vakuum-Technik 13 (2) Mar.,
1964, pp. 35-42

Ein Quadrupol-Hochfrequenz-
Massenspektrometer fuer
vakuumtechnische

Quadrupole-HF-mass spectrometer for vacuum technique; construction and performance of apparatus for analysis of residual gases in high and ultra high vacuum systems.

123-64-200

10413

Byakov, V. M.
Ershler, B. V.

Kokl. Akad. Nauk. SSSR
154 pp 669-672 Jan., 1964

The Mechanism of the Formation
of Molecular Products in the
Radiolysis of Water

Four basically different mechanisms were proposed in the literature for the formation of H_2 and H_2O_2 when water is irradiated with gamma rays or electrons.

997-62-100

10414

Cadwell, J. J.

U. S. Gov't. Contract
AT (45-1) - 1350 June, 1962
General Electric Company

List of Leak Detection Methods

The choice of a leak detection method depends on one or more of the following factors: (1) the maximum allowable leakage rate, (2) the complexity of the detection equipment, (3) the maximum allowable hole size, and (4) the time during which the leakage must be minimum. A method is presented of selecting from graphs the most suitable method for a given situation.

980-64-900

10415

Calori, A.
Persano, A.Riv. Ing. Nucl. 2 pp. 297-303
Jan. - Apr., 1964Evaluation of the Errors in the
Determination of the Leakage
Rate During Leak Testing of
Reactor Containers

The evaluation of the errors made in the determination of the leak rate during leak testing of reactor containers is discussed. The limits of validity of the results of such measurements are indicated. (in Italian)

082-64-151

10416

Cambey, L. A.
Ormrod, J. H.
Barber, R. C.Can. J. Phys. 42 (1) Jan., 1964
p. 103-112Effect of Magnetic Field
Nonuniformities on Performance
of Double-Focusing Mass
Spectrometers

Two types of nonuniformities considered; in "azimuthal nonuniformity" vertical component of magnetic field varies along central ion path; in "radial nonuniformities" magnetic field is function of displacement on direction normal to central ion path.

378-64-151

10417

Cuthbert, J.
Hart, F.
Prosser, N. J. D.J. Sci. Instr. 41 (7) July
1964 pp. 131-135Improvements to M. S. 2 Type
Mass Spectrometer

Modifications to ion source, pumping system, amplifiers and electron beam control unit of M. S. 2 mass spectrometer are discussed, that have made possible reduction in its detection limit on gas analysis; unconventional operating techniques are used to detect trace impurities at ppm level in nonroutine, semi-quantitative analysis.

997-64-900

10418

Davis, E.M.

U.S. Gov't Contract
AT (45-1)-1350 Feb., 1964
General Electric Co.
Hanford Atomic Products Operation
Richland, Wash.

PRTR Secondary Activity
Detection System

The detection system uses five scintillation tubes to measure the gamma activity of water entering and leaving the four heat exchangers of the secondary coolant system, thereby detecting leaks. The system is described and operating experience is summarized. Notably, the background in the area of the detectors masks the low-level count rate expected from small leaks. Recommendations for improving detection (including detector relocation) are made.

997-64-600

10419

Davis, R.M.
Wasan, D.T.
Wilke, C.R.

U.S. Gov't Contract
W-7405-eng-48 Aug., 1960
Lawrence Radiation
California University
Berkeley, California

Measurement of the Velocity
of Gases in a Field of Variable
Composition and Temperature

An experimental study is undertaken to observe the effects of mass transfer on the fully-developed turbulent velocity field in a pipe.

980-63-200

10420

Descamps, Cl.

Industrielle Anwendung von
Radioisotopen pp 58-63 (Bern,
l'Ennergie Atomique, 1963)

Control of Mixing Processes, Wear
and Leakage by employing Radio-
active Tracers

Radioisotopes are widely used for homogeneity studies on mixing of solids or liquids. They are also helpful tools to evaluate the wear of mechanical pieces and to locate leaks. Some theoretical considerations concerning radioisotopes to be used in such problems and the way to work with them are presented. Several industrial applications of radioisotopes related to these different fields are given. (In French)

727-63-100

10421

Devrishian, C.

Safety Maintenance 125 (6)
May, 1963 pp. 34-40, 55

How to Select Combustible Gas
Detector Systems

Gas analysis methods and aims, detector systems, and properties of flammable gases and vapors are discussed.

980- -200

10422

Dwight, D. J.

United Kingdom Atomic Energy
Authority, Research Group
Radiochemical Centre
Amersham, Bucks, England

A New Method for Leak-Testing
Sealed Sources of Radium-226
and Thorium-228

A method has been developed in which radon leaking from radium-226 and thorium-228 sources under vacuum is quantitatively measured.

997-63-151

10423

Feldman, D. W.
Lange, W. J.
Rabinowitz, M.
Singleton, J. H.
Zollweg, R. J.

U. S. Gov' t. Contract
AT (30 -1) - 2823
Annual Report Jan. - Dec., 1963
Westinghouse Electric Corp.
Research Labs.
Pittsburgh, Pa.

The development of electron sources, high-sensitivity mass spectrometers, and teflon coatings for walls of ultrahigh vacuum systems is discussed, Photodesorption, thermal desorption, electron reflection by metal surfaces, and inelastic collisions of electrons at surfaces are discussed. The effects and residual gases (CO_2 , CO , and H) and the evolution of CO from tungsten surfaces on the attainment of ultrahigh vacua were studied.

378-64-151

10424

Fenner, N.C.
Ridley, R.G.J. Sci. Instr. 41 (3) Mar., 1964
pp. 157-159Mass Spectrometer Detector
Using Two Semiconductor
Magnetic Electron Multipliers

Development of detector using 2 multipliers with variable separation is described, that is capable of comparing 2 ion beams simultaneously over wide mass range.

980-64-130

10425

Fuhs, A.E.

Pyrodynamics I pp. 3-25
Jan.-Feb., 1964
Aerospace Corp.
El Segundo, CaliforniaThermal Ionization and Electro-
negative Species

Free electrons that are present in the ionized gas of the re-entry plasma sheath or the exhaust of rockets cause severe attenuation and reflection of electromagnetic waves. Conditions that are favorable or unfavorable for "soaking up" electrons were examined.

996- -200

10426

Gyorey, G.L.
Brownell, L. E.
York, M.Michigan University
Ann Arbor, MichiganLarge Scale Use of Radiotracers
for Leak Detection in the Saturn
Space Vehicle

The methods used in the leak testing of the Saturn vehicle are described. A study was made of the possible use of a radiotracer for leak detection when the vehicle is fueled or in flight. ^{85}Kr in the presence of a gamma background are discussed. It is concluded that a leak detection sensitivity of 10^{-4} cc/sec can be achieved with low gamma background conditions using a ^{85}Kr concentration of $1 \mu\text{C/cc}$. The advantages of this tracer method over other methods are described.

