EXPERIMENTAL EVALUATION OF THE RATE OF RISE TECHNIQUE FOR MEASURING OUTGASSING RATES IN VACUUM

by

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IV. LIST OF SYMBOLS

A area, cm^2

F conductance, liter-sec^{-1}-cm^{-2}

L leak rate, torr-liter-sec^{-1}

N number of molecules, molecules

P pressure, torr

Q outgassing rate, torr-liter-sec^{-1}

S pumping speed, liter-sec^{-1}

s ionization gage sensitivity, $torr^{-1}$

T temperature, ^{o}R

t time, sec

V volume, liter

x displacement, mm

V. INTRODUCTION

With an increase of interest in space flight and vacuum research, there has been a corresponding increase in the need for values of outgassing rates of many materials. In space flight the knowledge of the outgassing rates of components and materials used in construction of space vehicles allow the determination of pressures within sections of the vehicle, the contamination level of critical components, and the reliability of vehicle components. In the construction of an ultra high vacuum facility, the knowledge of the value of the outgassing rates of construction materials allows the chamber to be constructed of low outgassing materials, minimizing the amount of gas evolving from the chamber walls. With the initiation of a research program, the outgassing rates of the chamber, test objects, and instrumentation are needed to determine the level of vacuum obtainable during the investigation.

With the increased need for outgassing rates, more emphasis has been placed on the measurement of outgassing rates. The literature reports several techniques for the measurement of outgassing rates. Of these techniques, the rate of rise method is simple and convenient to apply, and hence of much interest. Because it is simple and easily applied, the rate of rise technique has been used by many experimenters to measure the outgassing rates of various materials. However, some experimenters have rejected its use as they felt that the dynamic nature of the technique introduced large errors into the outgassing measurements. There is presently in the literature no technical evaluation of the rate of rise method as to the suitability or unsuitability for measuring outgassing rates.

The purpose of this thesis is to experimentally evaluate the rate of rise technique of measuring outgassing rates and to investigate the errors and inaccuracies of the technique. The subject investigation was conducted under the auspices of the National Aeronautics and Space Administration at the Langley Research Center, Hampton, Virginia.

VI. REVIEW OF LITERATURE

Introduction

With the advent of space flight, there has been an increasing demand for the evaluation of the effects of space environment on spacecraft materials, especially the effects of vacuum outgassing. Outgassing is a general term referring to the evolution of gas from a material in vacuum. Outgassing cannot only affect the properties of a material but can also retard the efforts to simulate the vacuum of space. In many cases the lowest pressures in a vacuum simulator are limited by outgassing rather than design criteria. Thus, it is often necessary to determine outgassing rates for the purpose of designing a vacuum facility as well as for evaluating the effects of the vacuum of space.

When confronted with the problem of measuring the outgassing rate of a material, one inevitably searches the literature to find a technique for making the measurements. Several methods for measuring outgassing rates are documented in the literature. These methods vary in the complexity and convenience of application.

The purpose of this literature review is to briefly discuss the techniques available for measuring outgassing rates and then to expound on the rate of rise method, discussing the errors associated with the technique. In conjunction with the error discussion, the skepticism concerning the rate of rise technique will be brought into focus. It should be noted here that the American Vacuum Society recognizes the

need for standardization in the field of outgassing measurement, and therefore, have issued a tentative AVS standard[1] on the reporting of outgassing data.

Techniques of Measuring Outgassing Rates

In searching the literature for methods of measuring outgassing rates, three methods are generally found, the weight loss method, the conductance method, and the rate of rise method. Each of these methods are frequently used by experimenters to measure outgassing rates and each has advantages and disadvantages associated with its use. The weight loss method is a direct method of measuring the outgassing rate where the rate of weight loss from a material in vacuum is attributable to the outgassing rate of the material. Caruso[2], in his study of outgassing, used the weight loss method and concluded that the technique was limited by the sensitivity of the weighing apparatus, as large amounts of outgassing have to occur before noticeable loss of weight appears.

The conductance and rate of rise method of measuring outgassing rates are based on simplifications of a general equation derived for a closed system of the type shown in figure 1. Greenwood[3] derived this equation. The approach used in the derivation is that a sample whose outgassing rate is to be measured is located in a volume, V_1, where there exists a pressure, P_1, and a temperature, T_1. Volume, V_1, is a vacuum in which free molecular flow exists. A molecular balance for the system is performed in which equations are written for the rate at which molecules enter V_1, the rate at which molecules leave

V_1, and the rate of change of molecules in V_1. From figure 1 the following quantities are defined:

V_1 = Volume of vacuum vessel

P_1 = Pressure existing in volume V_1

P_2 = Pressure at exit side of orifice

T_1 = Temperature of volume V_1

N_1 = Number of molecules present in volume V_1

N_c = Number of molecules entering V_1 from the chamber walls

N_s = Number of molecules entering V_1 from the test material

N_L = Number of molecules leaving V_1 through the orifice

The equation derived by Greenwood from the molecular balance of the system in figure 1 is

Rate of change of number of molecules in V_1	=	Rate at which molecules enter V_1	-	Rate at which molecules leave V_1

that is,

$$V_1 \frac{dP_1}{dt} = Q - FA(P_1 - P_2) \tag{6.1}$$

where:

V_1 = Volume of vacuum vessel

$\frac{dP_1}{dt}$ = Rate of change of pressure in V_1

Q = Outgassing rate of both sample and chamber

F = Conductance of orifice as defined by Dushman[4]

A = Area of orifice

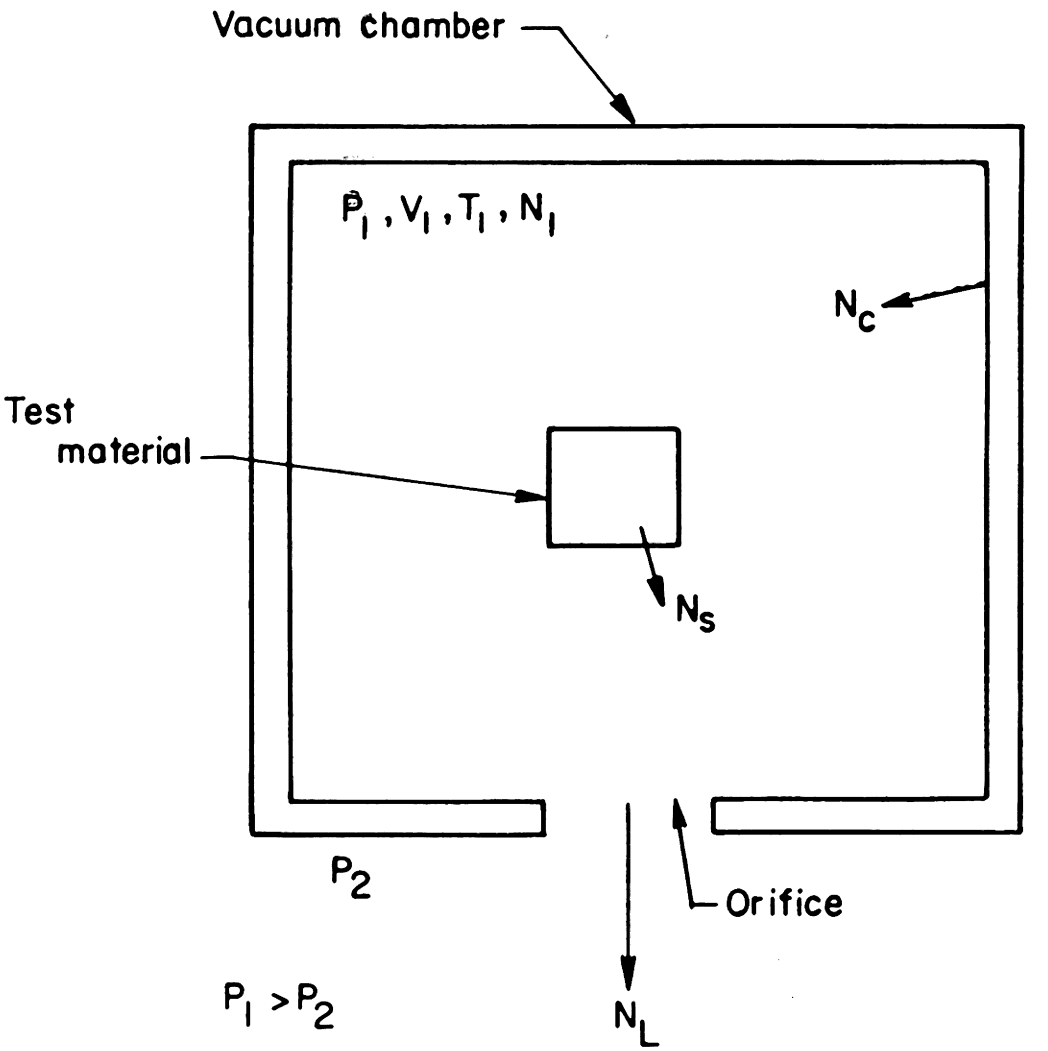


Figure 1.- System configuration for derivation of the outgassing equation.

P_1 = Pressure in V_1

P_2 = Pressure on exit side of orifice

Equation 6.1 was derived with the assumptions:

(1) That all gases are ideal gases and obey the equation of state for ideal gases,

(2) that isothermal conditions exist in the system,

(3) that the gases exhibit a Maxwellian velocity distribution,

(4) that the mean free paths of the gas molecules are large as compared to the largest dimension of the chamber,

(5) that P_1 is greater than P_2, and

(6) that the outgassing rate is defined in terms of the properties of the gases entering the system; that is, $Q \equiv \frac{d}{dt}(PV)$.

Both the conductance and rate of rise methods are based on simplifications of equation 6.1. The conductance method utilizes an equilibrium approach, whereby gas is continuously exhausted from V_1 through an orifice of known conductance. When steady state pressure conditions are reached in V_1 then the simplification that

$$\frac{dP_1}{dt} = 0$$

can be applied to equation 6.1. The resulting equation for the conductance method is

$$Q = FA(P_1 - P_2) \tag{6.2}$$

The application of equation 6.2 to a practical vacuum system requires that a vacuum source be attached to the exit side of the orifice such that gas is continuously exhausted from V_1. The orifice is located

between the vacuum source and the vessel containing the test specimen and the conductance of this orifice determines how much time will elapse between the time when the gas load is introduced into V_1 and when steady state pressure is reached. Therefore, by measuring P_1 and P_2 with pressure gages and by knowing the conductance and area of the orifice, the outgassing rate can be determined by equation 6.2.

There are certain errors[5], such as gage pumping, associated with the violation of the theory of equation 6.2 when applying the conductance method to an actual vacuum system, but these will not be discussed in this thesis. There is a second group of errors, those associated with the measurement of the terms in equation 6.2 and these will be discussed briefly. The conductance method of measuring outgassing rates requires the determination of an area, a conductance, and two pressures. The area of the orifice is easily measured. With free molecular flow, the conductance of an orifice varies as the inverse square root of the molecular weight of the gas passing through the orifice.[6] Therefore, to determine the conductance term, the composition of the outgassing products from the sample must be known. In many outgassing studies this composition is not known and may change during the investigation. Because of the difficulty in determining the outgassing composition, frequently the conductance term is determined by assuming a molecular weight. The pressure terms in equation 6.2 are usually measured with ionization gages and since the sensitivity of such a gage depends upon the composition of the gas present, the determination of the pressure terms requires a knowledge of the gas composition. The use of the conductance method

without knowledge of the outgassing composition can introduce sizeable error into the outgassing measurements and care should be taken to insure that the assumption of a molecular weight is practical for the investigation under study. The need for two pressure gages and the accurate determination of the conductance of the orifice can complicate the experimental investigation.

The conductance method uses an equilibrium approach, but the rate of rise method employs a dynamic approach. The product of conductance and area, FA, in equation 6.1 can be replaced by the net pumping speed of the vacuum pumps, S, at the orifice. This substitution will aid in the discussion of the rate of rise method. The resulting equation is

$$V_1 \frac{dP_1}{dt} = Q - S(P_1 - P_2) \qquad (6.3)$$

The simplification applied to equation 6.3 in using the rate of rise technique is that the pumping speed, S, is made zero by isolating volume V_1 from the pumping source. Equation 6.3 then becomes

$$Q = V_1 \frac{dP_1}{dt} \qquad (6.4)$$

and the outgassing rate is a function of a constant, V_1, and a variable, the rate of change of pressure in the isolated volume. Applying the rate of rise method to a practical vacuum system requires that a valve exist in the system such that volume V_1 can be isolated from the vacuum source, making S zero in equation 6.3. A pressure gage is positioned in the closed volume to record the rate of change of pressure due to outgassing. As in the conductance method the pressure gage used is an ionization gage and the sensitivity of the

gage depends on the gas composition. However, since the rate of rise method only requires the knowledge of the gas composition to determine $\frac{dP_1}{dt}$ in equation 6.4, a sensitivity for nitrogen can be used and outgassing rates reported in equivalent nitrogen units. This is consistent with applied vacuum technology, since ionization gages are frequently calibrated for nitrogen and pressures are reported in equivalent nitrogen units. There are additional gage effects which can introduce error into a rate of rise measurement, such as the Blears effect[7] and gage conductance. These errors are of interest only in special cases and can be minimized or eliminated, and hence will not be discussed.

Violation of Initial Assumptions: Rate of Rise

In deriving equation 6.4 for the rate of rise method, the assumption was made that no gas was being removed from the test volume; that is, the pumping speed, S, in equation 6.3 is made zero. In the application of the technique, any process which removes gas from the test volume violates this assumption and hence is a source of error. There are two processes which can frequently occur during a rate of rise measurement and which result in the removal of gas from the closed volume. These include adsorption pumping by the chamber walls and ionization gage pumping.

The procedure used in applying the rate of rise method to the measurement of an outgassing rate is that the test specimen is placed in a vacuum vessel and the vessel is pumped to a pressure where free molecular flow exists. To make a measurement, a valve is closed,

isolating the test chamber and sample from the vacuum source and then the change in pressure with time due to outgassing is recorded. From the recorded pressure behavior the outgassing rate of the chamber and test specimen is calculated and then by appropriate correction for the chamber outgassing, the outgassing rate of the test specimen can be determined. After the pressure has been recorded for the required time, the valve is opened and the pressure returns to its initial level. Initially, before the valve is closed, there is a quasi-equilibrium condition between the number of molecules striking the chamber walls and the number of molecules leaving the chamber walls; that is, the number of molecules remaining on the walls is constant over a finite time. The walls of a vacuum chamber can accommodate a finite quantity of gas and this quantity is dependent upon the properties of both the gas and the walls. With the system in this quasi-equilibrium condition the walls may be saturated with gas or may possess the ability to accommodate additional gas molecules. When the valve is closed to take a measurement, the quasi-equilibrium state is upset and as the pressure rises in the closed volume due to outgassing, the number of molecules striking the chamber walls becomes greater than those leaving the walls. In an attempt to return to a quasi-equilibrium state, the walls, depending upon its degree of saturation, retain some of the impringing molecules. When the valve is opened, the system returns to a quasi-equilibrium state. This retaining of gas molecules by the chamber walls is called adsorption. There is a finite period of time in which adsorption occurs and this time can be small (micro seconds) if the walls are near saturation or large (hours) if the walls are fairly free of molecules when the valve is closed. Among the factors influencing the rate of adsorption are

the condition of the chamber walls, the gas species present, the pressure, and the temperature. This adsorption process reduces the pressure in the volume and leads to a reduction in the observed outgassing rate. Not enough data is available to evaluate the significance of adsorption effects as applied to the rate of rise technique, but it is well known that these adsorption effects can introduce large errors.[8,9,10]

Some work has been done on evaluation of the adsorption effects; for example, Markley[11] found a pressure, 5×10^{-4} torr, above which adsorption seemed to have no effect on rate of rise measurements. Others, like Wightman[12], are investigating adsorption effects in general. The preliminary results from Wightman's work indicate that pressures much lower than 5×10^{-4} torr may be necessary before significant adsorption of some common gases occur. For the present time the effects of adsorption in a rate of rise measurement cannot be evaluated due to the lack of adsorption data.

It is a well known fact that the operation of an ionization gage in vacuum results in the pumping of gas molecules. This pumping action is a source of widespread investigation [13-18]. Since the determination of the pressure term in equation 6.4 usually involves the use of an ionization gage, the assumption that S is equal to zero is again violated. Although the literature indicates various gage pumping mechanisms and a variety of pumping speeds, it is readily accepted that a reduction in gage emission current decreases the pumping action of the gage. Greenwood[19] in his investigations of outgassing rates by the rate of rise method showed that known air

leaks could be more closely measured if the gage emission current was reduced. It will be shown later in this thesis that the error introduced into the rate of rise measurement by gage pumping can be accounted for and made negligible for many applications.

Rate of Rise Pressure Traces

Besides the simplicity offered by the rate of rise technique, another advantage attributed to the technique is that the recorded pressure-time trace may be used to detect the presence of adsorption, gage pumping, and other error introducing processes. Since these processes affect the pressure-time behavior by affecting the pumping speed, equation 6.3 will be evaluated for different pumping speed behavior. From these considerations, the type of pressure-time traces that might be expected in a rate of rise measurement can be developed.

In the first case, if the assumption that S in equation 6.3 is zero is valid, then equation 6.4 applies. Rewriting this equation without the subscripts gives

$$Q = V \frac{dP}{dt} \tag{6.5}$$

If during the measurement period, (typically a few seconds to several minutes), the outgassing rate, Q, is assumed to be constant, then equation 6.5 can be intergrated.

Equation 6.5 becomes

$$P = Qt/V + P_o \tag{6.6}$$

where:

P = Pressure at any time in the isolated volume

Q = Outgassing rate

t = Time after valve closure

V = Volume of test chamber

P_o = Initial pressure in test volume at t = o

Equation 6.6 shows pressure as a linear function of time.

A pressure-time plot of this equation is shown in figure 2(a). Equation 6.6 and figure 2(a) are representative of the theoretical outgassing rate as predicted by the theory.

A second case occurs when a process occurs in the closed test volume that pumps gas at a constant rate, then S in equation 6.3 is constant and intergrating with this condition results in

$$P = (Q/S)(1 - \exp(-St/V)) + (P_o)\ (\exp(-St/V)) \qquad (6.7)$$

where:

P = Pressure at any time

Q = Outgassing rate

S = Pumping speed of process removing gas (constant)

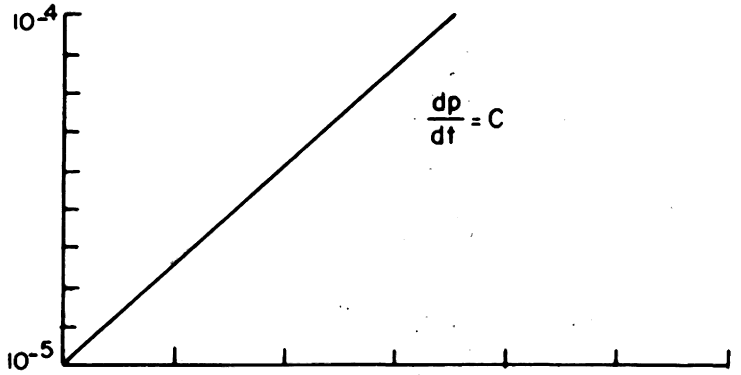
t = Time after valve closure

V = Volume of test chamber

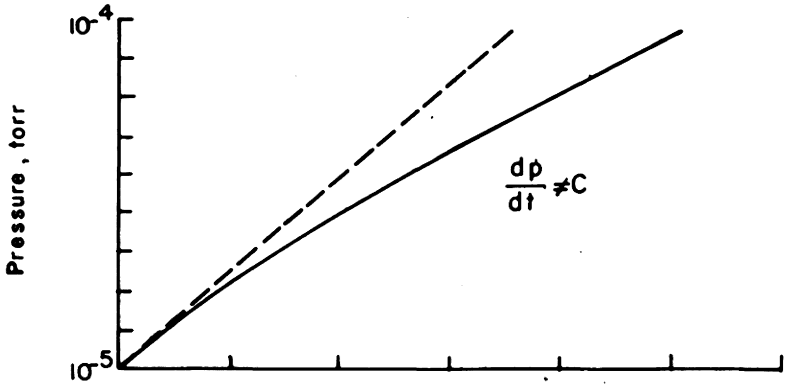
P_o = Initial pressure at t = o

A pressure-time plot of equation 6.7 is shown in figure 2(b). Also the theoretical pressure profile of figure 2(a) is replotted as a reference. Any process which removes gas at a constant rate will have a characteristic pressure profile similar to that of figure 2(b).