002-63-410

10427

Hogan, D. P.

Am. Gas J. 190 (2) Feb., 1963
pp. 30-33

Sonic Pinpointing of Leaks

Features of new sonic device developed at the Institute of Gas Technology, Chicago, Illinois that makes it possible to detect leak location without disturbing large areas of street surface; detector, which operates by introducing constant sonic signal into pipe, is based on principle that sound present in gas steam inside pipe will issue from leak along with escaping gas.

684-64-151

10428

Hunt, W. W.
Huffman, R. E.
McGee, K. E.Rev. Sci. Instruments 35 (1)
Jan., 1964 pp. 82-87Observation and Identification of
Ion Dissociation Processes
Occurring in Drift Tube of Time-of-
Flight Mass Spectrometer

Shifts in ion flight times, produced by flat top potential barrier, applied within drift tube of time-of-flight mass spectrometer, are defined analytically in terms of height of potential barrier; since these flight time shifts are selectively dependent on mass, charge, and kinetic energy of ions involved, they can be used to separate and identify products of ion dissociation processes occurring in drift tube.

684-64-151

10429

Hunt, W. W.
Huffman, R. E.
et. al.Rev. Sci. Instruments 35 (1)
Jan., 1964 pp. 88-94Time-of-Flight Mass Spectrometer
Adapted for Studying Charge Transfer,
Ion Dissociation, and Photoioniza-
tion.

Modifications in mass spectrometer are described, to permit use of drift tube as reaction/collision chamber for kinetic studies of charge transfer, ion dissociation, and similar processes, and to produce ions in source by pulsed or continuous electron or photon beams.

842-64-100

10430

Keinel, G.

Vakuum Tech. 13 (2) Mar., 1964
pp. 47-52

Lecksuche an Hochvakuum - und
Ultravakuum - Anlagen

Leak detection in high and ultra-high vacuum systems; leak detection using additive gases, as tracers; with getter ion pumps, rare gases can be used; with molecular drag pumps and diffusion pumps propane, butane, freon 12, and helium are most suitable tracer gases; sensitivity of method exceeds that of leak detectors based on mass spectrometer principle.

967-63-151

10431

Kellman, V. M.
Rodnikova, I. V.

Zhurnal Tekhmicheskoi Frziki
33 (4) Apr., 1963 pp. 387-392
see also English translation
in Soviet Physics, Tech. Physics
8 (4) Oct., 1963 pp. 289-292

Mass-spektrometry s dvumernymi
elektricheskimi i magnitnymi
polyami

Mass spectrometers with 2-dimensional electric and magnetic fields; relative position of electric and magnetic 2-dimensional fields is such that the whole system remains 2-dimensional along common direction; investigation of electron optical properties of system leads to important results for construction of mass spectrometer.

842-63-151

10432

Kirchner, F.
Benninghoven, A.

Vakuum Tech. 12 pp. 207-209
Nov., 1963

On a Double-Focusing Mass
Spectrometer with Counter-
Indicator as High-Sensitivity
Leak Detector

A mass spectrometer leak detector is described, which has a higher detection sensitivity for small test gas amounts and a shorter call time for the ion measurement apparatus than other apparatuses. The method of operation and results of using this leak detector are discussed. (In German)

997-64-112

10433

Klingman, C.L.
Meeks, J.C.U.S. Bur. Mines - Report
Investigations 6353, 1064
17 pages.Apparatus for Detecting Helium
Leaks

Instrument that is sensitive, portable, safe, and low in cost was developed by the Bureau of Mines for detecting helium leaks by comparing thermal conductivity and flow rate of atmosphere around suspected leak against reference of helium-free air; vacuum pump pulls sample gas through restriction and over thermistor elements, combined electrical output of which is affected by thermal conductivity and flow rate of sample gas; instrument could also detect, but not identify, other gases such as methanem, hydrogen, argon, carbon dioxide and freon 12.

999-62-151

10434

Kraeutle, K.

Thesis, Munich, Universitat,
1962, 149 pages (\$2.00)A Mass Spectrometer for the
Determination of Relative
Abundances of Li Isotopes in
Solids

Methods for the measurements of the natural isotopic abundance of Li are discussed. The construction of a mass spectrometer for the analysis of solids is described. The influence of mass dependence of the evaporation, focusing of the ions, and secondary effects on the collector on accuracy and reproducibility are discussed in detail. An equation for the vapor pressure of LiCl for pressures between 10^{-10} and 10^{-6} mm Hg is given. The accuracy for abundance measurements of LiCl is 0.2%. With this method an isotopic ratio for Li of 12.41 was obtained.

329-63-151

10435

Mamyrin, B.A.
Shustrov, B.N.Instrum. and Exp. Tech, 3
May-June, 1963 pp. 493-496Measurement of Mass Spectrum of
Residual Gases by Means of
Mass Spectrometers with High
Sensitivity and Resolving Power

It is shown that, for vacuum of 10^{-7} to 10^{-7} to torr and instrumental sensitivity of about $5 \cdot 10^{-12}$ torr, mass spectrum lines of residual gases are revealed in adjustment to virtually any mass number in range from 2 to 45 mass units; in this, many mass numbers correspond to complex mass multiple.

980-64-622

10436

Mason, E.A.
Weissman, S.
Wendt, R.P.

Phys. Fluids 7 pp. 170-173
Feb., 1964

Composition Dependence of
Gaseous Thermal Diffusion Factors
and Mutual Diffusion Coefficients

Radioactive tracers are used to measure thermal diffusion factors and mutual diffusion coefficients for several binary gas systems in which one component is present in trace concentration only. The systems are H_2-Ar^{37} , T_2-Ar , T_2-CO_2 , and T_2-N_2 .

997-63-413

10437

May, H.E.

U.S. Gov't Contract
AT(45-1)-1350
Physical Testing Memorandum
Report PT-63-8 Sept., 1963
General Electric Co.
Hanford Atomic Products Operation
Richland, Washington

Helium and Ultrasonic Leak Test
on N-Reactor steam Generator 4A

The ultrasonic leak detector indicated possible leaks in 37 tubes in the N-Reactor steam generator 4A. However, interpretation of the information was difficult due to high background noise. A Veeco-9A helium leak detector was next used, and leaks in 8 of the 37 suspect tubes were confirmed. The helium leak detector and its operation are described. A correlation of the ultrasonic and helium leak test data is given.