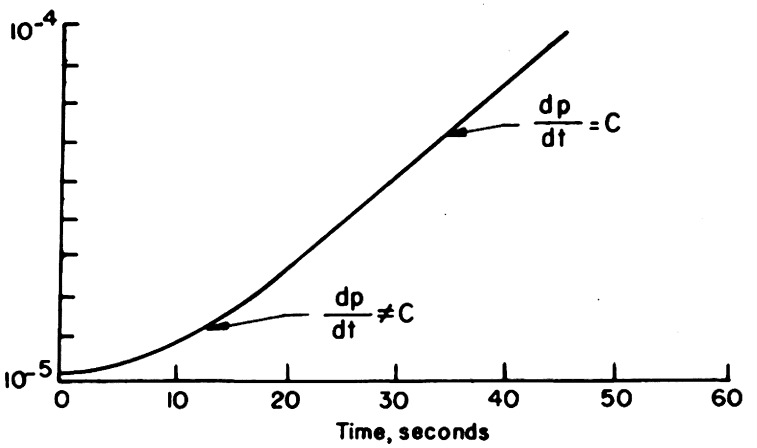
A third case occurs when a process takes place in the test volume that removes gas at a time dependent rate. Generally for the



(a) Zero pumping speed.



(b) Constant pumping speed.



(c) Pumping speed function of time.

Figure 2.- Theoretical rate of rise pressure traces.

processes known to exhibit a time dependent pumping, the pumping rate is a decreasing function of time; that is, the pumping speed decreases as time after valve closure increases. Figure 2(c) is a representation of a pressure-time trace that might be obtained in a rate of rise measurement if gas is being removed from the isolated volume at a time decreasing rate. Characteristics of the pressure curve for such a process are an increasing pressure-time slope with increasing time, and, after some finite time, a constant slope. Figures 2(a), 2(b), and 2(c) were constructed for a definite value of outgassing rate, initial pressure, and test volume. Variations of these parameters result in similar pressure profiles.

Many authors, when using the rate of rise technique, have used the shape of the pressure-time trace to conclude that particular processes are occurring or not occurring in the test volume. For example, Markley[20] concluded from pressure-time traces that adsorption of gas on the chamber walls was resulting in large errors in the observed outgassing rates. The pressure-time trace observed was similar to that of figure 2(c). This conclusion may be correct but it should not be based solely on the pressure-time behavior. Also, it is often concluded that a linear pressure-time behavior[21] after the valve is closed is indicative of the nonexistence of gas removal processes. This is not necessarily true as combinations of gas removal processes may result in a linear pressure-time behavior and correspondingly large quantities of gas may be pumped. The shape of the pressure profile during an outgassing measurement can be helpful

in determining the processes occurring, but additional information about the processes must be known before conclusions can be drawn.

Need for an Experimental Program

Because of the adsorption and gage pumping processes present in the use of the rate of rise method and the fact that in many cases the effects of these processes cannot be evaluated, the technique is treated in various ways in the literature. Many authors[22,23] in the discussion of the techniques available for measuring outgassing rates, discard the use of the technique due to the errors inherent in the dynamic nature of the technique. No mention is made of the magnitude of these errors or the methods available to reduce their effects. Other authors[24,25] use the rate of rise technique as if no errors existed. In one particular case the pressure-time trace was taken over a span of 100 minutes, and an average outgassing rate quoted. No comments were made concerning the large errors that exist in using such a large time span. A third group of authors, such as Greenwood[26], have recognized the existence of errors in the technique and have attempted to evaluate the technique over a limited range of leak rates. The literature treatment of the rate of rise technique is inadequate and inconsistent, therefore leading to skepticism in the application of the technique. There is a definite need for an experimental evaluation of the technique for a useful range of outgassing rates and for a variety of gases. The purpose of this thesis is to present such an evaluation of

the rate of rise technique and to discuss the minimization of the observed errors.

Summary

In the literature review several methods of measuring outgassing rates were discussed. Emphasis was placed on the rate of rise technique and it was established as a simple and easy technique to apply. The literature review showed that the documentation of the rate of rise technique is inadequate and inconsistent. Based on the literature review, an experimental program was initiated to experimentally evaluate the technique.

VII. THE INVESTIGATION

Application of the Rate of Rise Method

When using the rate of rise method to measure outgassing rates, equation 6.4 is applied to an isolated vacuum vessel and the rate of pressure rise in the vessel is measured. The application of equation 6.4 in the rate of rise measurement assumes that the outgassing rate is constant throughout the measurement period and that gas is not being removed from the test volume. Under these assumptions, a linear pressure time trace would result, and dP/dt can be replaced by $\Delta P/\Delta t$. In instances where nonlinear pressure traces occur, this same analogy can be made if at some arbitrary point on the pressure trace a tangent is drawn and then $\Delta P/\Delta t$ read from the tangent. (The selection of the tangent point will be discussed later.) Therefore, replacing dP/dt by $\Delta P/\Delta t$ equation 6.4 becomes

$$Q = V\,\Delta P/\Delta t \tag{7.1}$$

where:

Q = Outgassing rate, torr-liter-sec^{-1}

V = Volume of test chamber, liter

ΔP = Change in pressure during Δt, torr

Δt = Change in time, sec

The above equation is the equation from which the outgassing rate is calculated and will be referred to as the Rate of Rise Equation. The pressure-time behavior of the test volume during a measurement depends on the outgassing from the chamber walls, the leakage of atmospheric gas into the test volume, and the leakage of gas out of the test volume, as well as the outgassing from the test sample. Generally, the influence of these processes on the pressure behavior

are small as compared to the sample outgassing, but they should be evaluated for each research investigation to insure their minuteness. If the chamber is of good vacuum construction, then the leakage of atmospheric air into the test volume is negligible and can be verified by suitable leak checking of the chamber both before and after an experimental investigation. The use of a valve between the test chamber and the vacuum source which will seat reliably can eliminate the leakage of gas out of the test volume and this reliability can be verified by repeated leak checking of the valve closure. The chamber outgassing rate can be evaluated experimentally by applying the rate of rise method to the empty test volume. The outgassing rate of the test specimen should be the primary influence on the pressure behavior in the isolated test volume. With these considerations in mind, a research program was initiated to experimentally evaluate the rate of rise method of measuring outgassing rates.

Research Program

The purpose of the research program was to experimentally evaluate the rate of rise technique at room temperature for some common gases and for a practical range of outgassing rates. To accomplish this purpose, gases were allowed to leak into the isolated test volume at known rates, simulating typical specimen outgassing rates, and the resultant pressure rises recorded. Outgassing rates were calculated from the pressure rise data and compared to the known inleakage rates. Any discrepancies in the observed outgassing rates and the known inleakage rates were contributable to errors and

inaccuracies in the rate of rise technique. After a detailed investigation of the expected measurement errors, the effects of gage pumping and chamber adsorption were evaluated. A gas inlet system was constructed to leak a variety of test gases into the chamber with a range of flow rates of 5×10^{-6} torr-liter-sec^{-1} to 5×10^{-4} torr-liter-sec^{-1}. The gas inlet system will be discussed in detail in a later section of this thesis. Five flow rates were investigated: 5×10^{-6}, 1×10^{-5}, 5×10^{-5}, 1×10^{-4}, and 5×10^{-4} torr-liter-sec^{-1}. Three gases were investigated at each of the flow rates: helium, nitrogen, and methane. The selection and properties of the test gases will be discussed later.

A Bayard Alpert hot cathode ionization gage was used to measure the pressure rise in the test volume. One of the problems associated with the ionization gage is that the gage elements become contaminated after prolonged usuage and requires that the gage be periodically degassed. Since gage contamination depends on many factors and can not be defined, the gage was degassed prior to each pressure rise measurement. This provided a time in the test sequences at which the gage contamination would be similar. Since a degassed gage requires a finite time to return to equilibrium with its surroundings, it was necessary to evaluate the ability of the rate of rise technique to measure known outgassing rates at various times after gage degassing. The times investigated ranged from 5 minutes to 60 minutes after gage degassing. An additional part of the research program was to evaluate the effects of the reduction of gage emission current on the ability to measure a known leak rate. Gas leak rate combinations were

investigated for two emission currents: 10 ma (the manufacturer's recommended value) and 1 ma. For a given gas leak rate combination, a comparison of the rate of rise measurements at 10 ma and 1 ma will aid in the evaluation of the magnitude of gage pumping. To aid in the evaluation of adsorption effects in the application of the technique, test series at the leak rate of 1×10^{-5} torr-liter-sec^{-1} were taken across two pressure scales, 10^{-5} and 10^{-6} torr. If adsorption is occurring, it should have more effect at the lower pressure.

In summary, the research program consisted of a series of tests, each investigating the ability to measure a known leak rate at various times after gage degassing. The test series were conducted for various combinations of test gases, leak rates, gage emission currents, and pressures.

Test Apparatus

The test apparatus consisted of a vacuum chamber with associated vacuum equipment, a gas inlet system, and various types of instrumentation. The vacuum chamber used in this evaluation of the rate of rise technique was the chamber constructed by Greenwood[26] to measure the outgassing rates of two solid propellant fuels. Modifications were made to this test chamber in order that a test gas could be leaked into the test volume at a known rate, the gas load from valve closure could be minimized, and the ionization gage could be mounted in the manufacturer's recommended position. This particular vacuum chamber was selected for the evaluations because the chamber has proven reliable, incorporates a slide valve for

isolation of the test volume, and has sufficient pumping speed to reach the desired pressure levels. A photograph of this vacuum chamber is shown in figure 3. The vacuum source for the chamber is a 4-inch oil diffusion pump used in conjunction with a mechanical roughing pump. Incorporated in the diffusion pump is a water cooled cold cap and baffle, both of which reduce the amount of diffusion pump oil backstreaming. In addition, a liquid nitrogen cooled trap is located above the water baffle to further reduce backstreaming into the test volume. The liquid nitrogen level in the trap is maintained by an automatic level sensor. A 4-inch slide valve is located above the liquid nitrogen trap and is air operated to provide rapid isolation of the test volume from the vacuum source. The slide valve uses an elastomer o-ring as a seal and in order to minimize the gas load from the elastomer during valve closure, the valve seal was differentially pumped by a mechanical pump. All other chamber vacuum seals are metal seals, either aluminum or copper. The test volume is located above the slide valve and constructed of 347 stainless steel. The test volume of approximately 3.5 liters is composed of a highly polished stainless steel spool with a 10-inch stainless steel flange welded at the top. The mating 10-inch flange has three stainless steel instrumentation ports welded to it and a crushable aluminum o-ring forms the vacuum seal between the mating 10-inch flanges. Each instrumentation port has a 3-inch stainless steel flange welded to it and cooper gaskets form the seal for these flanges. Figure 4 is a photograph of the test volume with the instrumentation installed.

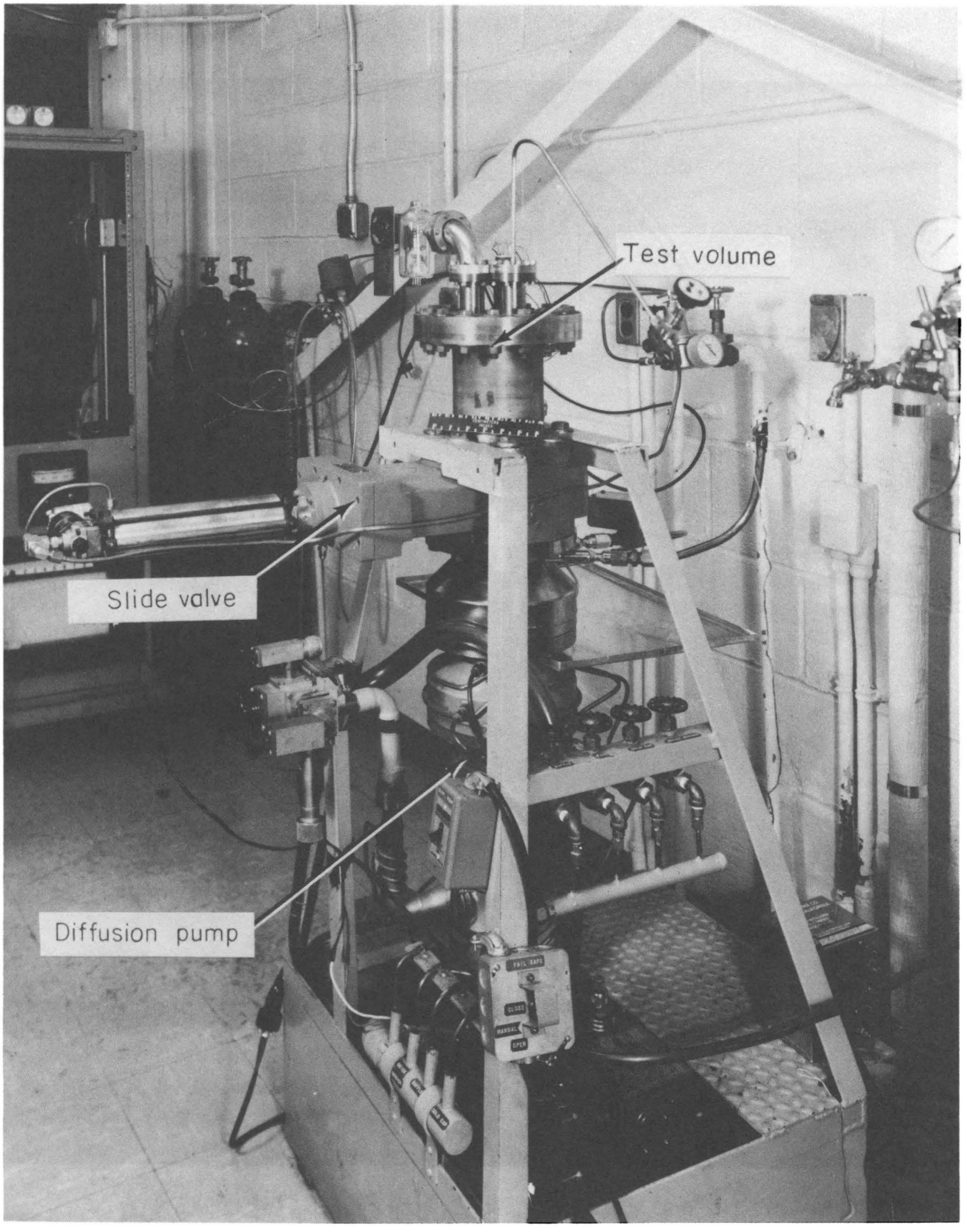


Figure 3.- 4-inch pumping station.

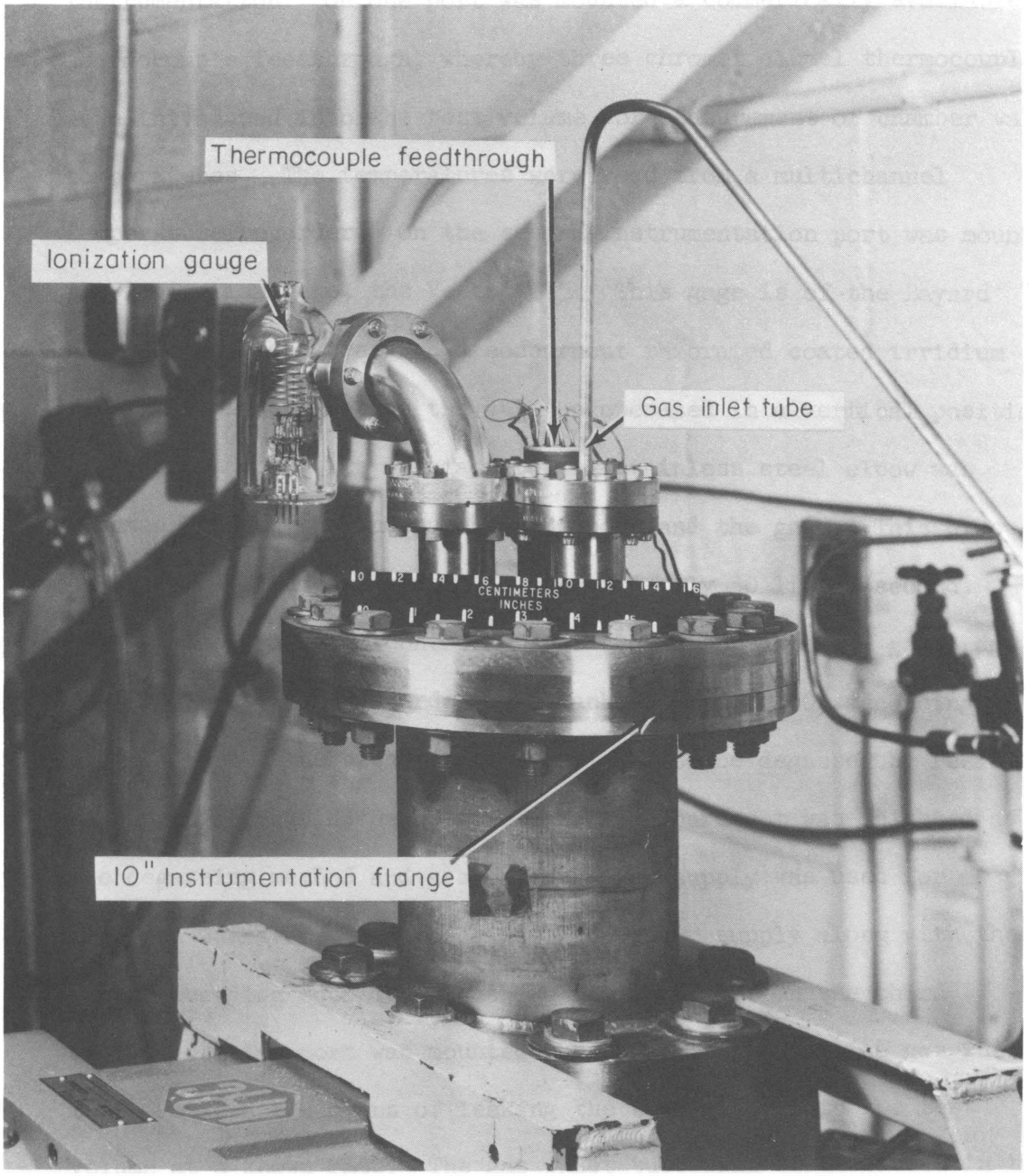


Figure 4.- Test volume.

The instrumentation ports were used for the mounting of test instrumentation. On one port was mounted a commerically available thermocouple feedthrough, whereby three chromel alumel thermocouples were introduced into the test volume for measurement of chamber wall temperatures. The temperatures were read from a multichannel temperature recorder. On the second instrumentation port was mounted the ionization gage, the Veeco RG75. This gage is of the Bayard Alpert design and features a nonburnout thiorated coated irridium filament. In order that the gage be operated in a vertical position as recommended by its manufacturer, a stainless steel elbow was mounted between the instrumentation port and the gage. This elbow has a calculated conductance of approximately 30 liters/second.

A standard Veeco controller was used to operate the gage, and the gage output was recorded on a Sanborn, Model 150, recording oscillograph. The Veeco gage is designed to be degassed by resistance heating of the grid; however, electron bombardment was selected as the degassing method and a separate power supply was used for electron bombardment of the gage. The power supply along with the other recording equipment is shown in figure 5. On the third instrumentation port was mounted the gas inlet tube. The gas inlet tube provided the means of leaking the test gases into the test volume at a known rate. The gas inlet tube will be discussed in the next section as a part of the gas inlet system.

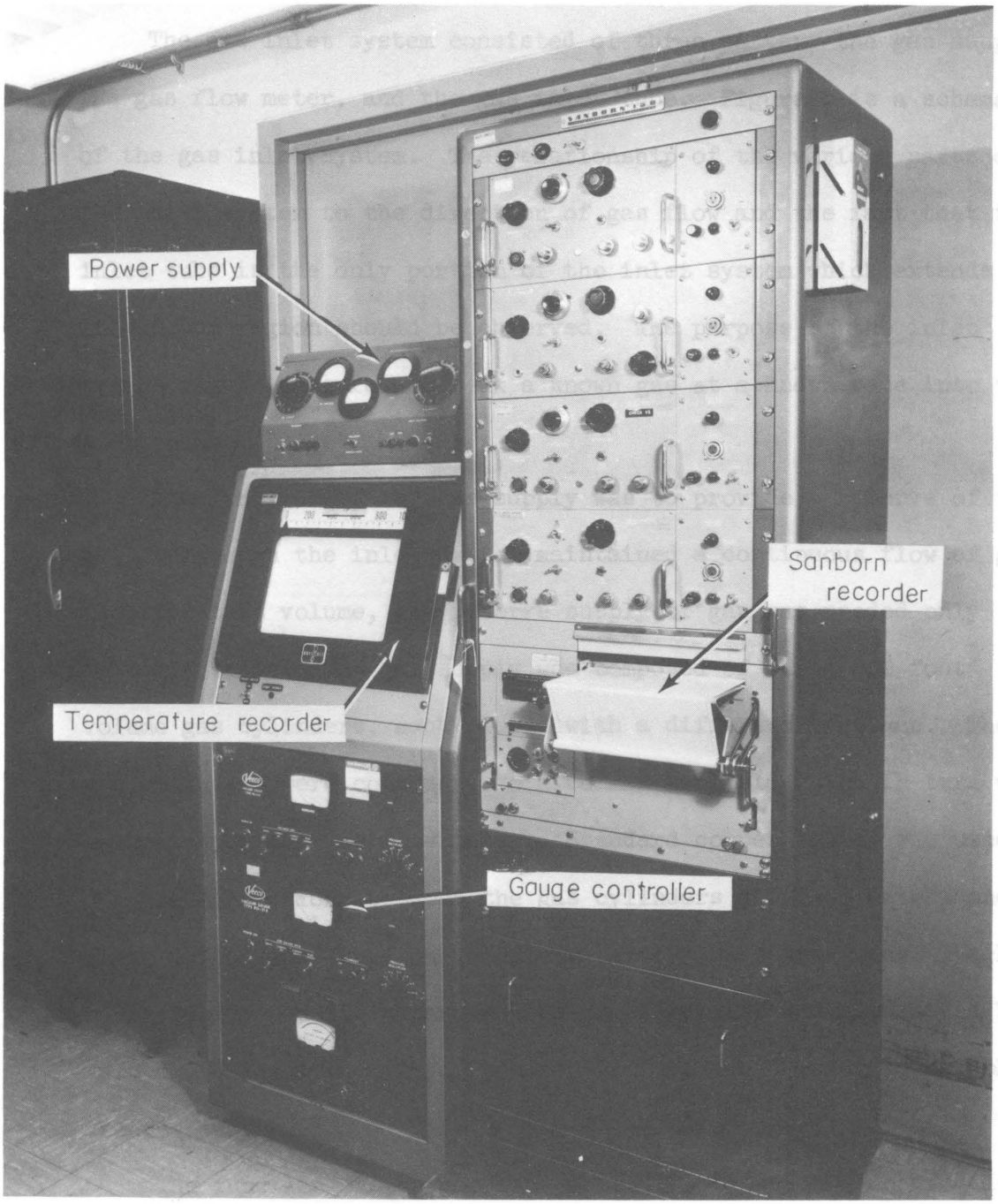


Figure 5.- Instrumentation.

Gas Inlet System

The gas inlet system consisted of three parts: the gas supply, the gas flow meter, and the gas inlet tube. Figure 6 is a schematic of the gas inlet system. The relationship of the various parts of the inlet system to the direction of gas flow and the fact that the inlet tube is the only portion of the inlet system which extends into the vacuum region should be observed. The purpose of the inlet system was to measure and leak a known gas at a known rate into the test volume.

The function of the gas supply was to provide a reserve of test gas. Although the inlet system maintained a continuous flow of gas into the test volume, the reserve supply of gas was needed only intermittently. The gas supply was composed of three 200 $foot^3$ volume gas cylinders, each filled with a different test gas. The gas cylinders were connected to a common manifold which in turn was connected to the gas flow meter. Standard copper tubing was used for the connections between the gas cylinders and flow meter, and these connections were operated at positive gage pressures. Each gas cylinder had its separate pressure regulator and each could be valved off independently of the others. Figure 7 is a photograph of the gas supply.

The function of the gas flow meter was to set the desired leak rate and to measure the leak rate set. The flow meter is commercially available and of constant pressure design, whereby the change in volume per unit time of a gas at constant pressure is a measure of the flow rate of gas into the test volume. The flow meter

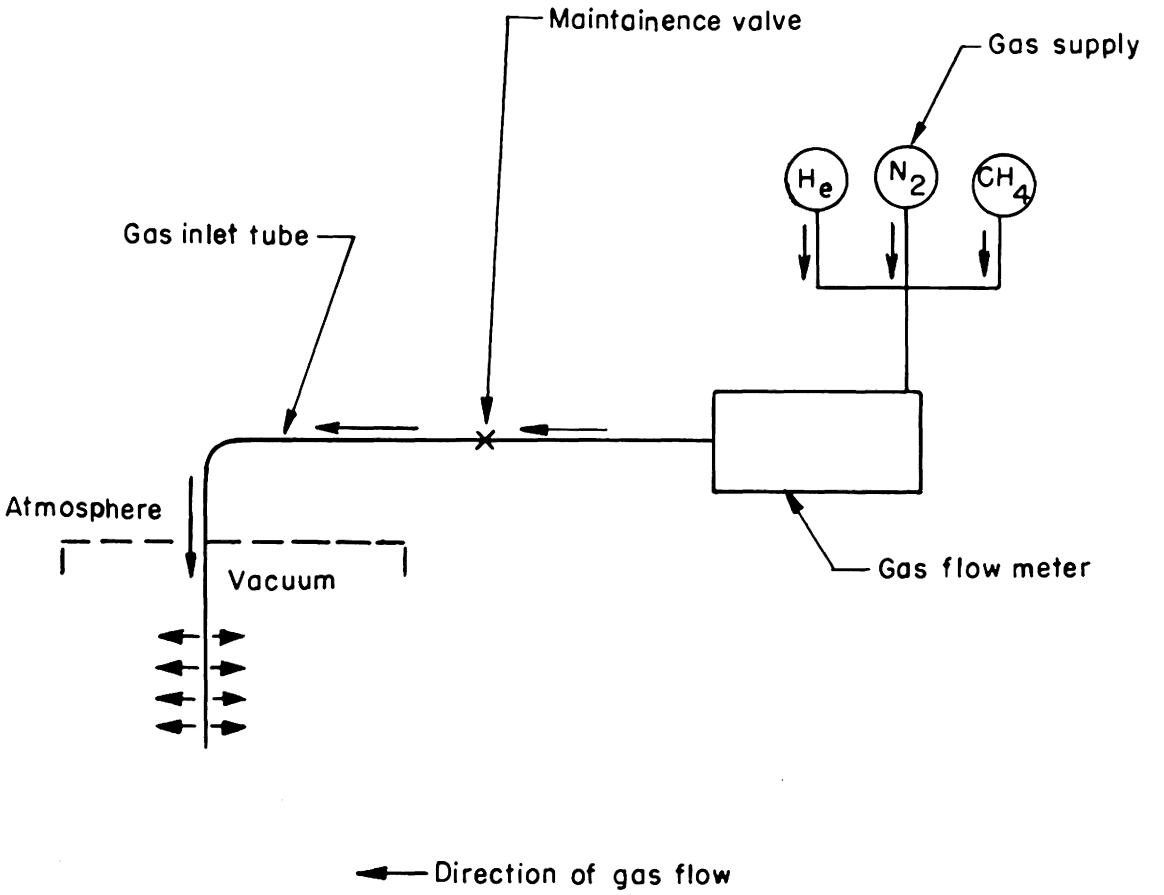


Figure 6.- Schematic of gas inlet system.

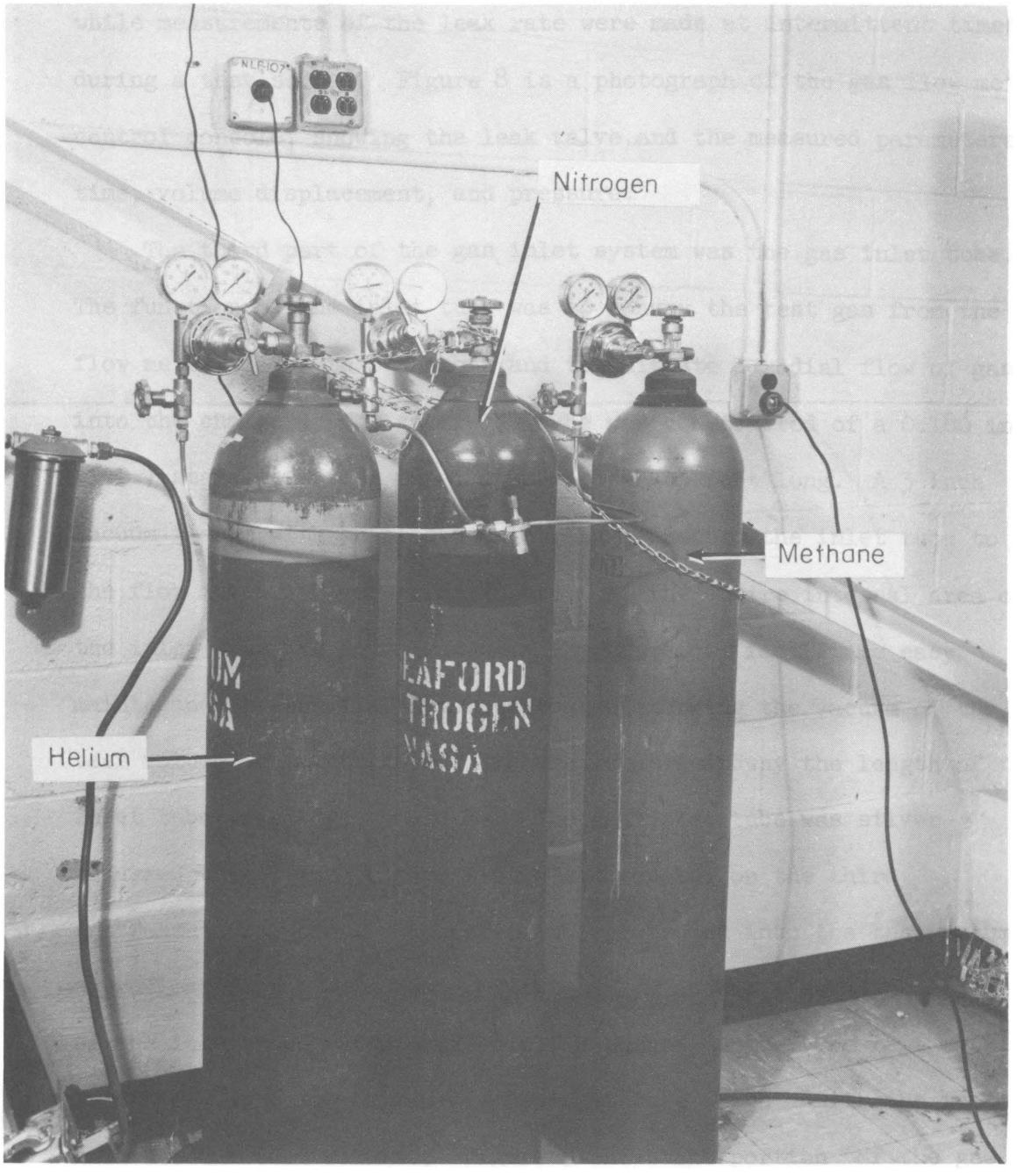


Figure 7.- Gas supply.

continuously leaked gas into the test volume at the desired rate, while measurements of the leak rate were made at intermittent times during a test series. Figure 8 is a photograph of the gas flow meter control console, showing the leak valve and the measured parameters of time, volume displacement, and pressure.

The third part of the gas inlet system was the gas inlet tube. The function of the inlet tube was to convey the test gas from the flow meter to the test chamber and to initiate a radial flow of gas into the chamber. The gas inlet tube was constructed of a 0.180 inch (I.D.) stainless steel tube, approximately 3 feet long. A 3 inch vacuum flange with an elastomer o-ring connected the inlet tube to the flow meter. Under normal operation, the entire internal area of the inlet tube was under vacuum. Therefore, to facilitate easy maintenance of the flow meter without disturbing the vacuum of the test volume, a maintenance valve was located midway the length of the inlet tube (see figs. 3 and 6). The gas inlet tube was silver soldered to a 3 inch vacuum flange and mounted on the third instrumentation port. The inlet tube protruded into the test volume approximately 9 1/2 inches. Of this 9 1/2 inches, the test section of the inlet tube was approximately 4 inches long. (The test section is defined as that portion of the inlet tube which admits the test gas into the test volume.) Figure 9 shows the portion of the gas inlet tube which protruded into the test volume. The test section is also shown in this photograph. The test section of the inlet tube was formed by sealing the open end of the tube with silver solder and drilling 0.022 inch diameter holes in the periphery of the 4-inch test

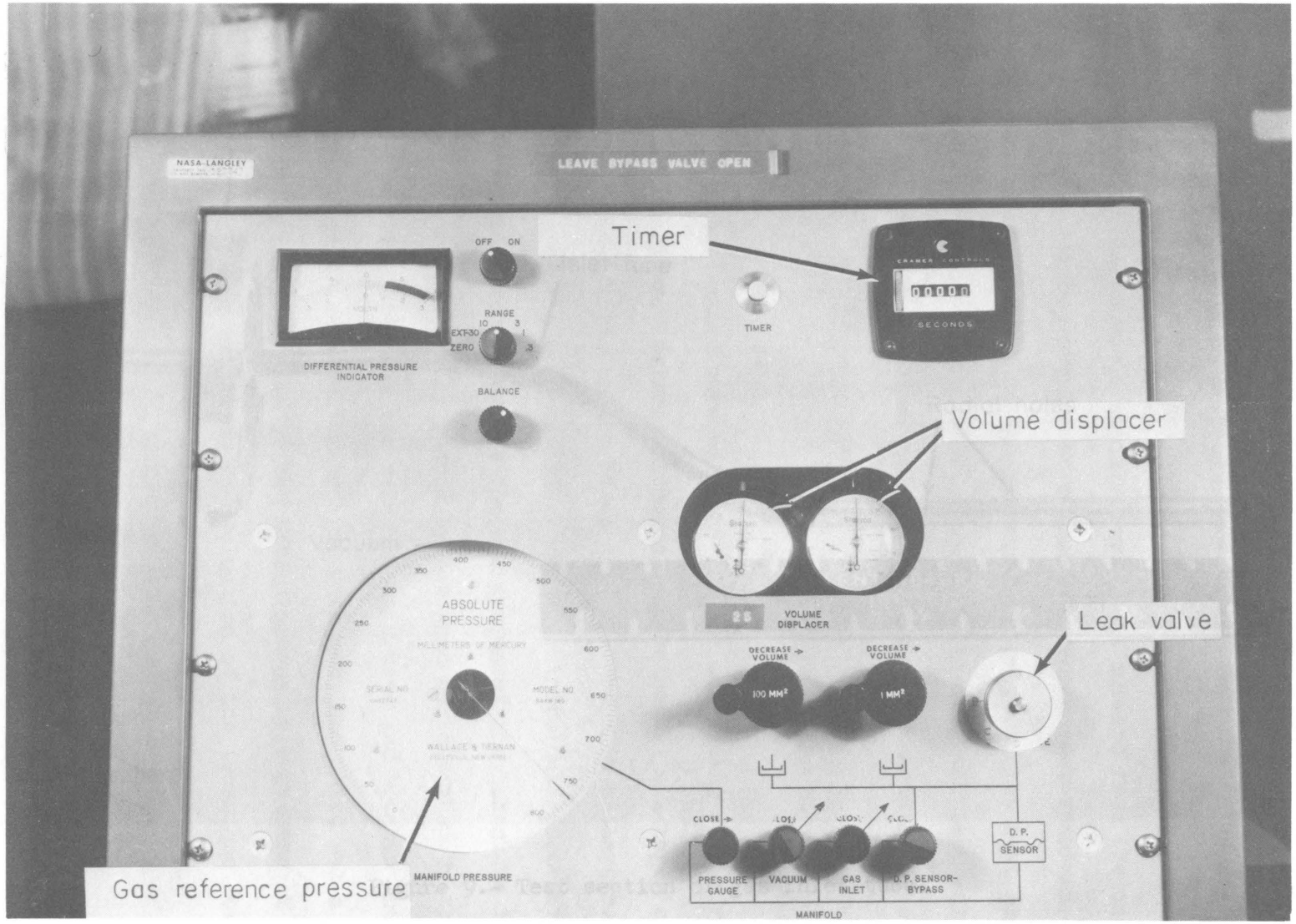


Figure 8.- Gas flow meter operating console.

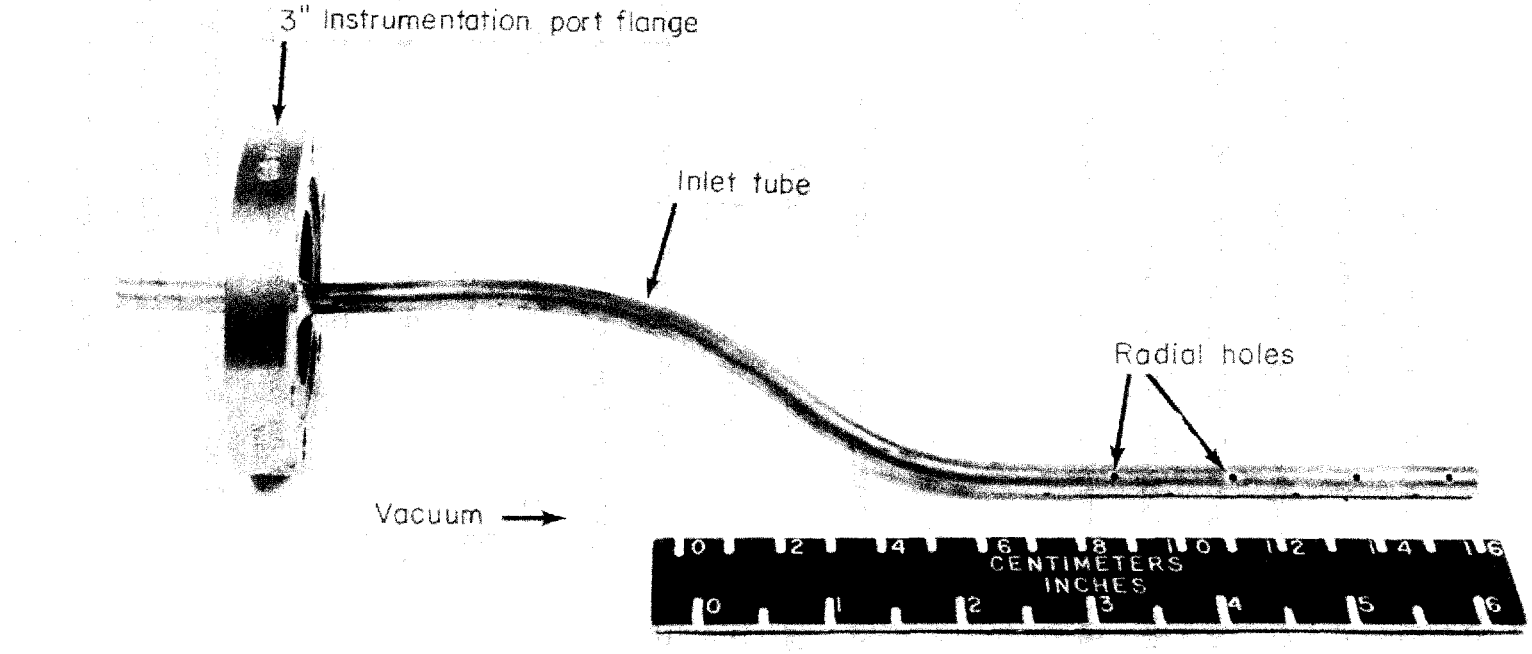


Figure 9.- Test section of gas inlet tube.

section. The holes were drilled 1/2 inch apart at 90° angles to each other. There was a total of 16 openings through which the gas flowed into the test volume. With the test section constructed in this manner, there was a radial flow of gas outward from the center of the test volume, similar to outgassing from a test specimen. With the inlet tube mounted on the instrumentation port, the test section of the inlet tube was in the approximate center of the test volume. Figure 10 shows a sketch of the test volume with the inlet tube in place.

Test Gases

Three test gases were selected for the evaluation of the rate of rise technique: helium, nitrogen, and methane. All three gases were selected, first, on the basis of their frequent appearance as outgassing products and, secondly, on their individual properties. Helium was selected to permit the evaluation of the technique for a gas which was not readily pumped by the gage nor adsorbed by the chamber walls. Nitrogen was selected as a test gas because the ionization gages were calibrated for nitrogen, and there was a sizeable quantity of data in the literature on gage pumping of nitrogen. Methane permitted an evaluation of the technique for a gas of hydrocarbon composition. The impurity contents of the test gases are given in tables 1, 2, and 3.

A list of test equipment is given in Appendix A.