980-65-151

10438

Munro, Donald F.

Research/Development 16
July, 1965 pp. 50-52

Simplifying Leak Detection

Technique using an electronic leak detector on vacuum systems. Tells advantages over conventional mass spectrometer and also has lower cost. Not good if absolute measurements of leaks are required. (Ultek Corp., Palo Alto Calif.)

980-63-130

10439

Nakao, F.

Matsushita Elec. Ind. Co. -
 Nat'l Tech. Report 9 (6)
 Dec., 1963 pp. 499-506

Effect of Temperature on Mass
 Spectra

Effect on ion source temperature is investigated.

980-64-900

10440

Nelson, C.T.
 Gibson, A.S.
 Koontz, R.L.

Trans. Am. Nucl. Soc. 7 pp. 183-
 184 June, 1964
 Atomics International
 Canoga Park, California

997-64-900

10441

Peters, A.H.
 Muhlbaier, D.R.

U.S. Gov't Contract
 AT(07-2)-1, Feb., 1964
 Du Pont de Nemours (E.I.)
 and Company
 Savannah River Lab., Aiken,
 South Carolina

Nondestructive Test of Carbon
 Beds for Reactor Containment
 Applications

A nondestructive technique was developed for evaluating carbon beds used in reactor containment applications. Freon-12 was used as a tracer to detect leak paths that would reduce the efficiency of carbon beds for removal of radioactive I₂ vapor. Leaks greater than 0.006% of the total flow were detected with an electron-capture-type instrument. The current method is suitable for testing dry carbon beds prior to field installation and at air velocities up to 20 ft/min.

335-63-900

10442

Roske, R.W.
 Fuller, D.H.

ISA-J 10 (3) Mar., 1963 pp.
 73-77

Making Detector Comparison Safe

Two readily reproducible techniques are proposed for determining linearity and speed of gas detector response.

335-63-100

10443

Roske, R.W.
Fuller, D.H.ISA Proc. Preprints 13 (3)
Oct., 1963, 9 pages

Method of evaluating Detectors

Evaluation of gas composition detectors by nature of their output signal with respect to concentration and speed of response; simple apparatus for comparing detectors is described.

997-64-600

10444

Ruby, Evain D.

U.S. Gov't Contract
AT(29-1)-1106 Feb., 1964
Dow Chemical Co.
Rocky Flats Div.
Golden, Colorado

An Apparatus for the Preparation
of Standard Gas Mixtures containing
Trace level Components

A gas blending system has been made for use in the preparation of standard gas mixtures. Blends can be made containing minor components at concentration levels down to 10 ppm by volume. These blends are used to calibrate various analytical instruments.

980-64-900

10445

Sansom, W.H.

Shipbuilder Marine Engine-
Builder pp. 34-38 Jan., 1964
Vickers-Armstrongs (Shipbuilders),
Ltd.
Barrows-in-Furness, Eng.

Quality Control and Non-Destructive
Testing in the Construction of
Marine Nuclear Power Plant Part II

Techniques for detection and measurement of gas leaks are briefly described, and some developments in radiographic examination of metal structures (particularly welds) are discussed.

980-60-151

10446

Schumacher, Ernest

Colloq. Spectros. Intern. 8th
Lucern, Stitz., 1959, pp. 29-39
1960

Problems of Mass Spectroscopy

For some mass spectrometers available on the market, some experimental problems are discussed. For quantitative analysis of trace amounts, relatively simple arrangements were found to be quite suitable and using the method of isotopic dilution it was possible to get both sufficient precision and sensitivity up to 10^{-15} g in special cases. Mass spectroscopy of fragments, on the other hand, needed more extended electronic installations and ion sources. Possibilities of a combination of mass spectroscopy and gas chromatography or pyrolysis as well as spectroscopy at very low temperatures and ion emission by field excitation are discussed.

996-49-630

10447

Skinner, W. C.

Thesis 1949
Vanderbilt University
Nashville, Tenn.

Studies in Ultrafiltration with
Metal Membranes

A study was made of preparation of porous silver membranes by chemical attack on the zinc of a silver-zinc alloy. This procedure was standardized so that results are readily reproducible, and membranes of uniform properties are produced.

999-63-900

10448

Steinherz, H. A.

New York, Reinhold Publishing
Corporation, 1963 366 pgs.

Handbook of High Vacuum Engineers

The design, construction, and operation of high-vacuum systems are discussed. Subjects covered include materials of construction, components for producing and measuring high-vacuum, leak detection, and system design. Applications described include vacuum furnaces, coating, and space simulation.

980-63-111

10449

Swift, R.L.

Nat'l Fire Protection Assn.,-
Quarterly 57 (2) Oct., 1963
pp. 168-176

Discussion concerned with portable indicators developed for combustible gases, toxic gases, and oxygen; operating principles and limitation of hot-wire instruments; single and dual filament indicators; problems with high flash point solvents; features of testers for specific compounds, multipurpose toxic gas indicators and analyzers; design of instrument used to measure oxygen deficiency in which flow of electricity through electrolytic solution is proportional to oxygen concentration in sample.

980-63-151

10450

Takeshita, I.

Matsushita Elec. Industry Co.
Nat. Tech. Report 9 (2) Apr.,
1963

Matrices in Ion Optics and their
Application to Mass Spectroscopy

Overall transfer matrix of complex mass spectrograph is calculated by matrix manipulation of transfer matrices of various parts of system; it is shown that multistage magnet system may increase resolving power of direction focusing instrument; methods for increasing resolving power of double focusing instrument are discussed. In Japanese with English summary.

329-63-151

10451

Tantsyrev, G.D.
Karpov, G.V.
Tal'roze, V.L.

Instr. and Exp. Tech.
3 May-June, 1963, pp. 489-492

Analytical Mass Spectrometer
Using Modulated Molecular
Beam

Spectrometer, where gas to be analyzed is admitted in the form of modulated molecular beam, is described.

329-64-151

10452

Zubkov, V.I.

Instr. and Exp. Tech.
Pribory i Tekhn. Eksperim.
2 pp. 127-128 Mar. - Apr., 1964

Device for Automatic Shifting
of a Mass Spectrometer Range

A device is proposed for registering mass spectra over the entire range of a mass spectrometer. The device automatically changes the range of the spectrometer. (In Russian)

364-50-630

10453

Weller, Sol
Steiner, Waldo A.

Journal of Applied Physics
Vol. 21, April 1950

Separation of Gases by Fractional
Permeation through Membranes.