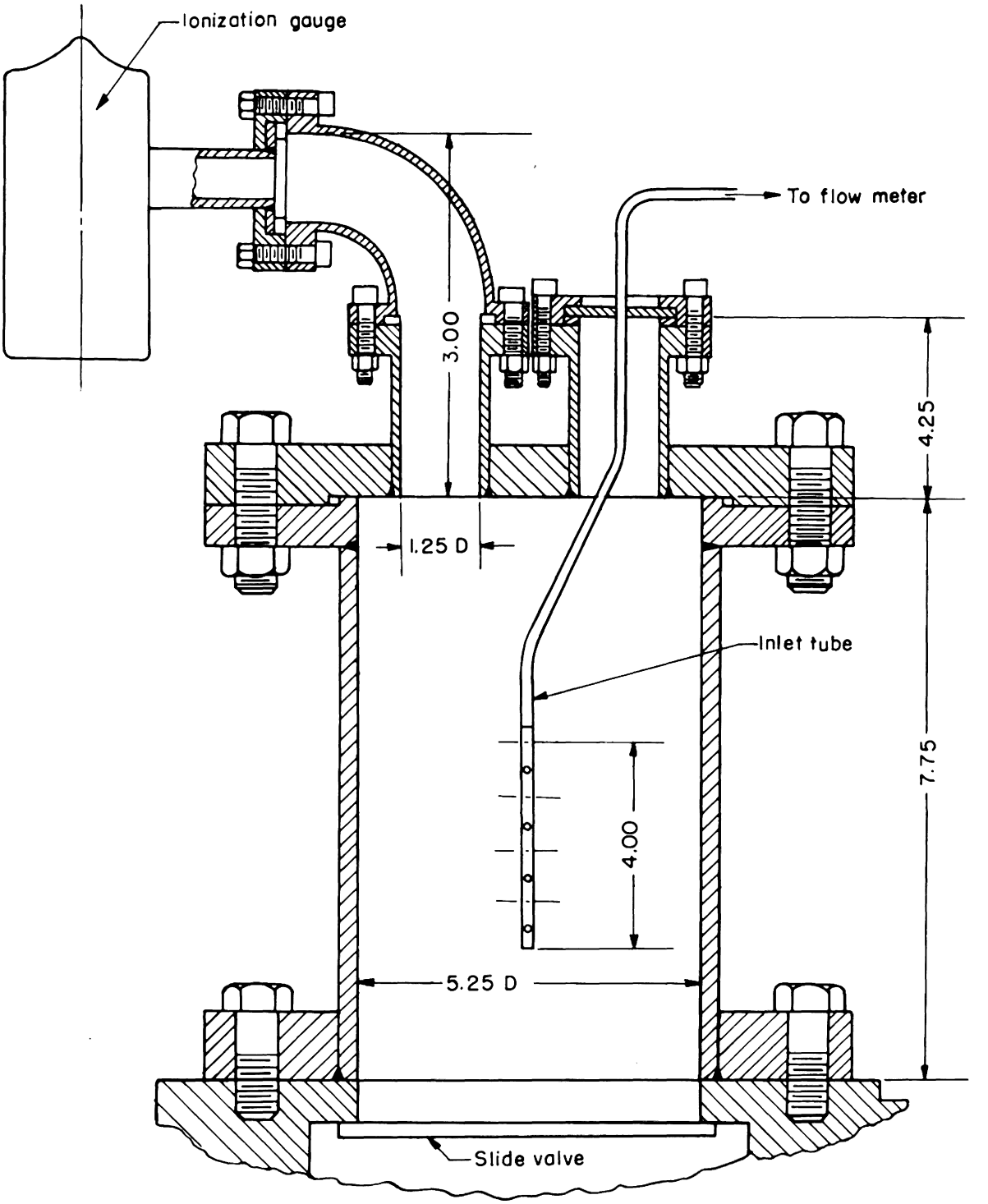


Figure 10.- Sketch of test volume. Dimensions in inches.

TABLE 1.- IMPURITY CONTENT OF HELIUM

Impurity gas	Impurity level
Air	20 ppm
Others	480 ppm

TABLE 2.- IMPURITY CONTENT OF NITROGEN (SEAFORD GRADE)

Impurity gas	Impurity level
Oxygen	20 ppm
Hydrogen	500 ppm
Argon	600 ppm
Water	0.002 percent by weight*

*At 55° C dew point

TABLE 3.- IMPURITY CONTENT OF METHANE (CT GRADE)

Impurity gas	Impurity level
Ethane	2200 ppm
Nitrogen	7900 ppm
Propane	160 ppm
Carbon dioxide	100 ppm
Oxygen + Argon	50 ppm
Water	3 ppm

Test Procedure

In applying the rate of rise technique to the test chamber, P in the Rate of Rise Equation, equation 7.1, becomes the pressure in the test volume, t becomes the time after valve closure, and V is the volume of the isolated test chamber.

Prior to the evaluation of the rate of rise technique, the test chamber, test gage, and gas inlet tube were baked for approximately

200 hours at 200° C. The purpose of the bake was to clean the chamber walls of contaminants that might have been present due to the installation of the test instrumentation. The bake cycle was repeated during the test program when required. After the bake, the vacuum chamber was completely leak checked with the Veeco MS-9 leak detector. The leak detector uses helium as a tracer gas and has the capability to detect leaks of the order of 10^{-10} torr-liter-sec^{-1}. Leak checks were made of the closure of the 4 inch slide valve and the gas flow meter. The test program was started only after the leak detector indicated that all leaks present were negligible; that is, less than 10^{-10} torr-liter-sec^{-1}. Additional leak checks of the facility were conducted at various times during the test program to insure that leaks had not developed in the chamber. After the chamber was leak checked, the liquid nitrogen trap was filled and remained operating throughout the evaluations.

A test series consisted of the evaluation of the ability of the rate of rise technique to measure a given leak rate as a function of time after the end of gage degassing. For a given test series the value of the leak rate, the test gas, the gage emission current, and the pressure scale remained constant. A total of thirty test series were conducted, investigating various combinations of leak rates, test gases, gage emission currents, and pressure scales. Each test series was composed of nine test runs, defined as follows:

Test run 1 was a rate of rise measurement of the outgassing rate of the empty test volume prior to inleakage of the test gas.

Test runs 2 through 9 were rate of rise measurements of the test volume at 5, 10, 15, 20, 30, 40, 50, and 60 minutes, respectively, after the end of gage degassing with a test gas being leaked into the test chamber at a known rate.

A test run consisted of the sequence of events necessary to measure the outgassing rate of the known leak at the specified time after the end of gage degassing. Each test series consisted of the nine test runs as defined above. The start of a test run was designated as the beginning of electron bombardment (degassing) of the ionization gage. The gage was electron bombarded for 10 minutes by applying approximately 6 volts to the filament and 800 volts potential between the filament and grid. A grid current of 60 to 100 milliamps resulted, depending upon the cleanliness of the gage. After the end of gage bombardment, a period of time existed in which the gage was allowed to cool. This time period ranged from 5 minutes to 60 minutes and was variable depending upon the test run being conducted. At the end of this time, the slide valve was closed. During this gage cooling period, several events took place. From the flow meter, a measurement of the leak rate of test gas into the test volume was made. Approximately two minutes from the end of the gage cooling period and in preparation for the rate of rise measurement, the liquid nitrogen trap was manually overfilled and the gage emission current set to test value. The gage was normally operated at 10 ma emission current except during a rate of rise measurement where it was operated at either 10 or 1 ma. At the prescribed time after gage

degassing the slide valve was closed, isolating the test volume. The rise of pressure in the test volume was measured with the Veeco ionization gage and a pressure-time history was obtained. The outgassing rate was calculated by multiplying the volume of the test chamber by the appropriate $\Delta P/\Delta t$ (change in pressure/change in time) as measured from the oscillograph trace. After the desired pressure rise was obtained, the slide valve was opened, designating the end of a test run.

For each test series, test run 1 was taken first in as much as the purpose of this run was to evaluate the outgassing rate of the test chamber prior to leaking in the test gas. Test runs 1 were conducted at 20 minutes after the end of gage degassing and with a gage emission current of 1 ma. For test run 1, the pressure in the test volume was allowed to rise three decades in pressure, following closure of the slide valve, from 10^{-7} to 10^{-4} torr. Approximately 10 minutes were required to obtain the 3 decade pressure rise. Figure 11 shows a pressure time history for a typical test run number 1. The three pressure plots shown in this figure are from one pressure trace taken for the empty system from 10^{-7} torr to 10^{-4} torr. The outgassing rate of the empty system was typically 1×10^{-6} torr-liter-sec^{-1} as read from the 10^{-7} torr pressure trace. The pressure-time behavior shown in figure 11 for the empty system will be discussed in detail in the Discussion and Results section of this thesis.

After the completion of test run 1, the test leak rate was set on the gas flow meter and test gas was leaked into the test volume.

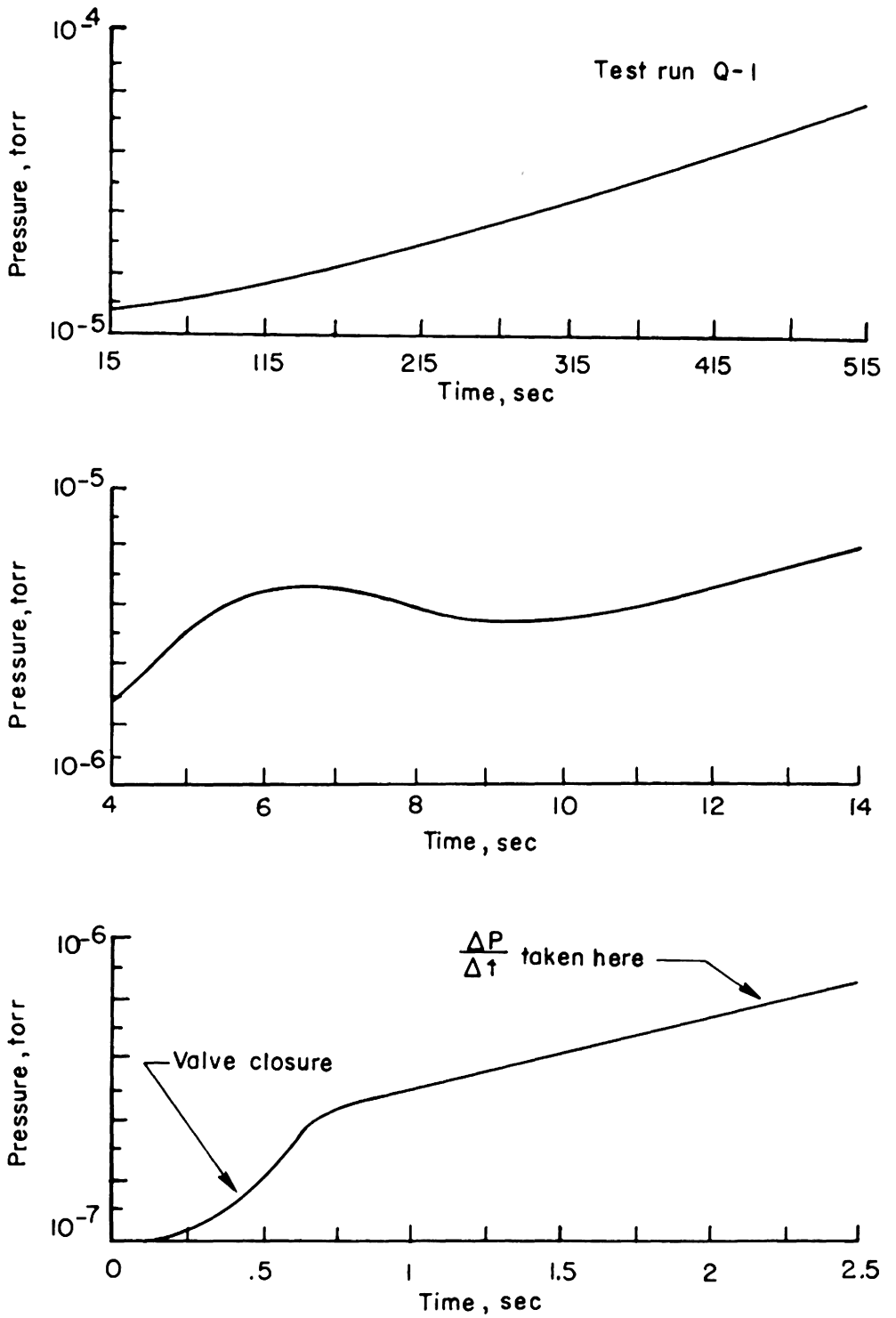
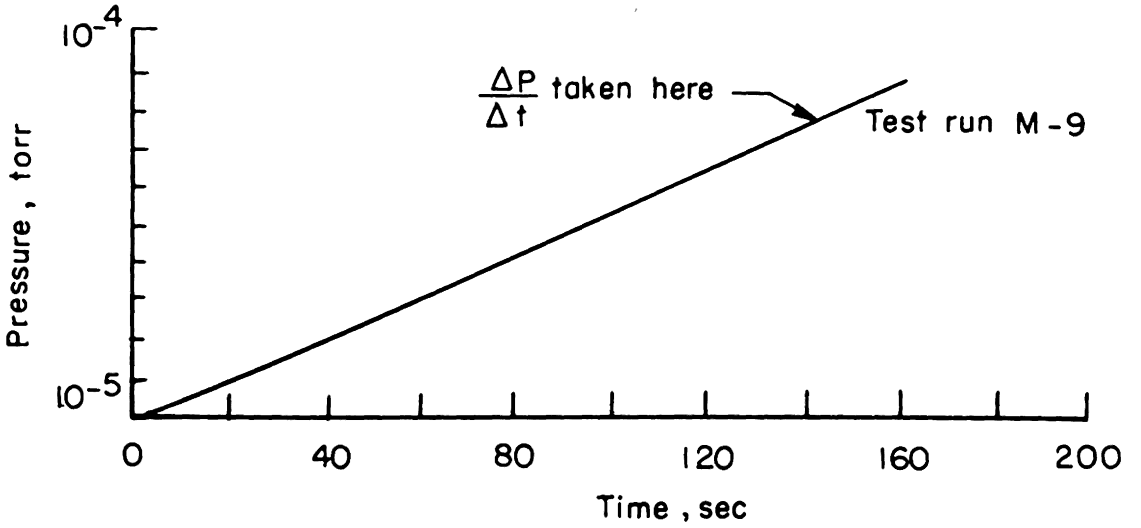


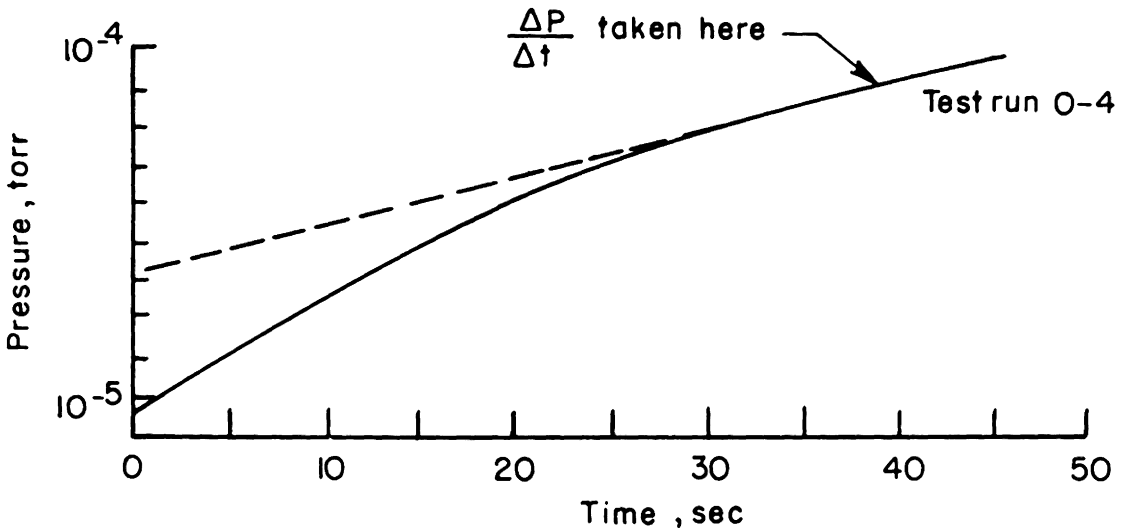
Figure 11.- Typical pressure time histories for test run 1 (empty system).

Approximately 45 minutes were required to set and stabilize the desired leak rate. Once the leak rate was set, the test gas continuously leaked into the test volume with only slight adjustments to the flow meter being necessary to maintain the desired leak rate. After the leak rate was stabilized, the remaining eight test runs were conducted. No numerical sequence was established for the completion of the remaining test runs. During these runs, the pressure was allowed to rise one decade, requiring from 3 to 120 seconds for the decade pressure rise depending on the test series being conducted. Thirty minutes were allowed between the end of a test run and the start of the next run. During this time the test volume was pumped to its equilibrium pressure determined by the test gas and the magnitude of the leak rate. After the completion of the last test run of a test series, the leak rate of gas into the chamber was cut off and the chamber was pumped for approximately 14 hours prior to the start of the next test series. In general, one test series was conducted each day.

Three general types of pressure-time traces were observed during the rate of rise measurements of the gas being leaked into the test volume. These pressure traces are shown in figures 12(a), (b), and (c). Figure 12(a) is a linear trace which was typically observed for the test series in which helium was the test gas. Figure 12(b) shows a nonlinear trace. This trace was the least linear pressure-time history observed for the test gases and was characteristic of only a few of the nitrogen and methane test series. The pressure trace observed for the majority of the test series is shown in

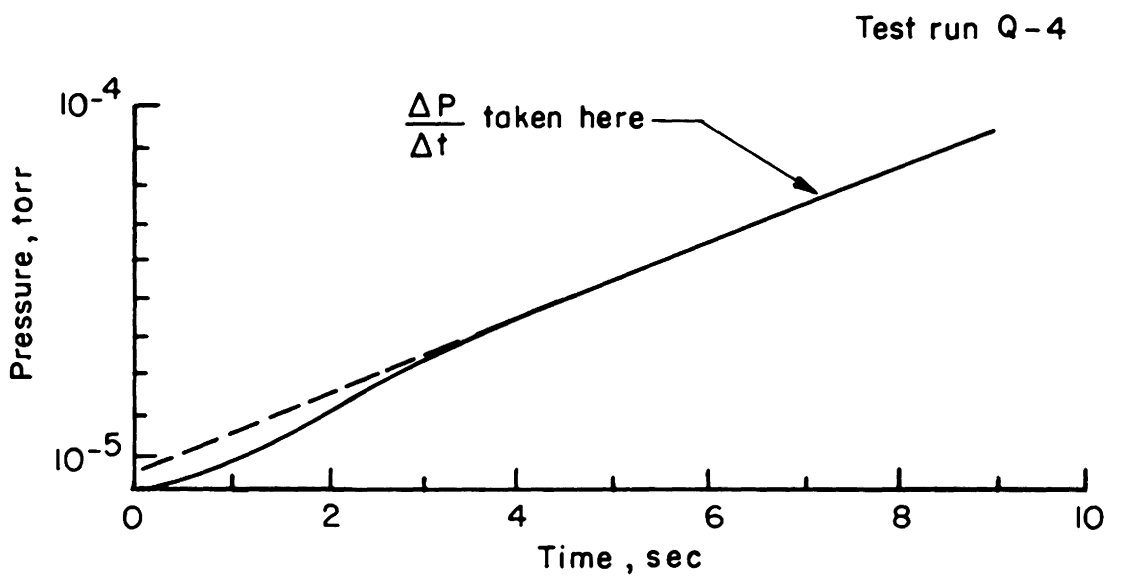


(a) Linear.



(b) Nonlinear.

Figure 12.- Typical pressure time histories.



(c) Nonlinear.

Figure 12.- Concluded.

figure 12(c). This trace is nonlinear initially, but becomes linear in the later portion. These pressure traces will be discussed in detail in the Discussion and Results section of the thesis.

Sample Calculations

In order to show the data reduction techniques used in the conversion of the observed experimental quantities into the outgassing rates and leak rates necessary to evaluate the rate of rise technique, a complete set of calculations will be presented for one run of Test Series Q. The characteristic parameters for Test Series Q were

Test Gas - Nitrogen

Nitrogen leak rate - 5×10^{-5} torr-liter-sec^{-1}

Gage emission current - 10 milliamps

Pressure scale - 10^{-5} torr

The sample calculations will be divided into three sections: the calculation of the leak rate of test gas into the test chamber, the calculation of the empty system outgassing rate, and the calculation of the outgassing rate as observed by the rate of rise technique.

Leak rate of test gas.- The leak rate of test gas into the test chamber was controlled and measured with the Varian constant pressure gas flow meter. The equation used in the calculation of the leak rate was

$$L = \frac{P_o A(x_2 - x_1) 527.67}{(t_2 - t_1) T_R} \times 10^{-6} \qquad (7.2)$$

where:

L = Leak rate, torr-liter-sec^{-1}

P_o = Reference pressure, torr

A = Area of displacement piston, mm^2

x_1 = Initial displacement of piston, mm

x_2 = Final displacement of piston, mm

t_1 = Time corresponding to x_1, sec

t_2 = Time corresponding to x_2, sec

527.67 = Leak rate reference temperature, ^oR (20^o C)

T_R = Room temperature, ^oR

This equation is developed in the Appendix B of the thesis. The experimental data for Run Q-4 as read from the flow meter was

P_o = 500 torr

A = 1 mm^2

x_1 = 0 mm

x_2 = 20 mm

t_1 = 0 sec

t_2 = 197.7 sec

T_R = 72^o F (532^o R)

substituting these quantities into equation 7.2 gave a leak rate of

$$L = \frac{(500)\ (1)\ (20-0)\ (527.67)}{(197.7-0)\ (532)} \times 10^{-6}$$

$$L = 5.02 \times 10^{-5} \text{ torr-liter-sec}^{-1}$$

This method was used to calculate the leak rate of the test gas for each test run. As seen from equation 7.2, the calculation of the leak rate is independent of the type of test gas.

Empty system outgassing rate.- The empty system outgassing rate will be calculated in terms of equivalent nitrogen units; that is, the gage sensitivity used in the calculation will be that for nitrogen gas. The equation used is that of 7.1

$$Q = V \frac{\Delta P}{\Delta t} \tag{7.3}$$

where:

Q = Outgassing rate of empty chamber, torr-liter-sec^{-1}

P = Pressure in isolated test volume, torr

t = Time after valve closure, sec

V = Volume of isolated test chamber, liter

The volume of the test chamber was measured by a water fill technique, and

$$V = V_G + V_C$$

where:

V = Volume of isolated test chamber, liter

V_G = Volume of test gage, liter

V_C = Volume of test chamber minus test gage, liter

$$V = 0.23 + 3.57$$

$$V = 3.8 \text{ liter}$$

This volume was constant throughout the evaluations and was used for all rate of rise calculations. The rate of pressure rise $\Delta P/\Delta t$ was read from pressure time trace of Run Q-1 (fig. 11) and was found to be

$$\frac{\Delta P}{\Delta t} = 2.5 \times 10^{-7} \text{ torr-sec}^{-1}$$

substituting the corresponding values into equation 7.3

$$Q = 3.8\ (2.5 \times 10^{-7})$$

$$Q = 9.5 \times 10^{-7}\ \text{torr-liter-sec}^{-1}$$

This outgassing rate is that rate observed for the empty chamber using the manufacturer's assumed gage sensitivity constant. Correcting this value for gage calibration

$$Q_{e.s.} = Q\ s_m/s_n \tag{7.4}$$

where:

$Q_{e.s.}$ = Corrected outgassing rate, torr-liter-sec^{-1}

Q = Observed outgassing rate from equation 7.4, torr-liter-sec^{-1}

s_m = Manufacturer's assumed sensitivity, torr^{-1}

s_n = Calibrated sensitivity for nitrogen, torr^{-1}

The manufacturer's sensitivity, s_m, was 10; calibration of the gage used in Test Q for nitrogen by a conductance method produced a value of s_n of 8.06. Substituting these values into equation 7.5

$$Q_{e.s.} = (9.5 \times 10^{-7})\ \frac{10}{8.06}$$

$$Q_{e.s.} = 1.18 \times 10^{-6}\ \text{torr-liter-sec}^{-1}$$

This is the outgassing rate of the empty system for Test Series Q, in equivalent nitrogen units.

Outgassing rate of test gas.- The outgassing rate of the test gas being leaked into the chamber will be calculated in terms of equivalent test gas units; that is, for helium, the outgassing rate will be in helium units. Equation 7.1 will again be used

$$Q = V \frac{\Delta P}{\Delta t} \tag{7.5}$$

where:

Q = Outgassing rate of empty chamber plus the leak rate of test gas into the test volume, torr-liter-sec^{-1}

P = Pressure in isolated test volume, torr

t = Time after valve closure, sec

V = Volume of isolated test chamber, liter

The volume was again 3.8 liters. The rate of pressure rise, $\Delta P/\Delta t$, was read from the pressure-time trace for Run Q-4 as indicated in figure 12(c). This was read as

$$\Delta P/\Delta t = 9.48 \times 10^{-6} \text{ torr-sec}^{-1}$$

Substituting the corresponding values in equation 7.5

$$Q = 3.8 \left(9.48 \times 10^{-6}\right)$$

$$Q = 3.64 \times 10^{-5} \text{ torr-liter-sec}^{-1}$$

Again, this outgassing rate is that observed by using the manufacturer's gage sensitivity constant. Correcting this outgassing for gage calibration as was done earlier (eq. 7.4)

$$Q_C = \left(3.64 \times 10^{-5}\right) \frac{10}{8.06}$$

$$Q_C = 4.48 \times 10^{-5} \text{ torr-liter-sec}^{-1}$$

Now this outgassing rate was that combined rate of the chamber and test gas, in nitrogen units as the gage was calibrated for nitrogen. To obtain the outgassing rate for only the test gas, the empty system must be substracted; that is,

$$Q_G = Q_C - Q_{e.s.} \tag{7.6}$$

where:

Q_G = Outgassing rate of test gas, nitrogen torr-liter-sec^{-1}

Q_C = Outgassing rate of empty system and test gas, nitrogen torr-liter-sec^{-1} (eq. 7.5)

$Q_{e.s.}$ = Outgassing rate of empty system, nitrogen torr-liter-sec^{-1} (eq. 7.4)

Substituting into equation 7.6

$$Q_G = (4.48 \times 10^{-5}) - (1.18 \times 10^{-6})$$

$$Q_G = 4.36 \times 10^{-5} \text{ torr-liter-sec}^{-1}$$

Now, Q_G, is the outgassing rate of the test gas in nitrogen units. Therefore, as a last step to obtain the outgassing rate of the test gas in the test gas units, Q_G must be corrected for the sensitivity of the gage for the test gas being investigated.

$$Q_L = Q_G / s_x / s_n \tag{7.7}$$

where

Q_L = Outgassing rate of the test gas, torr-liter-sec^{-1} (test gas units)

Q_G = Outgassing rate of the test gas in nitrogen units, torr-liter-sec^{-1} (eq. 7.6)

s_x/s_n = Ratio of sensitivity of ionization gage of test gas X as compared to calibrated sensitivity for nitrogen

The ratio of the sensitivity for the test gases to nitrogen were

Gas	s_x/s_n	Source
Nitrogen	1	Calibration
Helium	.2	Reference 27
Methane	1.70	Calibration

Since nitrogen was the test gas for test series Q, equation 7.7 becomes

$$Q_L = 4.36 \times 10^{-5}/1$$

$$Q_L = 4.36 \times 10^{-5} \text{ torr-liter-sec}^{-1}$$

The outgassing rate Q_L can be compared directly to the leak rate, L, calculated from equation 7.2 and from this comparison, the ability of the rate of rise technique to measure the given leak rate at prescribed conditions is evaluated.

Test Results

The results of this thesis evaluation of the rate of rise technique of measuring outgassing rates are presented in the following pages in both tabular and graphic form. Thirty test series are

presented in tabular form, ten for each of the three test gases. For each test gas the leak rates of 5×10^{-6} and 5×10^{-5} torr-liter-sec^{-1} are investigated for two values of gage emission current. The leak rates of 1×10^{-4} and 5×10^{-4} torr-liter-sec^{-1} are investigated for only one gage emission current and the leak rate of 1×10^{-5} torr-liter-sec^{-1} is investigated for two emission currents and two pressure scales. Of the ten test series presented in tabular form for each of the test gases, only eight are shown in graphic form as two of the four test series at the leak rate of 1×10^{-5} torr-liter-sec^{-1} are omitted from the data plots. (The omitted test series are tests AM, AN, AJ, AL, O, and P.) On each figure the broken line represents the value of the knonw leak rate of test gas into the test volume as determined from the gas flow meter measurements. The average leak rate shown in the tables is the arithmetic mean of the eight leak rates recorded for the test series.

TABLE 4.- RESULTS OF TEST SERIES F

Test gas: Helium

Average leak rate: 5.34×10^{-6} torr-liter-sec^{-1}

Emission current: 10 ma

Pressure scale: 10^{-6} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			7.38×10^{-7}
2	5	5.36×10^{-6}	3.11×10^{-6}
3	10	5.36×10^{-6}	3.79×10^{-6}
4	15	5.33×10^{-6}	3.31×10^{-6}
5	20	5.34×10^{-6}	3.61×10^{-6}
6	30	5.29×10^{-6}	2.74×10^{-6}
7	40	5.35×10^{-6}	2.73×10^{-6}
8	50	5.42×10^{-6}	2.81×10^{-6}
9	60	5.38×10^{-6}	2.56×10^{-6}

TABLE 5.- RESULTS OF TEST SERIES GG

Test gas: Helium

Average leak rate: 5.06×10^{-6} torr-liter-sec^{-1}

Emission current: 1 ma

Pressure scale: 10^{-6} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			5.96×10^{-7}
2	5	5.03×10^{-6}	5.69×10^{-6}
3	10	5.08×10^{-6}	5.37×10^{-6}
4	15	5.05×10^{-6}	4.22×10^{-6}
5	20	5.13×10^{-6}	4.62×10^{-6}
6	30	5.00×10^{-6}	5.17×10^{-6}
7	40	5.10×10^{-6}	5.34×10^{-6}
8	50	5.08×10^{-6}	5.17×10^{-6}
9	60	5.02×10^{-6}	4.98×10^{-6}

TABLE 6.- RESULTS OF TEST SERIES AM

Test gas: Helium

Average leak rate: 1.04×10^{-5} torr-liter-sec^{-1}

Emission current: 10 ma

Pressure scale: 10^{-6} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			8.55×10^{-7}
2	5	1.04×10^{-5}	9.22×10^{-6}
3	10	1.04×10^{-5}	9.94×10^{-6}
4	15	1.05×10^{-5}	1.04×10^{-5}
5	20	1.05×10^{-5}	1.00×10^{-5}
6	30	1.04×10^{-5}	1.02×10^{-5}
7	40	1.02×10^{-5}	1.11×10^{-5}
8	50	1.04×10^{-5}	1.09×10^{-5}
9	60	1.04×10^{-5}	1.06×10^{-5}

TABLE 7.- RESULTS OF TEST SERIES AN

Test gas: Helium

Average leak rate: 9.77×10^{-6} torr-liter-sec^{-1}

Emission current: 1 ma

Pressure scale: 10^{-6} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			8.77×10^{-7}
2	5	9.83×10^{-6}	1.33×10^{-5}
3	10	9.82×10^{-6}	1.58×10^{-5}
4	15	9.83×10^{-6}	1.59×10^{-5}
5	20	9.73×10^{-6}	1.56×10^{-5}
6	30	9.80×10^{-6}	1.44×10^{-5}
7	40	9.70×10^{-6}	9.70×10^{-6}
8	50	9.75×10^{-6}	1.04×10^{-5}
9	60	9.70×10^{-6}	8.60 × 10-6

TABLE 8.- RESULTS OF TEST SERIES M

Test gas: Helium

Average leak rate: 1.06×10^{-5} torr-liter-sec^{-1}

Emission current: 10 ma

Pressure scale: 10^{-5} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			9.16×10^{-7}
2	5	1.06×10^{-5}	8.92×10^{-6}
3	10	1.05×10^{-5}	8.42×10^{-6}
4	15	1.06×10^{-5}	7.22×10^{-6}
5	20	1.08×10^{-5}	7.92×10^{-6}
6	30	1.04×10^{-5}	7.22×10^{-6}
7	40	1.06×10^{-5}	8.62×10^{-6}
8	50	1.06×10^{-5}	8.22×10^{-6}
9	60	1.07×10^{-5}	8.62×10^{-6}

TABLE 9.- RESULTS OF TEST SERIES N

Test gas: Helium

Average leak rate: 1.01×10^{-5} torr-liter-sec^{-1}

Emission current: 1 ma

Pressure scale: 10^{-5} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			8.81×10^{-7}
2	5	1.00×10^{-5}	1.07×10^{-5}
3	10	1.00×10^{-5}	1.05×10^{-5}
4	15	1.00×10^{-5}	1.07×10^{-5}
5	20	1.00×10^{-5}	1.06×10^{-5}
6	30	1.02×10^{-5}	1.07×10^{-5}
7	40	1.01×10^{-5}	1.00×10^{-5}
8	50	1.00×10^{-5}	1.07×10^{-5}
9	60	1.01×10^{-5}	1.11×10^{-5}

TABLE 10.- RESULTS OF TEST SERIES S

Test gas: Helium

Average leak rate: 5.25×10^{-5} torr-liter-sec^{-1}

Emission current: 10 ma

Starting pressure: 10^{-5} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			1.10×10^{-6}
2	5	5.35×10^{-5}	4.65×10^{-5}
3	10	5.25×10^{-5}	4.41×10^{-5}
4	15	5.20×10^{-5}	4.65×10^{-5}
5	20	5.22×10^{-5}	4.43×10^{-5}
6	30	5.25×10^{-5}	4.55×10^{-5}
7	40	5.23×10^{-5}	4.36×10^{-5}
8	50	5.30×10^{-5}	4.43×10^{-5}
9	60	5.18×10^{-5}	4.57×10^{-5}

TABLE 11.- RESULTS OF TEST SERIES T

Test gas: Helium

Average leak rate: 5.15×10^{-5} torr-liter-sec^{-1}

Emission current: 1 ma

Pressure scale: 10^{-5} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			1.13×10^{-6}
2	5	5.12×10^{-5}	5.88×10^{-5}
3	10	5.16×10^{-5}	5.82×10^{-5}
4	15	5.16×10^{-5}	5.53×10^{-5}
5	20	5.18×10^{-5}	5.88×10^{-5}
6	30	5.15×10^{-5}	5.20×10^{-5}
7	40	5.08×10^{-5}	5.69×10^{-5}
8	50	5.18×10^{-5}	5.11×10^{-5}
9	60	5.15×10^{-5}	5.43×10^{-5}

TABLE 12.- RESULTS OF TEST SERIES Z

Test gas: Helium

Average leak rate: 1.05×10^{-4} torr-liter-sec^{-1}

Emission current: 1 ma

Pressure scale: 10^{-4} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			9.94×10^{-7}
2	5	1.03×10^{-4}	1.40×10^{-4}
3	10	1.06×10^{-4}	1.34×10^{-4}
4	15	1.03×10^{-4}	1.33×10^{-4}
5	20	1.04×10^{-4}	1.39×10^{-4}
6	30	1.05×10^{-4}	1.39×10^{-4}
7	40	1.04×10^{-4}	1.42×10^{-4}
8	50	1.05×10^{-4}	1.39×10^{-4}
9	60	1.05×10^{-4}	1.41×10^{-4}

TABLE 13.- RESULTS OF TEST SERIES AH

Test gas: Helium

Average leak rate: 5.11×10^{-4} torr-liter-sec^{-1}

Emission current: 1 ma

Pressure scale: 10^{-4} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			9.30×10^{-7}
2	5	5.15×10^{-4}	6.47×10^{-4}
3	10	5.14×10^{-4}	6.88×10^{-4}
4	15	5.07×10^{-4}	6.58×10^{-4}
5	20	5.11×10^{-4}	6.71×10^{-4}
6	30	5.00×10^{-4}	6.40×10^{-4}
7	40	5.11×10^{-4}	6.65×10^{-4}
8	50	5.10×10^{-4}	6.46×10^{-4}
9	60	5.18×10^{-4}	6.59×10^{-4}

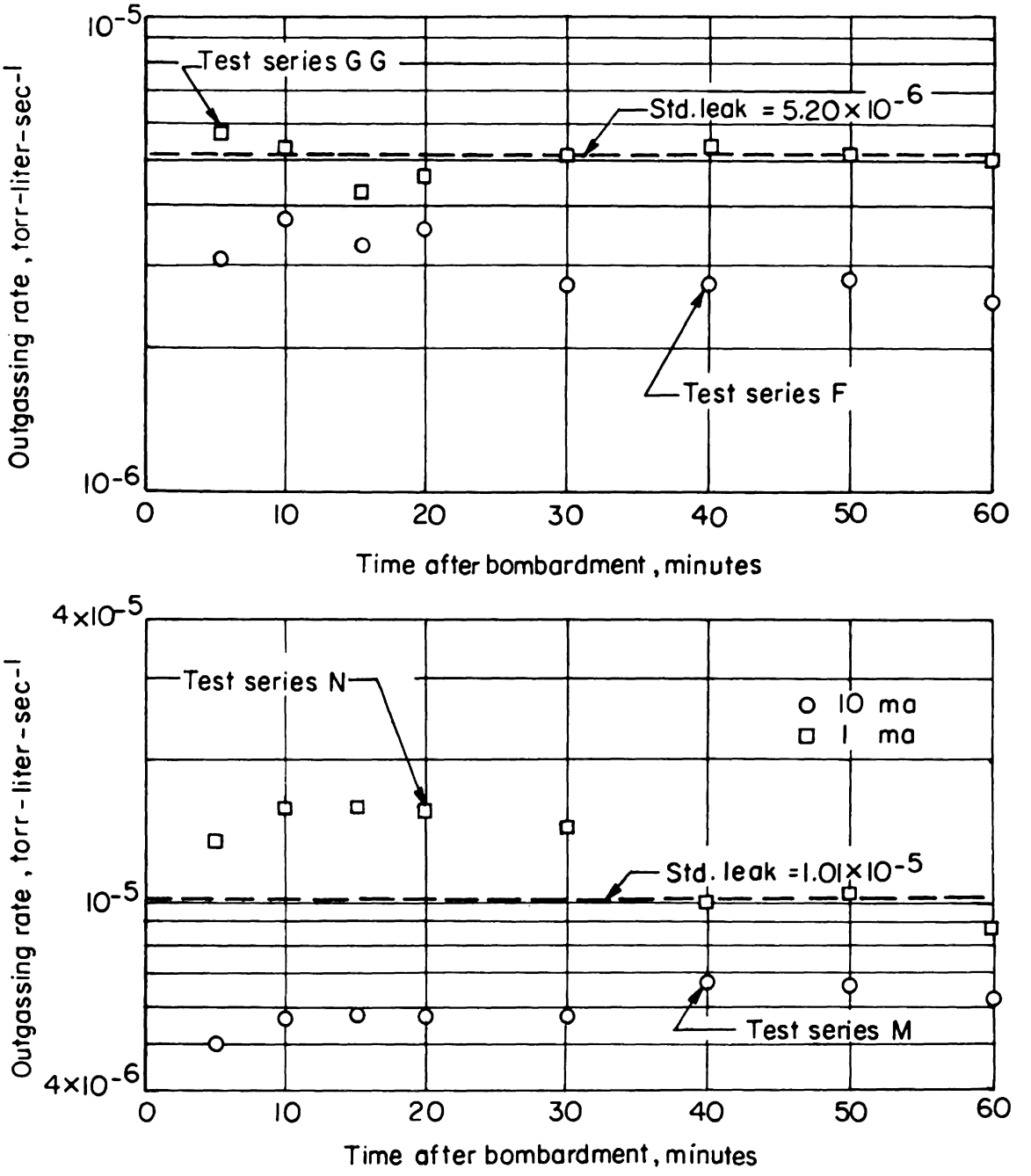


Figure 13.- Results of rate of rise measurements for helium.