The permeability of a number of thin organic toward oxygen and nitrogen have been measured. For an ethyl cellulose film the studies were extended to include carbon dioxide, helium and hydrogen.

322-54-630

10454

Brubaker, D. W.
Kammumeyer K.

Industrial and Engineering Chemistry
Vol. 46, No. 4. pp. 733 (1954)

Separation of Gases by Plastic
Membranes.

Data on permeability of four plastic membranes to helium, hydrogen, oxygen, nitrogen, carbon dioxide, ammonia, and sulfur dioxide have been obtained. The plastic films included in this study were polyethylene, trifluoromonochloroethylene, a copolymer of polyvinyl chloride with polyvinyl acetate, and cellulose acetate-butyrate. Most of the gas permeabilities increased exponentially with an increase in temperature.

364-60-630

10455

Lieby, C.C. Jr.
Chen, C.L.

Journal of Applied Physics
Vol. 31. No. 2, Feb., 1960

Diffusion Coefficients, Solubilities,
and Permeabilities for Helium, Neon,
Hydrogen and Nitrogen in Vycor
Glass.

The permeability of Corning Vycor glass (0.25 mm thick) is given for helium at six temperatures ranging from 299° K to 723° K, for neon and hydrogen at 673° K and 723° K, and for nitrogen at 673° K. On the basis of the observed permeation rates, it is estimated that the filtered helium has an impurity content of less than 1/50 th if its unfiltered value.

322-52-630

10456

Brubaker, D.W.
Kammermeyer, K.

Industrial Engineering Chemistry
Vol. 44, pp 1465-1473
June 1952

Separation of Gases by Means
of Permeable Membranes

Study of permeability of plastic membranes to gases. The effect of temperature upon the permeation of gases through plastic films is not readily predictable. Information on the rate of gas permeation through a number of selected plastic membranes at different temperatures were obtained. Gases used in this experiment were nitrogen, oxygen, hydrogen, and carbon dioxide.

004-47-630

10457

Sarge, T.W.

Analytical Chemistry 19396
1947

Determinative of Gas Permeability
of Saran Films

A modified manometric apparatus for measuring gas permeabilities of films having extremely low transmission requirements is described. Experimental results of equilibrium transmission for Saran (trade mark, Dow Chemical Company) films measured by a variable pressure technique are reported and discussed.

980-50-630

10458

Van Amerongen, G.J.

Journal of Polymer Science
Vol. V, No. 3, 1950
pp. 307-332

Influence of Structure of Elastomers
in their Permeability to Gases.

Study of rubber membranes and their given permeabilities to given gas.
Helium, hydrogen, nitrogen, and carbon dioxide were used in the experiments.

980-54-630

10459

Meares, Patrick

Journal of American Chemical
Society
Vol. 76, pp. 3415, 1954

The Diffusion of Gases Through
Polyvinyl Acetate

The diffusion of helium, neon, argon, oxygen and hydrogen through high
molecular weight polyvinyl acetate is studied in the temperature ranges 4° -
44° C.

322-65-630

10460

Stein, Sinclair, Garcis
Vahldieck and Mohr

Industrial Engineering Chemistry
Vol. 57, No. 2, Feb., 1965
pp.49-60

Helium Recovery by Permeation

Describes a new process of helium recovery from natural gas by a selective
permeation through membranes of Teflon FEP, a copolymer of tetrafluorethylene
and hexafluoropropylene.

382-22-630

10461

Williams, G. A.
Ferguson, J. B.

Journal of American Chemistry
Vol. XLIV, No. 10, October
1922.

The Diffusion of Hydrogen and
Helium Through Silica Glass
and other Glasses.

Study of permabilities of silica and other glasses to helium and hydrogen.
The permeability is proportional to the gas pressure and is an exponential
function of temperature. At 500° C helium had a permeability of 22 as
compared to hydrogen.

CHAPTER VIII
THE DUAL-FLOW PRINCIPLE APPLIED TO THE
HALOGEN GRADIENT DETECTOR

Currently available systems of leak detection and location, using freon concentration measuring equipment, suffer from background contamination difficulties. Freon, being a heavy gas, exhibits troublesome pooling effects. Also, large leaks tend to swamp out nearby smaller leaks. To overcome the attendant reduction of sensitivity of detectors responding to total halogen concentration, use may be made of time-derivative action by inserting a capacitor in the current measuring circuit to block out the average (dc) component. A halogen gun so operated (automatic zero mode, auto balance mode) may be made to give a rough space gradient indication by moving its probe uniformly through the air. However, this requires considerable skill and may also, if high velocity is used in an attempt to increase sensitivity, disturb the distribution it seeks to investigate.

Under Contract NAS8-2563, a two-diode halogen gradient sensitive bridge was developed and tested. This instrument proved to be capable of locating small leaks in the presence of background so strong that the ordinary halogen gun was saturated (off scale) on its least sensitive range. These experiments are described on Page 47 of the Second Formal Report (June 1963.)

A serious problem with the practical realization of the advantages of this bridge results from the differences in response curves of the individual diodes,

and especially from their different rates of aging. This necessitates constant rebalancing of the bridge for each different background and for each time of use. In order to overcome this difficulty, the time-sharing halogen gradient detector was developed.

This detector ingests samples through two probes at slightly different locations; either right and left probes, or fore and aft probes may be used. The probes are connected to the diode through a gating valve which causes the diode to accept samples alternately from each probe. When the two probes are in the same halogen concentration, the samples seen by the diode are identical, and a constant (dc) current results. Where one probe takes in stronger halogen concentration than the other, as it must if a gradient is present and not normal to the probe axis, the diode current will have an ac component. The ac component is separated from the dc component and amplified to give the gradient signal. A prototype instrument was built and checked out. Experience with this first model led to design changes in the circuitry and the building of two more prototype detectors. These two improved circuits have been delivered to NASA in Huntsville for further experimentation.

After several minor circuit and output indicator modifications, AeroVac Corporation was asked to produce several working models of the time-sharing gradient detectors. AeroVac was also instructed to design and build the mechanical probe and associated pumping equipment.

Although the time-sharing model was much superior to the older dual-diode system in terms of stability, the newer model was limited in terms of response time.

This limitation was inherent in the low switching rate (4 cycles per second) required by decay time considerations* connected with switching the flow of gas through the single diode. Proper operation of the diode limited this switching rate to approximately 4 cycles per second. In addition, transients introduced by the on-off operation led to undesirable sound effects in the audio leak indicator. In attempting to produce an even better system, we experimented with the dual-flow principle which involves separating the standard GE diode into two equal halves divided longitudinally. The two halves are both electrically and pneumatically separated.

The two halves of the cathode operate with a common anode (and a common heater) to form a dual-diode. Two steady streams of flow operating with the common anode provide a reasonably even temperature and offset the troublesome stability problems previously encountered using the two completely separate diodes. Sampling is now performed at a rate established electronically and optimized for more efficient leak detection. This switching in the demonstration model at a chosen 400 cycle rate is performed by alternately applying operating voltage to one cathode and then the other. The switching rate was chosen rather arbitrarily due to availability of 400 cycle choppers and circuit components. (A 60 cycle chopper is specified in the schematic of Figure 36).

Operators having experience with our earlier time-sharing halogen gradient detector estimate that the response time of the dual-flow system is at least four times better than the older model. In addition, the dual-flow system has a much more workable type of pickup which consists of two 6-inch tubes two millimeters

* Chapter II

in diameter and with the inlets located side by side. The valve-switching apparatus is no longer needed which considerably simplifies construction of the gun. In fact, with very slight mechanical modifications the standard GE gun can be used. Finally, there are no distracting noises produced primarily in the older model by the mechanical valve mechanism.

Since no system designed and constructed by human effort ever attains perfection, at this point it seems desirable to list those factors which might tend to produce unequal response from the two diode halves even when the freon concentrations are identical. Using the standardization laboratory, several days were spent in taking measurements of ionization current versus freon concentration in gas flow through the two halves of the dual-diode. This data is listed in Table 8 and plotted in Figure 34. Since a rather elaborate setup was required even with the use of the standardization laboratory to obtain precisely-known concentrations in the order of a few parts per million of freon, appreciable time intervals were necessary which unfortunately permitted some moderate and unavoidable variations in operating conditions between readings. Therefore, the curves shown in Figure 34 are slightly pessimistic in comparison with actual leak detection operation conditions of the dual-flow system. Since diode contamination even for moderate values of freon concentration does change the diode characteristics, an effect similar to hysteresis in magnetic circuits is exhibited. This causes the data points to be spread throughout an area rather than falling along a single curve. Other environmental factors difficult to control precisely likewise produce a spreading out of the data points. These factors include locally generated power supply fluctuations appearing in a

random fashion, slight variations in storage tank pressures (maintained at approximately 50 psi), slight fluctuations in heater voltage (fed from a regulated supply), slight variations in temperature of the sample gas fed to the probe, and a slight background contamination of freon in the environment.

Using the single available sample of split-cathode diode, the two curve areas do partially overlap which indicates that the two halves are reasonably balanced and should indicate the presence of a concentration gradient accurately enough for the application described. The demonstration model which is shown in Figures 37A and 37B has indicated by actual test that the concept gives a good oscilloscope indication of gradient with a completely acceptable response time even with the rather unsophisticated circuitry employed (see Figure 38). Time and funds do not permit additional data checkouts, using the dual-flow concept and the schematic shown in Figure 36. However, the Figure 36 electronics circuitry operated successfully with the two-diode model, neglecting the time-varying instability and drift of the two separate diodes. There is no reason at this point to question the workability of the schematic given with the inherently more stable split-cathode single diode.

An abbreviated description of some of the features of the electronic circuitry shown in Figure 36 will be included at this point. A much more extensive explanation of the electronic features and directions for operation of the equipment are given in the Second Formal Report, NAS8-2563 (June 1963), on pages 39-51.

Each of the two probe nozzles picks up a steady flow of air-freon mixture in the vicinity of a leak. Since a gradient in concentration is normally present under

these circumstances, the probe near the leak will carry a slightly higher concentration of freon. By the complex ionization process described in detail in Chapter II, the diode-half carrying the heavier freon concentration will conduct differently from the other half, resulting in the unbalance of a direct current bridge. The potential difference is applied to the two grids of V_1 . These grids are also fed with two 60-cycle voltages 180° out of phase with each other. A high-gain differential amplifier and chopper arrangement to obviate the effects of drift plus a regulated power supply accomplish the desired electronic stability.

Test results stated in the Second Formal Report indicate that the detector is capable of locating a leak of less than 6×10^{-5} SCIM in an ambient background contamination capable of causing half scale deflection on the intermediate-range scale of the standard H-2 General Electric halogen detector. In the same circumstances, the standard halogen detector showed no signal variation near the leak and was completely incapable of detecting it.

It is regrettable that limited funding and contract time did not permit the transistorization and miniaturization of the electronics package shown in Figure 36. However, as long as the standard hot-anode platinum diode is heated electrically, the large power (40 watt) demand requires a power cord connection to the 60-cycle supply in any case. Therefore, the small amount of additional power required by conventional vacuum tubes is not a serious objection. If a propane-heated diode described in Interim Report No. 1, NAS8-11199 (April 1965), pages 62-71, is constructed at some future date, then it would be important for complete portability to transistorize the electronics package, and use batteries as a primary power source for the circuitry.

Parts Per Million	Diode 1	Diode 2
0.59	1.04	1.04
0.59	1.00	1.00
1.14	1.10	1.10
0.92	1.09	1.09
0.97	1.05	1.10
1.30	1.18	1.20
2.05	1.20	1.25
2.26	1.27	1.26
3.00	1.40	1.48
3.33	1.34	1.44
2.92	1.35	1.40
3.88	1.40	1.50
3.97	1.50	1.55
2.43	1.20	1.35
4.23	1.49	1.70
4.40	1.40	1.45
5.10	1.65	1.75
5.50	1.70	2.10
5.50	1.65	1.78
6.80	2.25	2.27
6.80	1.63	1.85
6.55	1.80	2.05
7.90	1.75	2.35
7.90	1.85	2.40
8.00	1.80	2.17
8.57	1.65	1.90
9.40	1.80	2.60
10.00	2.35	2.60
10.20	2.20	2.50
10.70	2.30	2.80
8.00	1.70	2.40
11.20	2.20	2.65
12.60	2.15	2.20
10.50	1.85	2.37
12.20	2.00	2.20
14.10	2.40	2.85
15.00	2.70	2.95
13.70	2.40	2.80
13.00	2.60	2.90
17.00	2.35	2.70
17.00	2.80	2.65
17.00	2.85	3.40

THE SPLIT CATHODE DUAL-FLOW DIODE

Note (a): All ionization currents given in microamperes.