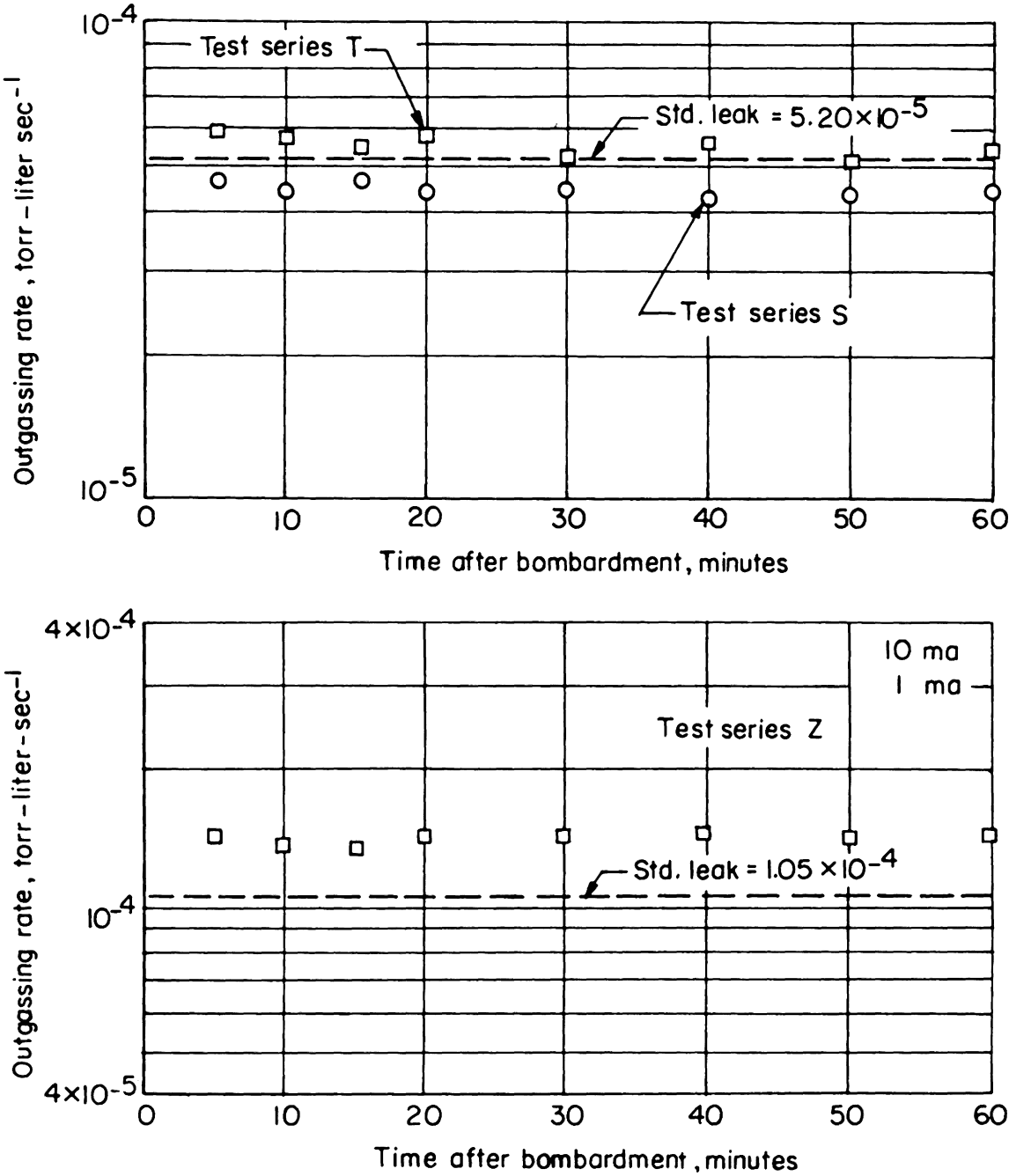


Figure 14.- Results of rate of rise measurements for helium.

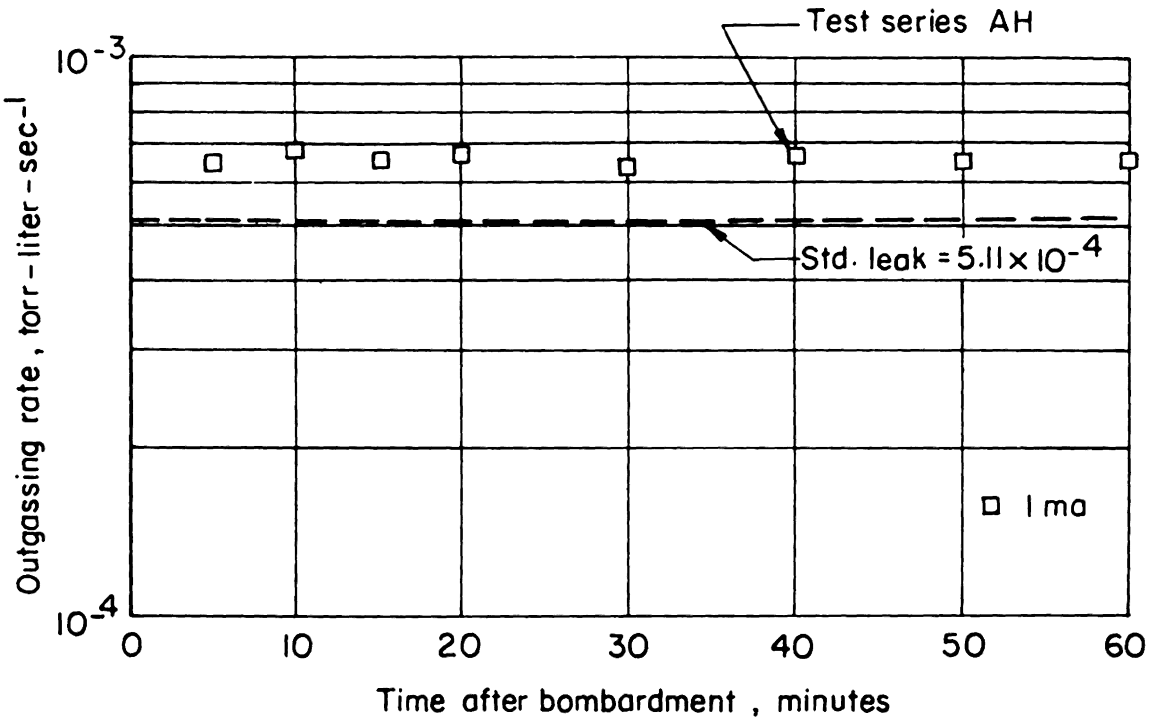


Figure 15.- Results of rate of rise measurements for helium.

TABLE 14.- RESULTS OF TEST SERIES CC

Test gas: Nitrogen

Average leak rate: 4.97×10^{-6} torr-liter-sec^{-1}

Emission current: 10 ma

Pressure scale: 10^{-6} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			6.88×10^{-7}
2	5	4.94×10^{-6}	2.83×10^{-6}
3	10	4.89×10^{-6}	3.21×10^{-6}
4	15	4.75×10^{-6}	3.27×10^{-6}
5	20	4.85×10^{-6}	3.19×10^{-6}
6	30	5.03×10^{-6}	3.08×10^{-6}
7	40	5.23×10^{-6}	3.21×10^{-6}
8	50	5.01×10^{-6}	3.21×10^{-6}
9	60	5.03×10^{-6}	3.39×10^{-6}

TABLE 15.- RESULTS OF TEST SERIES DD

Test gas: Nitrogen

Average leak rate: 5.49×10^{-6} torr-liter-sec^{-1}

Emission current: 1 ma

Pressure scale: 10^{-6} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			6.93×10^{-7}
2	5	5.32×10^{-6}	5.77×10^{-6}
3	10	5.36×10^{-6}	6.04×10^{-6}
4	15	5.58×10^{-6}	6.36×10^{-6}
5	20	5.55×10^{-6}	6.61×10^{-6}
6	30	5.63×10^{-6}	6.97×10^{-6}
7	40	5.51×10^{-6}	6.74×10^{-6}
8	50	5.43×10^{-6}	6.90×10^{-6}
9	60	5.53×10^{-6}	6.63×10^{-6}

TABLE 16.- RESULTS OF TEST SERIES K

Test gas: Nitrogen

Average leak rate: 1.02×10^{-5} torr-liter-sec^{-1}

Emission current: 10 ma

Pressure scale: 10^{-6} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			7.26×10^{-7}
2	5	1.01×10^{-5}	1.01×10^{-5}
3	10	1.04×10^{-5}	1.00×10^{-5}
4	15	1.01×10^{-5}	1.00×10^{-5}
5	20	1.02×10^{-5}	9.98×10^{-6}
6	30	1.00×10^{-5}	9.99×10^{-6}
7	40	1.03×10^{-5}	9.99×10^{-6}
8	50	1.01×10^{-5}	9.96×10^{-6}
9	60	1.02×10^{-5}	9.98×10^{-6}

TABLE 17.- RESULTS OF TEST SERIES L

Test gas: Nitrogen

Average leak rate: 1.04×10^{-5} torr-liter-sec^{-1}

Emission current: 1 ma

Pressure scale: 10^{-6} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			8.26×10^{-7}
2	5	1.04×10^{-5}	1.49×10^{-5}
3	10	1.04×10^{-5}	1.53×10^{-5}
4	15	1.04×10^{-5}	1.45×10^{-5}
5	20	1.04×10^{-5}	1.57×10^{-5}
6	30	1.04×10^{-5}	1.52×10^{-5}
7	40	1.06×10^{-5}	1.52×10^{-5}
8	50	1.03×10^{-5}	1.57×10^{-5}
9	60	1.03×10^{-5}	1.40×10^{-5}

TABLE 18.- RESULTS OF TEST SERIES AJ

Test gas: Nitrogen

Average leak rate: 1.01×10^{-5} torr-liter-sec^{-1}

Emission current: 10 ma

Pressure scale: 10^{-5} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			1.03×10^{-6}
2	5	1.01×10^{-5}	8.00×10^{-7}
3	10	9.90×10^{-6}	9.90×10^{-7}
4	15	1.01×10^{-5}	1.13×10^{-6}
5	20	1.01×10^{-5}	1.02×10^{-6}
6	30	1.02×10^{-5}	1.04×10^{-6}
7	40	1.01×10^{-5}	1.05×10^{-6}
8	50	1.01×10^{-5}	1.90×10^{-6}
9	60	9.82×10^{-6}	9.90×10^{-7}

TABLE 19.- RESULTS OF TEST SERIES AL

Test gas: Nitrogen

Average leak rate: 1.01×10^{-5} torr-liter-sec^{-1}

Emission current: 1 ma

Pressure scale: 10^{-5} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			9.32×10^{-7}
2	5	1.02×10^{-5}	9.27×10^{-6}
3	10	1.01×10^{-5}	8.85×10^{-6}
4	15	1.01×10^{-5}	9.47×10^{-6}
5	20	1.01×10^{-5}	9.17×10^{-6}
6	30	1.01×10^{-5}	1.03×10^{-5}
7	40	1.02×10^{-5}	1.07×10^{-5}
8	50	1.01×10^{-5}	1.04×10^{-5}
9	60	1.00×10^{-5}	9.67×10^{-6}

TABLE 20.- RESULTS OF TEST SERIES Q

Test gas: Nitrogen

Average leak rate: 4.97×10^{-5} torr-liter-sec^{-1}

Emission current: 10 ma

Pressure scale: 10^{-5} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			1.18×10^{-6}
2	5	5.02×10^{-5}	4.68×10^{-5}
3	10	4.94×10^{-5}	4.23×10^{-5}
4	15	5.02×10^{-5}	4.36×10^{-5}
5	20	4.90×10^{-5}	4.18×10^{-5}
6	30	4.97×10^{-5}	4.32×10^{-5}
7	40	4.92×10^{-5}	4.28×10^{-5}
8	50	5.00×10^{-5}	4.22×10^{-5}
9	60	4.98×10^{-5}	4.41×10^{-5}

TABLE 21.- RESULTS OF TEST SERIES R

Test gas: Nitrogen

Average leak rate: 5.14×10^{-5} torr-liter-sec^{-1}

Emission current: 1 ma

Pressure scale: 10^{-5} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			1.08×10^{-6}
2	5	5.17×10^{-5}	6.54×10^{-5}
3	10	5.13×10^{-5}	6.84×10^{-5}
4	15	5.12×10^{-5}	7.32×10^{-5}
5	20	5.15×10^{-5}	6.84×10^{-5}
6	30	5.15×10^{-5}	7.39×10^{-5}
7	40	5.14×10^{-5}	7.21×10^{-5}
8	50	5.16×10^{-5}	6.54×10^{-5}
9	60	5.08×10^{-5}	6.24×10^{-5}

TABLE 22.- RESULTS OF TEST SERIES X

Test gas: Nitrogen

Average leak rate: 1.00×10^{-4} torr-liter-sec^{-1}

Emission current: 1 ma

Pressure scale: 10^{-4} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			1.29×10^{-6}
2	5	1.00×10^{-4}	1.11×10^{-4}
3	10	1.00×10^{-4}	1.15×10^{-4}
4	15	1.00×10^{-4}	1.19×10^{-4}
5	20	1.01×10^{-4}	1.15×10^{-4}
6	30	9.90×10^{-5}	1.13×10^{-4}
7	40	1.00×10^{-4}	1.16×10^{-4}
8	50	1.01×10^{-4}	1.21×10^{-4}
9	60	1.00×10^{-4}	1.19×10^{-4}

TABLE 23.- RESULTS OF TEST SERIES AE

Test gas: Nitrogen

Average leak rate: 5.10×10^{-4} torr-liter-sec^{-1}

Emission current: 1 ma

Pressure scale: 10^{-4} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			6.92×10^{-7}
2	5	5.14×10^{-4}	6.22×10^{-4}
3	10	5.05×10^{-4}	6.13×10^{-4}
4	15	5.05×10^{-4}	6.01×10^{-4}
5	20	5.08×10^{-4}	6.26×10^{-4}
6	30	5.23×10^{-4}	6.65×10^{-4}
7	40	5.08×10^{-4}	6.13×10^{-4}
8	50	5.08×10^{-4}	6.21×10^{-4}
9	60	5.10×10^{-4}	6.27×10^{-4}

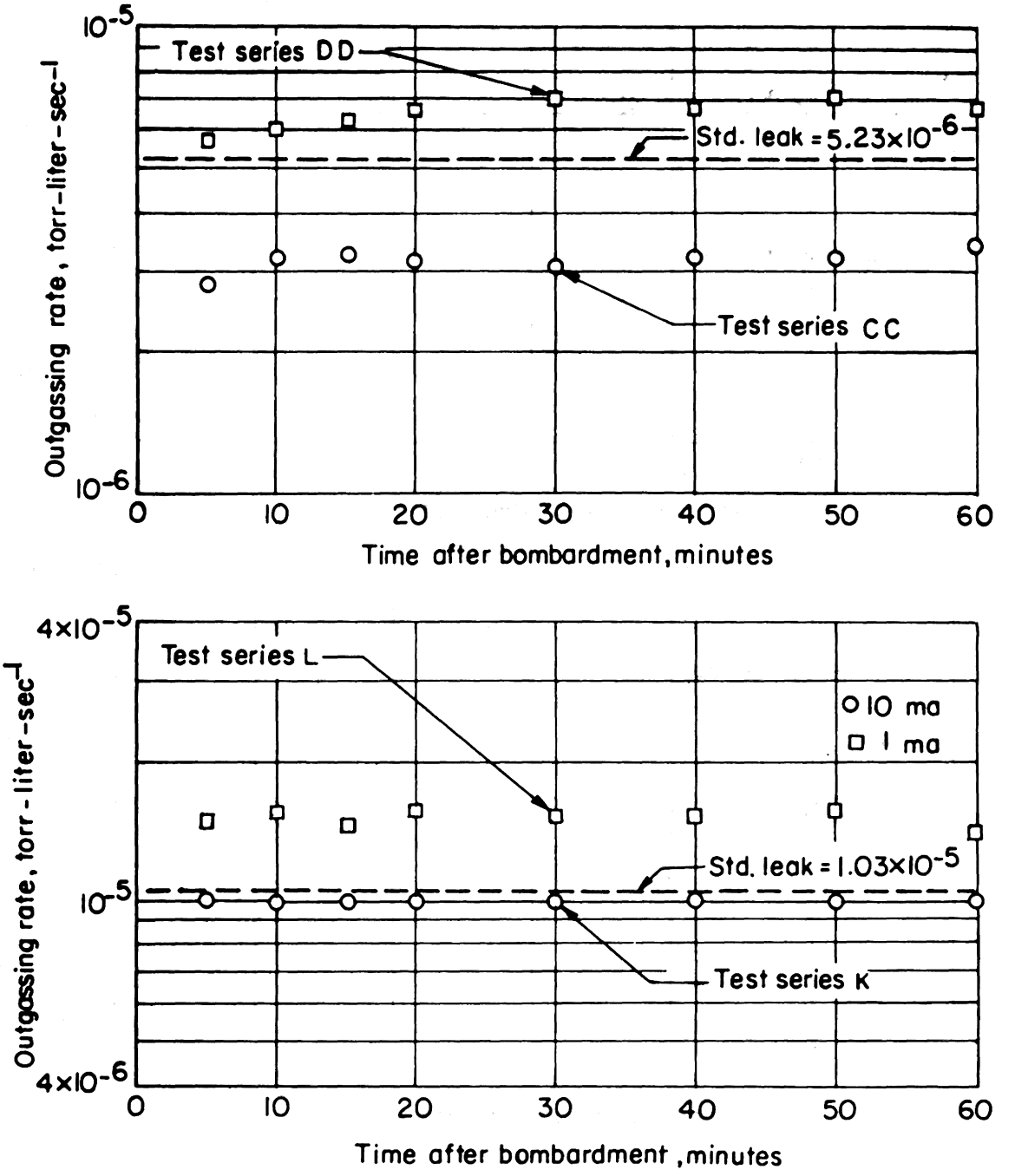


Figure 16.- Results of rate of rise measurements for nitrogen.

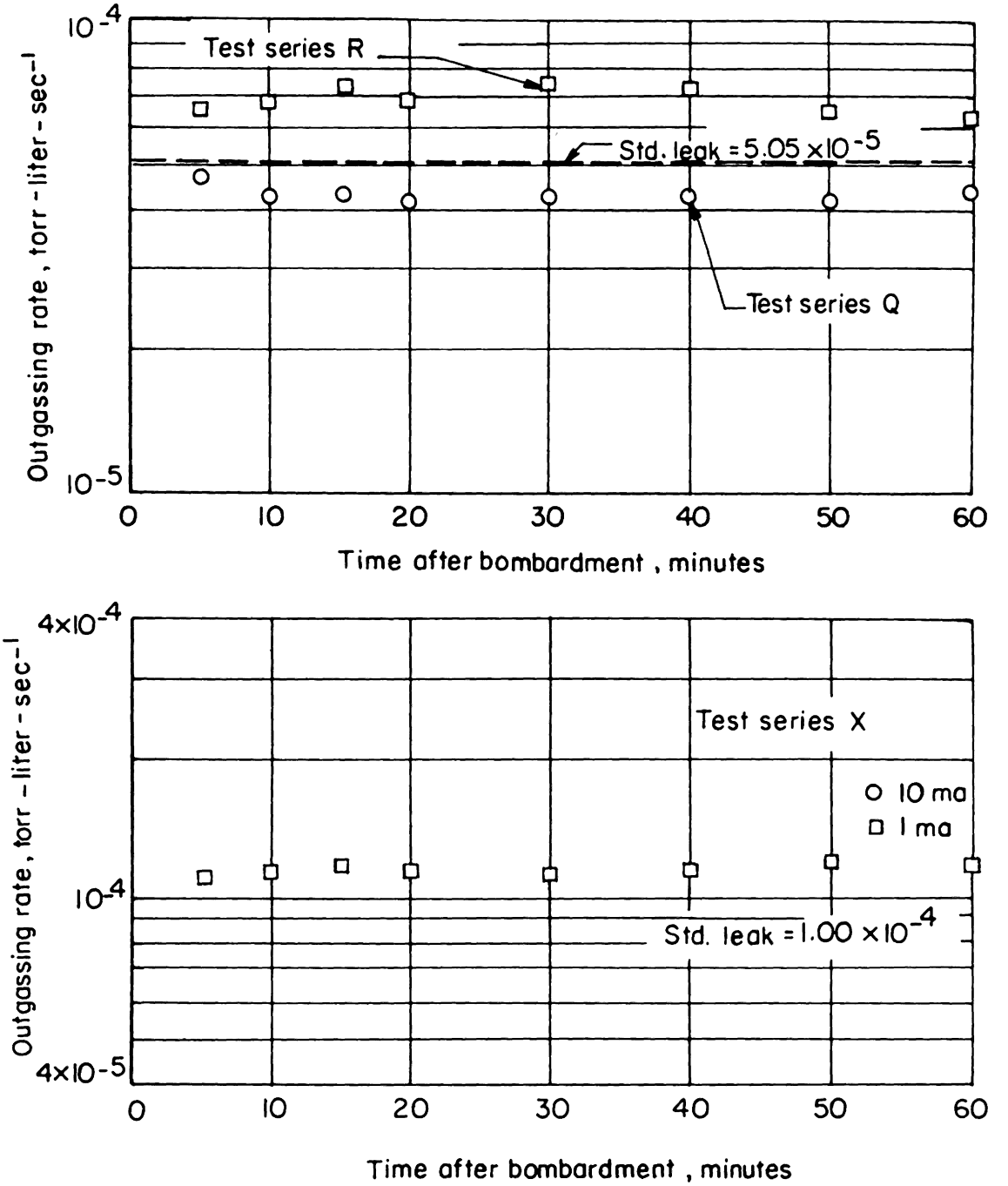


Figure 17.- Results of rate of rise measurements for nitrogen.