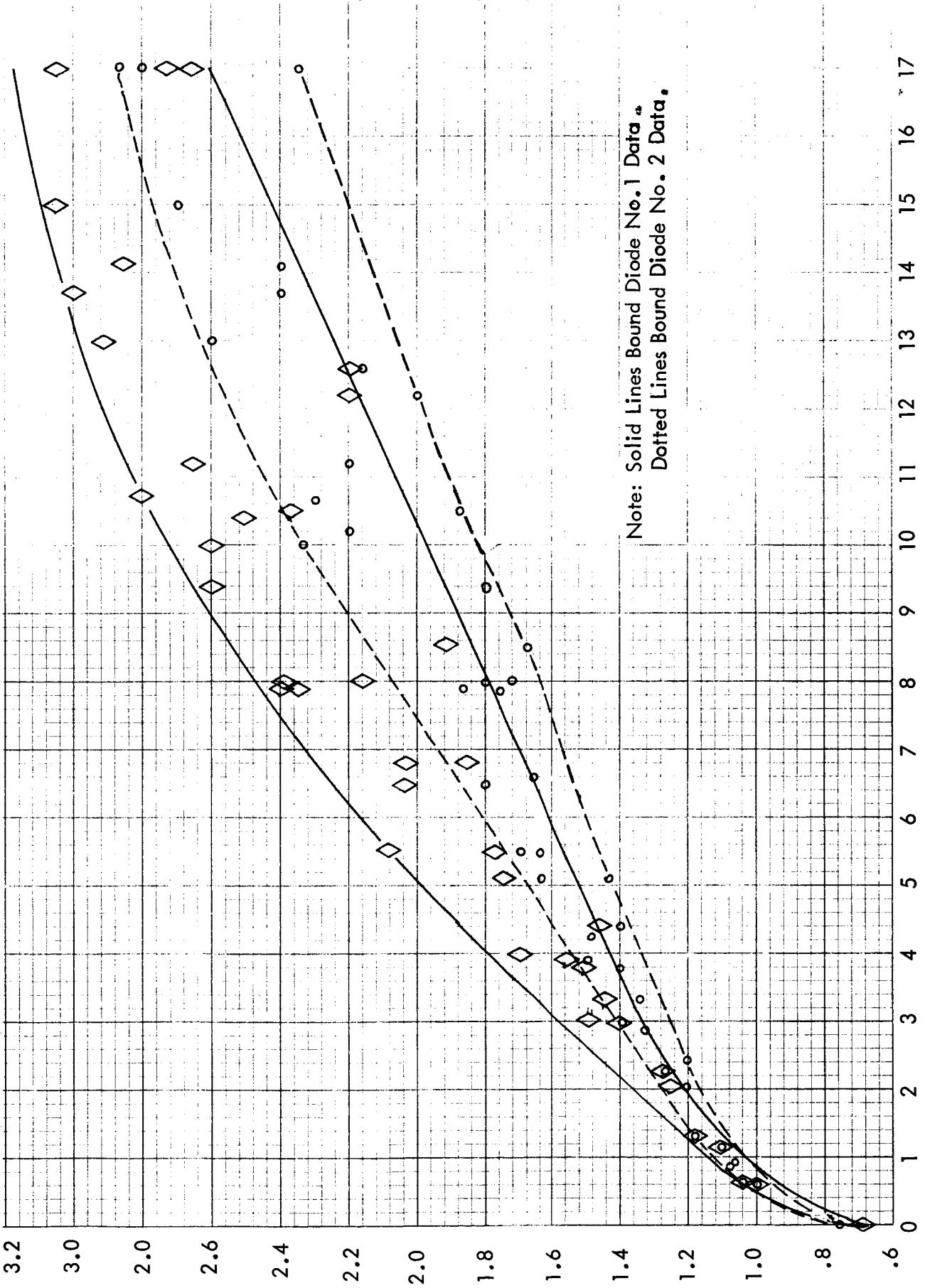
Note (b): Pressure in sample tank = 50 psi (gage)

Note (c): Diode 1 idle current = 0.76 (average of 22 readings)

Note (d): Diode 2 idle current = 0.68 (average of 22 readings)

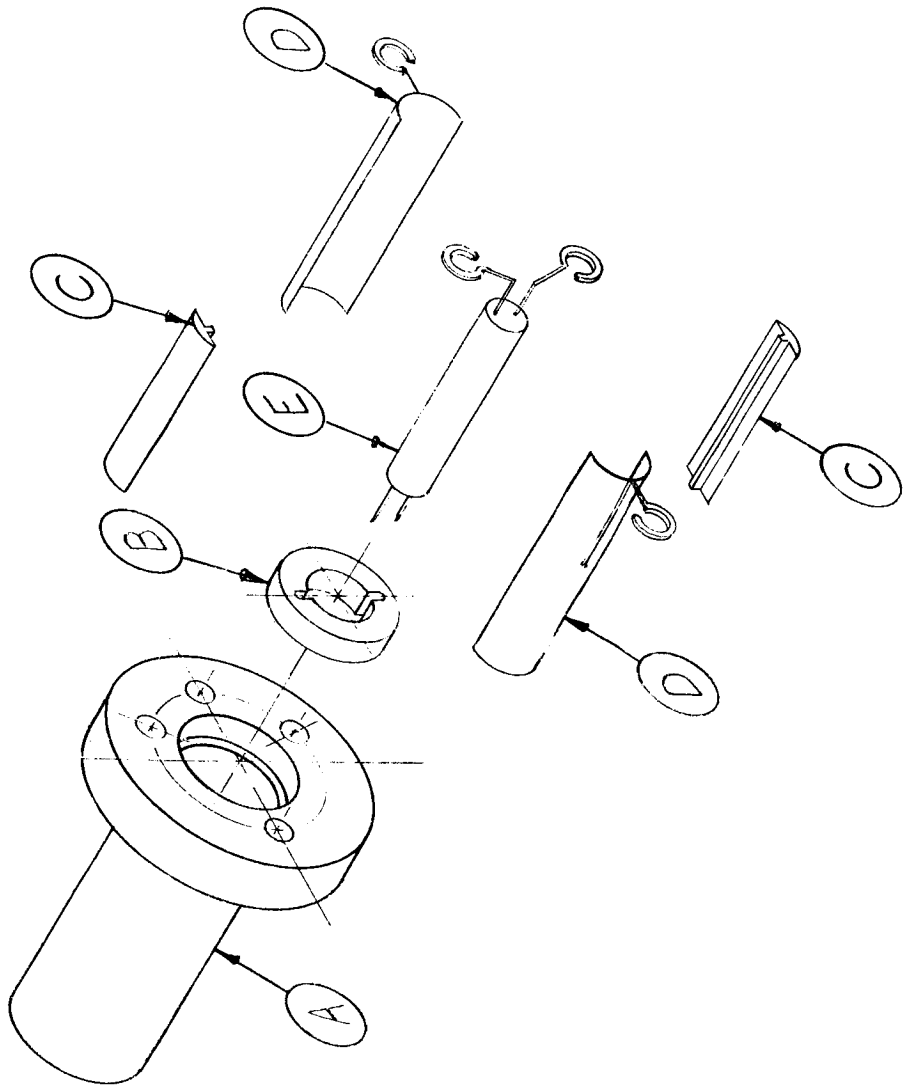
Ionization Current vs Freon Concentration

Table 8



Freon Concentration in Parts Per Million.
Ionization Current of Dual Flow Diode.

Figure 34



A... Ceramic Sleeve Base

B... Ceramic Bushing Retainer

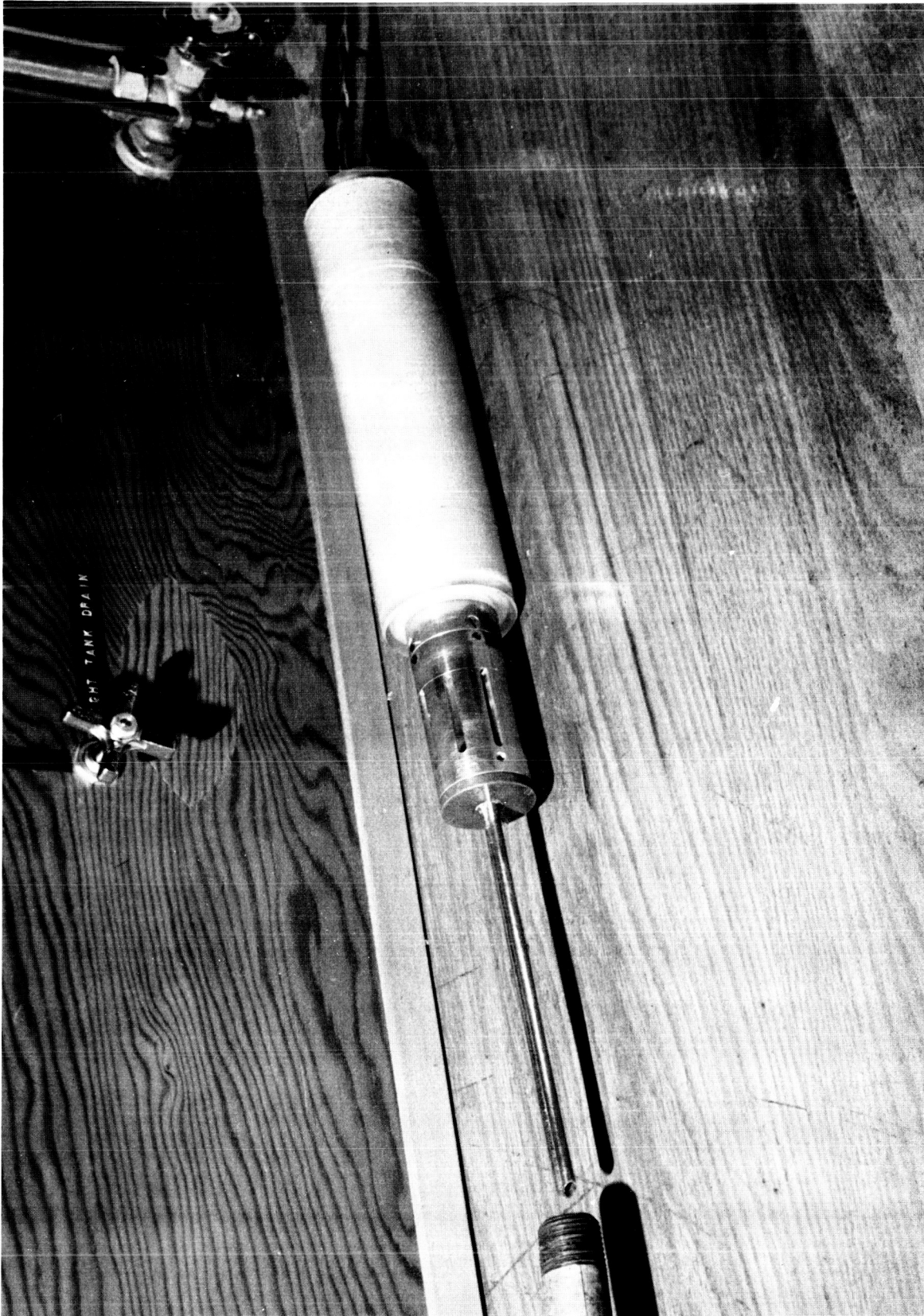
C... Ceramic Flow Separators
(Fabricated by Ohio University)

D... Split Platinum Cathode
(Machined by Ohio University)