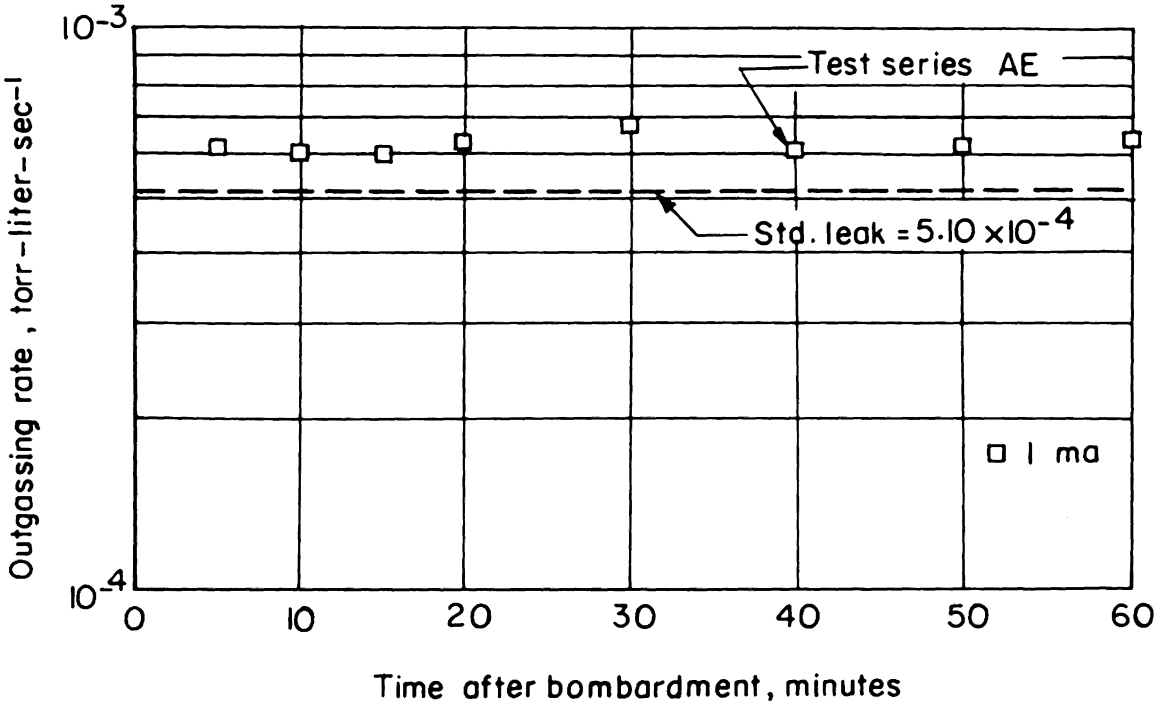


Figure 18.- Results of rate of rise measurements for nitrogen.

TABLE 24.- RESULTS OF TEST SERIES I

Test gas: Methane

Average leak rate: 5.42×10^{-6} torr-liter-sec^{-1}

Emission current: 10 ma

Pressure scale: 10^{-6} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			8.23×10^{-7}
2	5	5.42×10^{-6}	4.30×10^{-6}
3	10	5.40×10^{-6}	4.08×10^{-6}
4	15	5.34×10^{-6}	4.17×10^{-6}
5	20	5.45×10^{-6}	4.17×10^{-6}
6	30	5.45×10^{-6}	4.35×10^{-6}
7	40	5.50×10^{-6}	4.36×10^{-6}
8	50	5.38×10^{-6}	3.96×10^{-6}
9	60	5.40×10^{-6}	4.21×10^{-6}

TABLE 25.- RESULTS OF TEST SERIES J

Test gas: Methane

Average leak rate: 5.42×10^{-6} torr-liter-sec^{-1}

Emission current: 1 ma

Pressure scale: 10^{-6} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			8.14×10^{-7}
2	5	5.46×10^{-6}	7.17×10^{-6}
3	10	5.36×10^{-6}	7.27×10^{-6}
4	15	5.49×10^{-6}	7.47×10^{-6}
5	20	5.47×10^{-6}	7.52×10^{-6}
6	30	5.42×10^{-6}	7.02×10^{-6}
7	40	5.33×10^{-6}	7.02×10^{-6}
8	50	5.45×10^{-6}	6.92×10^{-6}
9	60	5.36×10^{-6}	6.70×10^{-6}

TABLE 26.- RESULTS OF TEST SERIES AO

Test gas: Methane

Average leak rate: 1×10^{-5} torr-liter-sec^{-1}

Emission current: 10 ma

Pressure scale: 10^{-6} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			1.02×10^{-6}
2	5	1.01×10^{-5}	9.20×10^{-6}
3	10	1.01×10^{-5}	9.20×10^{-6}
4	15	1.01×10^{-5}	8.70×10^{-6}
5	20	1.01×10^{-5}	9.20×10^{-6}
6	30	1.00×10^{-5}	9.20×10^{-6}
7	40	1.00×10^{-5}	8.93×10^{-6}
8	50	1.00×10^{-5}	8.70×10^{-6}
9	60	1.00×10^{-5}	9.20×10^{-6}

TABLE 27.- RESULTS OF TEST SERIES AP

Test gas: Methane

Average leak rate: 1.01×10^{-5} torr-liter-sec^{-1}

Emission current: 1 ma

Pressure scale: 10^{-6} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			1.13×10^{-6}
2	5	1.01×10^{-5}	1.17×10^{-5}
3	10	1.00×10^{-5}	1.21×10^{-5}
4	15	1.01×10^{-5}	1.28×10^{-5}
5	20	1.00×10^{-5}	1.28×10^{-5}
6	30	1.00×10^{-5}	1.23×10^{-5}
7	40	1.02×10^{-5}	1.23×10^{-5}
8	50	1.01×10^{-5}	1.24×10^{-5}
9	60	1.00×10^{-5}	1.21×10^{-5}

TABLE 28.- RESULTS OF TEST SERIES O

Test gas: Methane

Average leak rate: 9.44×10^{-6} torr-liter-sec^{-1}

Emission current: 10 ma

Pressure scale: 10^{-5} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			1.16×10^{-6}
2	5	9.32×10^{-6}	3.40×10^{-7}
3	10	9.55×10^{-6}	1.09×10^{-6}
4	15	9.50×10^{-6}	1.78×10^{-6}
5	20	9.59×10^{-6}	2.25×10^{-6}
6	30	9.00×10^{-6}	2.18×10^{-6}
7	40	9.57×10^{-6}	2.40×10^{-6}
8	50	9.52×10^{-6}	2.31×10^{-6}
9	60	9.46×10^{-6}	2.40×10^{-6}

TABLE 29.- RESULTS OF TEST SERIES P

Test gas: Methane

Average leak rate: 1.04×10^{-5} torr-liter-sec^{-1}

Emission current: 1 ma

Pressure scale: 10^{-5} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			7.88×10^{-7}
2	5	1.03×10^{-5}	1.15×10^{-5}
3	10	1.03×10^{-5}	1.23×10^{-5}
4	15	1.03×10^{-5}	1.23×10^{-5}
5	20	1.04×10^{-5}	1.26×10^{-5}
6	30	1.05×10^{-5}	1.23×10^{-5}
7	40	1.07×10^{-5}	1.19×10^{-5}
8	50	1.05×10^{-5}	1.23×10^{-5}
9	60	1.03×10^{-5}	1.19×10^{-5}

TABLE 30.- RESULTS OF TEST SERIES U

Test gas: Methane

Average leak rate: 4.92×10^{-5} torr-liter-sec^{-1}

Emission current: 10 ma

Pressure scale: 10^{-5} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			9.72×10^{-7}
2	5	4.92×10^{-5}	4.15×10^{-5}
3	10	4.94×10^{-5}	4.06×10^{-5}
4	15	4.93×10^{-5}	3.92×10^{-5}
5	20	4.94×10^{-5}	4.12×10^{-5}
6	30	4.88×10^{-5}	3.84×10^{-5}
7	40	4.88×10^{-5}	3.98×10^{-5}
8	50	4.91×10^{-5}	4.09×10^{-5}
9	60	4.92×10^{-5}	4.02×10^{-5}

TABLE 31.- RESULTS OF TEST SERIES V

Test gas: Methane

Average leak rate: 4.92×10^{-5} torr-liter-sec^{-1}

Emission current: 1 ma

Pressure scale: 10^{-5} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			9.58×10^{-7}
2	5	4.92×10^{-5}	6.40×10^{-5}
3	10	4.94×10^{-5}	6.08×10^{-5}
4	15	4.92×10^{-5}	6.20×10^{-5}
5	20	4.90×10^{-5}	6.29×10^{-5}
6	30	4.92×10^{-5}	6.04×10^{-5}
7	40	4.90×10^{-5}	6.29×10^{-5}
8	50	4.91×10^{-5}	6.03×10^{-5}
9	60	4.92×10^{-5}	6.07×10^{-5}

TABLE 32.- RESULTS OF TEST SERIES AB

Test gas: Methane

Average leak rate: 1.07×10^{-4} torr-liter-sec^{-1}

Emission current: 1 ma

Pressure scale: 10^{-4} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			6.22×10^{-7}
2	5	1.05×10^{-4}	1.10×10^{-4}
3	10	1.06×10^{-4}	1.14×10^{-4}
4	15	1.06×10^{-4}	1.14×10^{-4}
5	20	1.07×10^{-4}	1.14×10^{-4}
6	30	1.07×10^{-4}	1.13×10^{-4}
7	40	1.07×10^{-4}	1.15×10^{-4}
8	50	1.09×10^{-4}	1.12×10^{-4}
9	60	1.08×10^{-4}	1.14×10^{-4}

TABLE 33.- RESULTS OF TEST SERIES AK

Test gas: Methane

Average leak rate: 4.70×10^{-4} torr-liter-sec^{-1}

Emission current: 1 ma

Pressure scale: 10^{-4} torr

Test run no.	Time (min)	Leak rate (torr-liter-sec^{-1})	Outgassing rate (torr-liter-sec^{-1})
1 (empty system)			8.92×10^{-7}
2	5	4.75×10^{-4}	5.02×10^{-4}
3	10	4.72×10^{-4}	5.39×10^{-4}
4	15	4.72×10^{-4}	5.39×10^{-4}
5	20	4.70×10^{-4}	5.30×10^{-4}
6	30	4.70×10^{-4}	5.30×10^{-4}
7	40	4.66×10^{-4}	5.23×10^{-4}
8	50	----------	----------
9	60	4.67×10^{-4}	5.27×10^{-4}

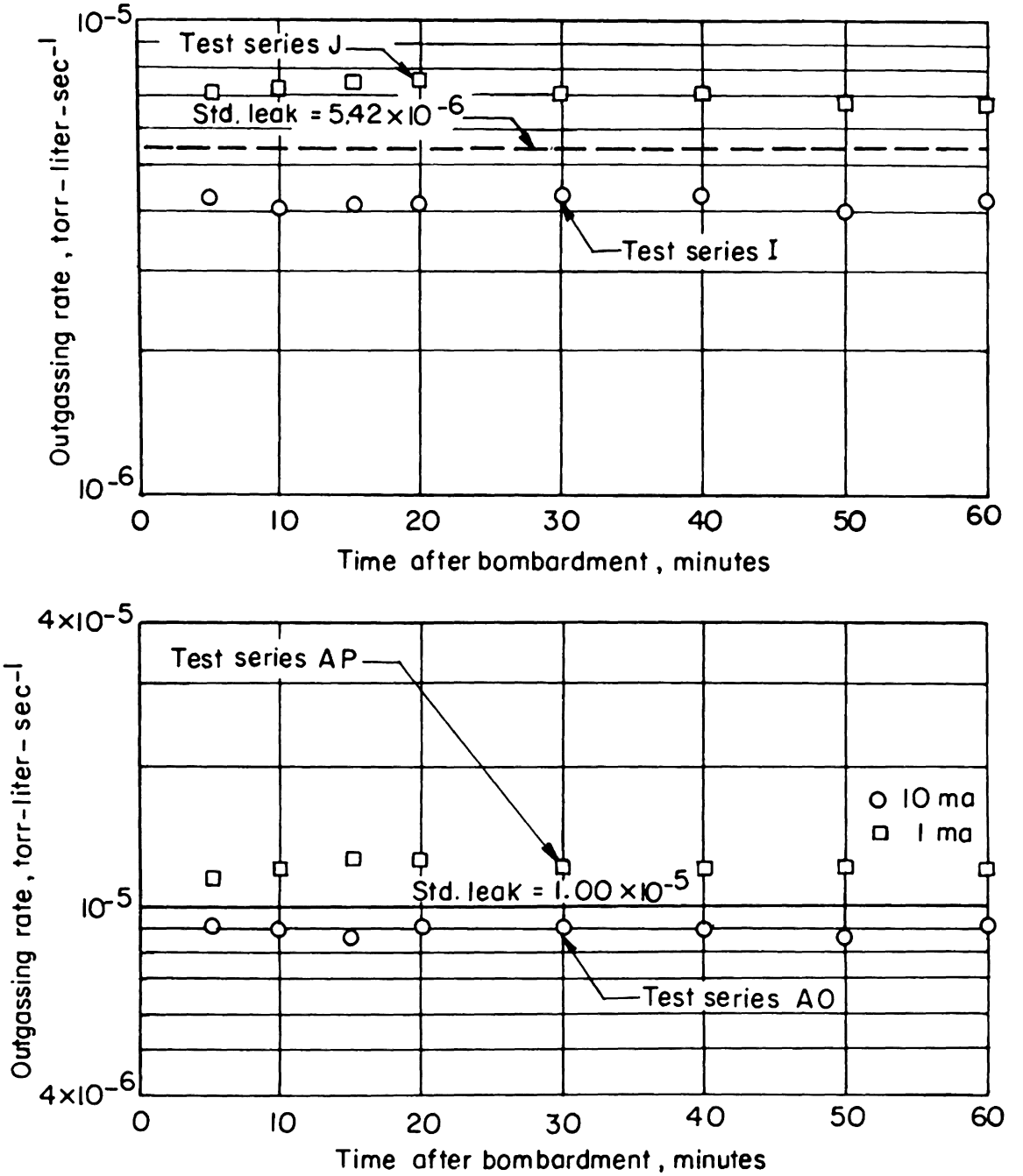


Figure 19.- Results of rate of rise measurements for methane.

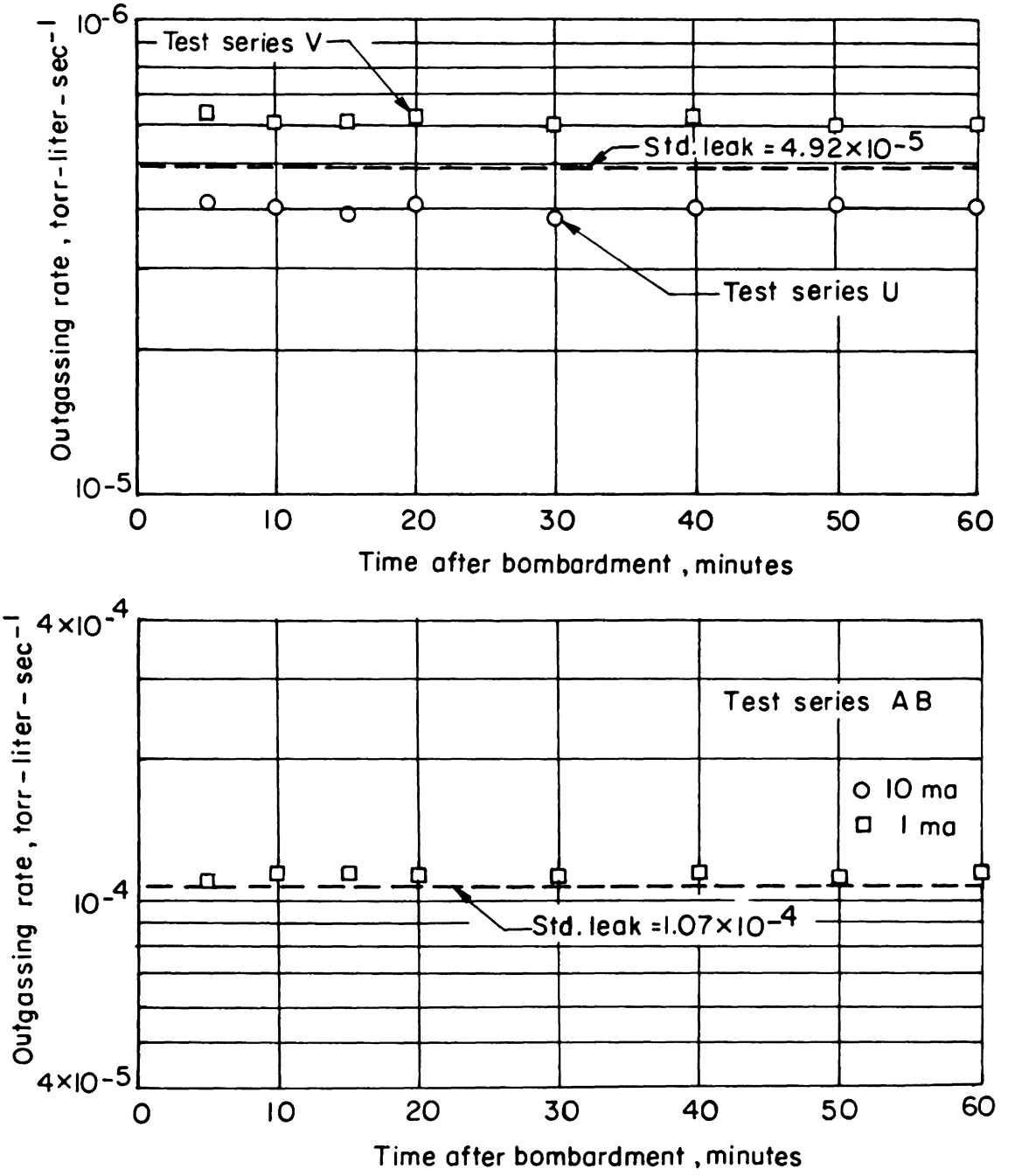


Figure 20.- Results of rate of rise measurements for methane.

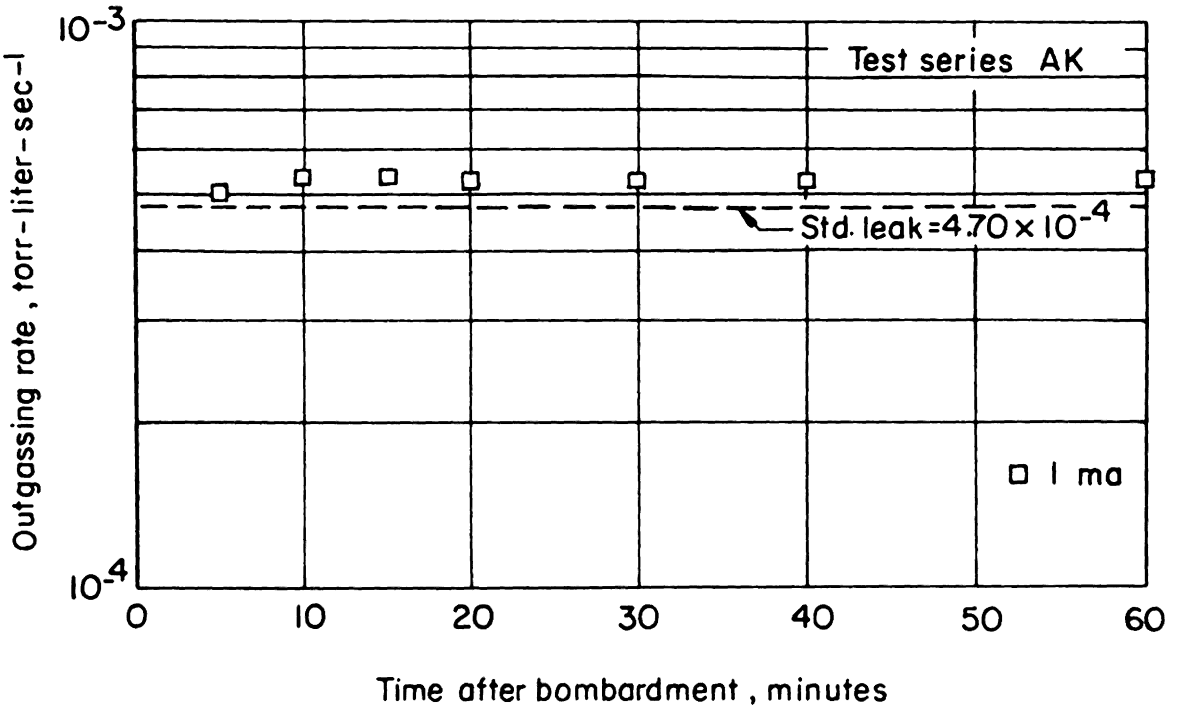


Figure 21.- Results of rate of rise measurements for methane.