E... Platinum Anode and Heater

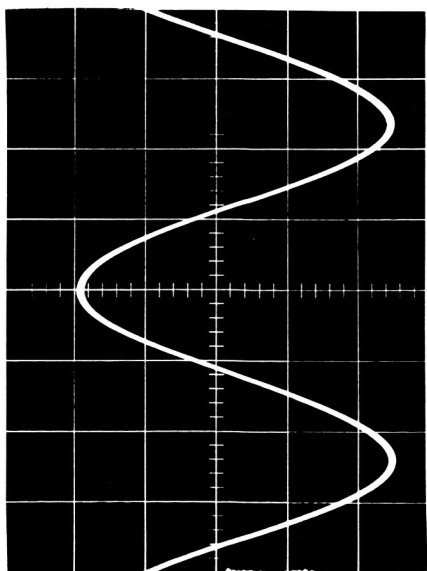
Isometric of Dual-Flow Diode

Figure 35

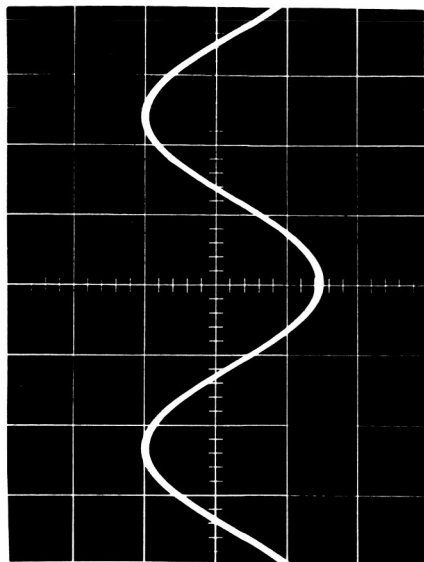


Model of Dual Flow Diode Probe

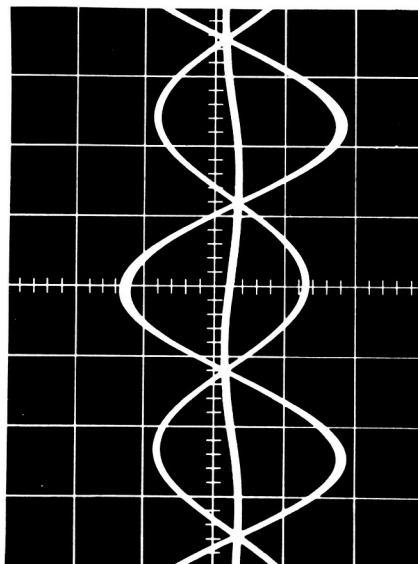
Figure 37 A



Probe Centerline 0.5 cm to Left of Leak *



Probe Centerline 0.5 cm to Right of Leak *

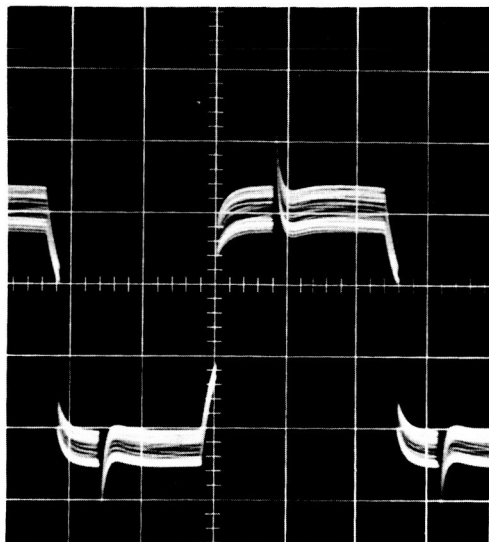


Triple Exposure as Probe Passes Leak *

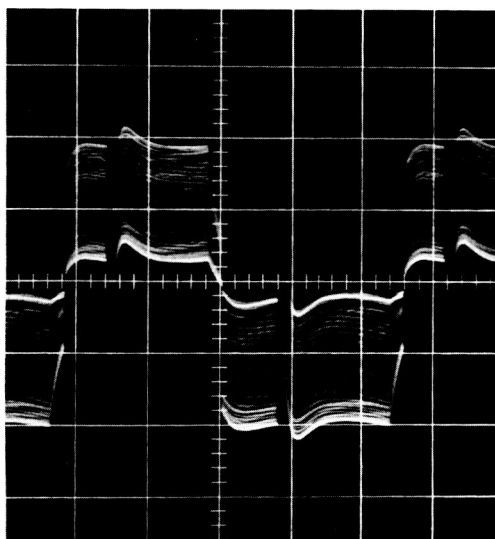
* Leak Rate = 0.2 oz. per year

Demonstrator Amplifier Output Voltage

Figure 38



Chopper Output Voltage
(probe 0.5 cm Left of Leak)



Chopper Output Voltage
(probe 0.5 cm Right of Leak)

Chopper Output Voltage Waveform

Figure 39

CHRONOLOGY OF RESEARCH PERFORMED

FIRST FORMAL REPORTLeak Detection Technique Improvement
Study For Space Vehicles

NAS 8 - 2563

September 1962

- I State-of-the-Art Survey
 - A General
 - B Experimental Evaluation of the Halogen Leak Detector
 - C Thermal Conductivity Type Leak Detector
 - D Evaluation of Ultrasonic Translator
- II Literature Search
- III Locally Generated Ideas
 - A Resonant Cavity Scanning System
 - B Radon-222 and Polonium-210 as Tracer Gases in Leak Detection
 - C Solid State Trigger Devices
- IV Approaches Suggested by the Literature Search
 - A The Hot Ceramic Detector
 - B Vibrating Capacitor
 - C Miniaturized Mass Spectrometer Leak Detector
- V Suggestions for Test Procedure Modification
- VI General Theoretical and Experimental Studies of Leaks
 - A Concentration of Leaking Gas as a Function of Leak Rate and Distance from Leak
 - B Proposed Ultrasonic Spectrum Analysis of Leak Noise

SECOND FORMAL REPORTLeak Detection Technique Improvement
Study For Space Vehicles

NAS 8 - 2563

September 1962

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- II General Detection and Location of Leaks
 - A Range of Required and Attainable Sensitivity
 - B Factors Involved in the Evaluation of Sensitivity Figures
 - C Sampling Efficiency
 - D Tracer Economy
 - E Problems of Noise and Contamination
 - F Some Problems of Gradient Sensing
- III State-of-the-Art Devices
 - A Group 1 Pressures and Flow Meters
 - B Group 2 Gas Collectors
 - C Group 3 Tracer Methods
 - 1 Halogen Detectors
 - 2 Mass Spectrometers
 - 3 Thermal Conductivity Methods
 - 4 Infrared Detectors
 - 5 Radioactive Tracers
 - 6 Olfactory Detection
 - D Group 4 Acoustical Methods
 - E Group 5 Miscellaneous Methods

IV Halogen Gun Studies

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- B Experimental Evaluation of the Halogen Leak Detector
- C The Halogen Gradient Detector
- D Correlation of Tests on G.E. H-2 Halogen Detector, NRL Thermistor Bridge, and O.U. Halogen Bridge
- E Operation of Halogen Detectors

V Thermistor Bridge

- A Functional Description
- B Advantages
- C Operation of Thermal Conductivity Bridges

VI Miniaturized Mass Spectrometer

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VII Acoustical Methods

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 - 2 Effect of Small Obstructions between Leak and Detector
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VIII Systems Concepts - Radar Cavity System

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- B Tunnel Diode Triggered Oscillator - The Active Approach

IX Miscellaneous Devices

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- C Properties of Mercaptans
 - 1 Toxicity
 - 2 Physical Properties
 - 3 Chemical Properties
 - 4 Availability
- D Methods Involving Radioactivity
 - 1 Health Considerations
- E Vibrating Capacitor Electrometer
- F TCNQ Detectors
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X Bibliography

- A Literature Search
- B Key to Symbols
- C Bibliography
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XI Selected Abstracts

XII Conclusions and Recommendations

INTERIM REPORT NO. 1

Leak Detection Technique Improvement
Study For Space Vehicles

NAS 8 - 11199

April 1965

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- II Literature Search and Bibliography Automation
- III State-of-the-Art Survey
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 - B Operation of the Gradient Detector
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 - 2 Manual Operation
 - 3 High Background
 - C Circuit Differences Between the Model 1 and Model 2
- VI Acoustical Methods
- VII Halogen Diode Improvement Studies
- VIII Vibrating Capacitor Studies
- IX Americium 241 Study