VIII. ERRORS AND ACCURACY

This section of the thesis will be concerned with the measurement errors involved in the experimental evaluation of the rate of rise technique. The ultimate purpose of this section is to obtain a measurement error band within which the rate of rise measurements should lie. Any deviation of the rate of rise measurements from this error band will be the results of factors other than measurement errors.

Leak Rate Accuracy

In Appendix C an equation is developed for the accuracy of the leak rate measurement as read from the gas flow meter. This equation was derived from considerations of the maximum possible deviations of the measured quantities required in the measurement of a leak rate. The equation developed is

$$\Delta L/L = \left(\frac{\Delta P_o}{P_o} + \frac{\Delta V_p}{V_p} + \frac{\Delta t}{t} \right) \times 100 \qquad (8.1)$$

where:

$\Delta L/L$ = Maximum error in leak rate, percent

ΔP_o = Uncertainty in pressure measurement

P_o = Pressure measurement

ΔV_p = Uncertainty in volume measurement

V_p = Volume measurement

Δt = Uncertainty in time measurement

t = Time measurement

The uncertainty quantities required for equation 8.1 include observation uncertainties and physical limitations of the flow meter. Equation 8.1 was evaluated for each leak rate measurement and the results are shown in Table 34.

TABLE 34.- FLOW METER ACCURACY

Leak rate (torr-liter/sec)	Maximum error (± percent)
5×10^{-6}	6
1×10^{-5}	6
5×10^{-5}	2
1×10^{-4}	2
5×10^{-4}	3

The deviations of the measured leak rates from the set leak rate were less then those values shown in Table 34. A ±5 percent deviation was selected as a maximum expected variation of the leak rate from the set leak rate. All leak measurements within this ±5 percent band were accepted as accurate leak measurements. Those few measured data points outside this band were unacceptable and hence repeated.

Pressure Measurement Accuracy

The pressure measurement accuracy was determined from the calibration data; the accuracy of the gage was found to be ±8 percent. This accuracy level was determined from considerations of the uncertainties involved in the calibration procedure.

Another factor that can influence the gage reading and the pressure accuracy is the degree of cleanliness of the gage. It is difficult to develop a criteria for measuring gage cleanliness. In

the rate of rise evaluations the criteria used to indicate gage cleanliness was the value of the grid current during electron bombardment of the ionization gage. Although a direct correlation between grid current and cleanliness was not possible, there was evidence that a grid current of 90 to 100 ma, while electron bombarding the gage with 6 volts on the filament and 800 volts between filament and grid, existed for a "clean" gage. The evidence was that the gage was free of the effects usually associated with a contaminated gage. Contamination can cause a gage to be overly sensitive to emission current variations, to release discrete packets of gas at frequent intervals causing the gage output to fluctuate, and to indicate a higher base pressure than exists in the vacuum chamber. None of these effects were observed for a gage which reached a grid current of 90 to 100 ma during electron bombardment. This grid current was easily obtainable with a 10 minute bombardment of the gage prior to each rate of rise measurement.

Test Volume Measurement

The volume of the test chamber was measured by a water fill technique. The accuracy of this technique was estimated at 1/2 percent by considering the procedure used in the water fill technique.

Accuracy in Selection of $\Delta P/\Delta t$ to be Used in Rate of Rise Equation

In the application of the rate of rise technique it is advantageous to obtain a linear pressure trace in order that $\Delta P/\Delta t$

can be obtained for use in the Rate of Rise Equation. In many instances the pressure trace obtained in a rate of rise measurement is nonlinear. In such cases it is necessary to establish some procedure for obtaining $\Delta P/\Delta t$ for use in the Rate of Rise Equation. Generally a point on the linear portion of the pressure-time trace is selected and a tangent is constructed to the pressure trace at this point. The $\Delta P/\Delta t$ used in the Rate of Rise Equation is read from the tangent. The problem arises in that pressure traces may have two or more linear portions or be completely nonlinear. In these circumstances the location of the tangent is based on speculation and can lead to error. Therefore it is desirable to establish a procedure whereby the tangent can be located on any pressure time trace. From examining rate of rise pressure traces it was observed that in the majority of cases the pressure-time traces tend more to linearity in the upper portion of a pressure decade than in the lower portion. It was decided at the beginning of the test program that the tangent line would be located in the upper portion of the pressure-time curve (between 8 and 10 on a pressure decade). Tangent lines are shown as broken lines in figures 12(b) and 12(c). The validity of selecting the tangent line in this manner is observed in the accuracy of the measurement of the known leak rate.

Rate of Rise Outgassing Rate Accuracy

In Appendix D an equation is derived for the accuracy of the outgassing rate as measured by the rate of rise technique. This

equation was derived on the basis of the maximum deviations of the measured quantities used in the rate of rise equation.

$$\frac{\Delta Q}{Q} = \left(\frac{\Delta V}{V} + \frac{\Delta P_a + \Delta P_b}{P_2 - P_1} + \frac{\Delta t_1 + \Delta t_2}{t_2 - t_1} \right) 100 \qquad (8.2)$$

Where:

$\Delta Q/Q$ = Maximum error in outgassing rate, percent

$P_2 - P_1$ = Pressure difference over which $\Delta P/\Delta t$ was read

ΔP_a = Uncertainty in gage calibration

ΔP_b = Uncertainty in reading pressure trace

$t_2 - t_1$ = Time difference over which $\Delta P/\Delta t$ was read

Δt_1 = Uncertainty in initial time measurement

Δt_2 = Uncertainty in final time measurement

ΔV = Uncertainty in chamber volume measurement

V = Volume of test chamber

Evaluating equation 8.2 for each rate of rise measurement resulted in an approximate error of ±15 percent in determining the outgassing rate. The major component of this ±15 percent error was the accuracy of the gage calibration procedure which was ±8 percent. By considering the 15 percent accuracy of the measurement of the outgassing rate plus the 5 percent uncertainty in the measurement of the leak rate with the flow meter, the known leak rate should be measurable to within ±20 percent. Therefore any rate of rise measurement falling within the ±20 percent of the known leak rate will be considered an accurate measurement of that leak rate. Those

measurements falling outside of this error band will be examined for possibility of violation of the assumption that pumping speed equals zero during a rate of rise measurement.

IX. DISCUSSION OF RESULTS

Introduction

The experimental evaluation of the rate of rise technique shows that the technique is applicable to the measurement of outgassing rates in the range investigated. Outgassing measurements reported from the 10 ma test series were lower than those reported at 1 ma, and in all cases the 10 ma test results were lower than the known leak rate of test gas into the test volume. Most 1 ma test series resulted in outgassing rates higher than the known leak rate of test gas. For most test series the time after gage bombardment was not a factor influencing the rate of rise results. Time after gage bombardment was only a factor for those 10 ma test series at the lower leak rates or for those for which required a long pressure trace time. There was a general trend for the higher leak rates to be more easily measured.

Pressure-Time Traces

During the experimental program four types or shapes of pressure-time traces were observed. These traces are shown in figure 11 for the empty system and in figures 12(a), (b), and (c) for the leak rate measurements. The pressure trace of figure 11 was taken at 1 ma gage emission current and covered three decades of pressure rise, 10^{-7} to 10^{-4} torr. During the three decade pressure rise the rate of pressure rise varied considerably. Throughout the 10^{-7} pressure scale the rate of pressure rise was constant, but on the 10^{-6} scale the rate of pressure rise began to decline until the pressure in the

test volume began to decrease. After a period of pressure decrease, the pressure on the 10^{-6} scale began to rise again. The pressure continued to rise at an increasing rate until at the upper portion of the 10^{-5} scale the rate of pressure rise approached that value observed on the 10^{-7} scale. Some process occurred in the test volume affecting the rate of pressure rise to an extent that widespread values of outgassing rates were reported depending upon the point at which $\Delta P/\Delta t$ was read. The type of pressure trace shown in figure 11 was typical of empty system data. Detailed investigation of this pressure phenomena resulted in the following conclusions: (1) The pressure phenomena was characteristic only of the empty system measurements and was not observed in leak rate measurements. (2) Corresponding to this pressure behavior was an approximate 200° F decrease in gage filament temperature. (A later investigation showed that this empty system pressure behavior was characteristic of only Veeco RG 75 ionization gages.) Therefore the observed pressure behavior was attributed to some undefined ionization gage phenomena, particular to the Veeco gage, and did not effect the leak measurement test series.

The three types of pressure traces encountered in the leak rate measurements are shown in figure 12. Pressure traces of type (c) were most frequently observed whereas linear traces of type (a) were observed for several of the helium test measurements. Linear traces are common for gases like helium which have low gage pumping and adsorption characteristics. Pressure traces similar to type (b) were observed in two test series, test AJ and Test O. Test AJ was

conducted with a N_2 leak rate of approximately 1×10^{-5} torr-liter-sec^{-1} and a gage emission current of 10 ma. Test 0 was conducted for methane gas having the same test conditions as Test AJ. Both tests were taken on the 10^{-5} torr scale with test AJ requiring approximately 120 seconds to traverse the fifth scale and Test 0, approximately 50 seconds. The outgassing rates as measured in tests AJ and 0 were approximately 1 decade lower than the measured leak rate of test gas into the test volume, indicating that gas was being pumped in the isolated test volume during these measurements. From a study of the shape of the pressure traces of the two tests it was concluded they were of the type shown in figure 2(b) for a constant pumping speed mechanism. It was suspected that a pumping mechanism with a small and constant speed in conjunction with a pressure trace over a long period of time could result in the decade error. To verify this supposition, tests K and A0 were conducted with same test parameters as tests AJ and 0, respectively, except the pressure traces were taken on the 10^{-6} torr decade requiring only 4 seconds to traverse the decade. The results of tests AJ and 0 were that the leak rate was measurable within the ±20 percent measurement error band. The results of tests AJ, K, 0, and A0 are tabulated in Tables 35 and 36. Figures 22 and 23 are a plot of these results. It should be observed that the short time test series measure the known leak rate within ±20 percent. Also, the time after gage bombardment affects the measured outgassing rates in the long time trace tests. To obtain an estimation of the pumping speed of the process removing gas from the chamber during these test measurements, typical pressure time

TABLE 35.- COMPARISON OF TEST AJ AND TEST K

Test gas: Nitrogen

Average leak rate: 1.02×10^{-5} torr-liter/sec

Emission Current: 10 ma

Test run no.	Time (min)	Outgassing rate, torr-liter-sec^{-1} Test AJ (long trace)	Test K (short trace)
2	5	8.00×10^{-7}	1.01×10^{-5}
3	10	9.90×10^{-7}	1.00×10^{-5}
4	15	1.13×10^{-6}	1.00×10^{-5}
5	20	1.02×10^{-6}	9.98×10^{-6}
6	30	1.04×10^{-6}	9.99×10^{-6}
7	40	1.05×10^{-6}	9.99×10^{-6}
8	50	1.90×10^{-6}	9.96×10^{-6}
9	60	9.90×10^{-7}	9.98×10^{-6}

TABLE 36.- COMPARISON OF TEST O AND TEST AO

Test gas: Methane

Average leak rate: 9.72×10^{-6} torr-liter/sec

Emission current: 10 ma

Test run no.	Time (min)	Outgassing rate, torr-liter-sec^{-1}	
		Test O (long trace)	Test AO (short trace)
2	5	3.40×10^{-7}	9.20×10^{-6}
3	10	1.09×10^{-6}	9.20×10^{-6}
4	15	1.78×10^{-6}	8.70×10^{-6}
5	20	2.25×10^{-6}	9.20×10^{-6}
6	30	2.18×10^{-6}	9.20×10^{-6}
7	40	2.40×10^{-6}	8.93×10^{-6}
8	50	2.31×10^{-6}	8.70×10^{-6}
9	60	2.40×10^{-6}	9.20×10^{-6}

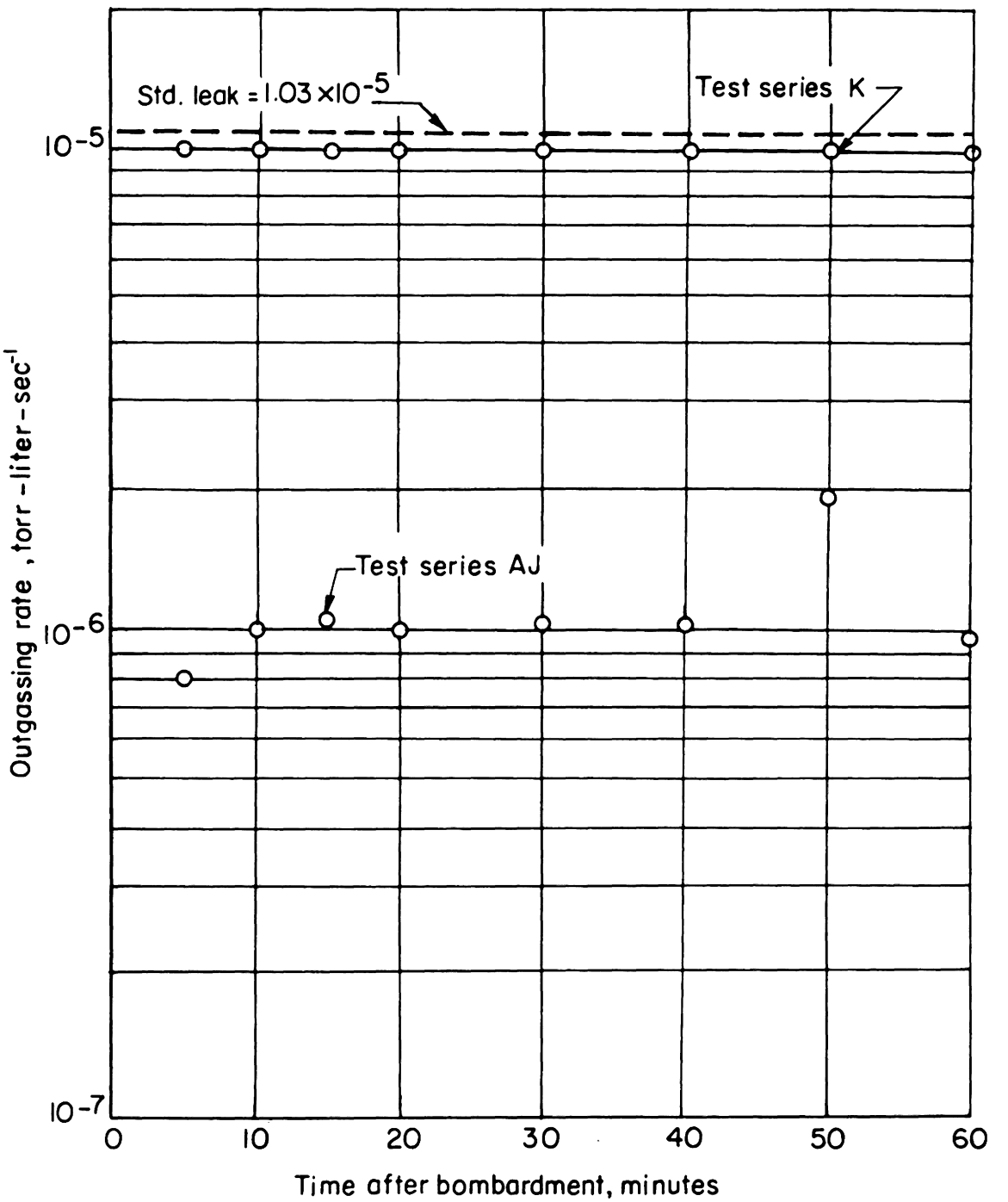


Figure 22.- Comparison of test K and AJ (nitrogen).

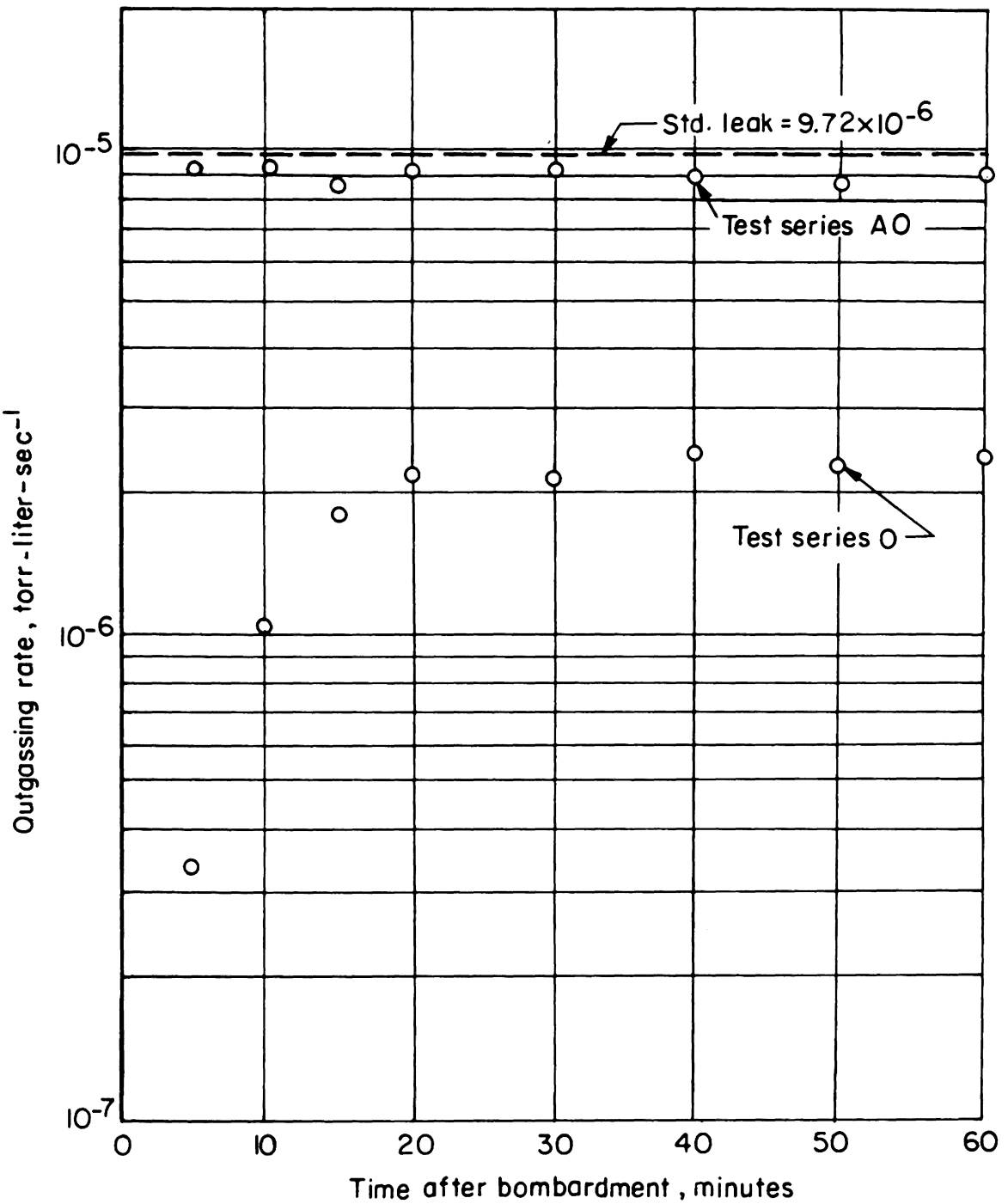


Figure 23.- Comparison of test O and AO (methane).

traces for the four tests are plotted in figures 24 and 25. In addition the pressure time traces for theoretical pressure rises having the same test parameters as tests AJ, K, O, and AO but with various values of constant pumping speeds are plotted. These theoretical pressure traces were calculated from equation 6.7. In figure 24 the pressure traces of the nitrogen tests AJ and K are compared to the theoretical pressure traces, revealing that a constant speed process was occurring in the test volume with a speed of the order of 0.1 to 0.2 liters-sec^{-1}. Figure 25 shows the same results for the two methane tests, O and AO.

Therefore, a pumping mechanism with a nearly constant speed of 0.1 to 0.2 liter-sec^{-1} combined with a long time pressure trace resulted in a decade error in outgassing rate. By proper selection of test parameters (trace length) the effects of this pumping mechanism was minimized, without reducing the pumping speed, allowing the outgassing rate to be measured within the ±20 percent measurement error band. It will be shown later that this pumping mechanism was ionization gage pumping and therefore could be further minimized if necessary by a reduction in gage emission current.

Adsorption Effects

From the results of the evaluation of the rate of rise technique, there was no eivdence of adsorption occurring. Adsorption is not a factor influencing rate of rise measurements for the test gases and pressure ranges investigated.

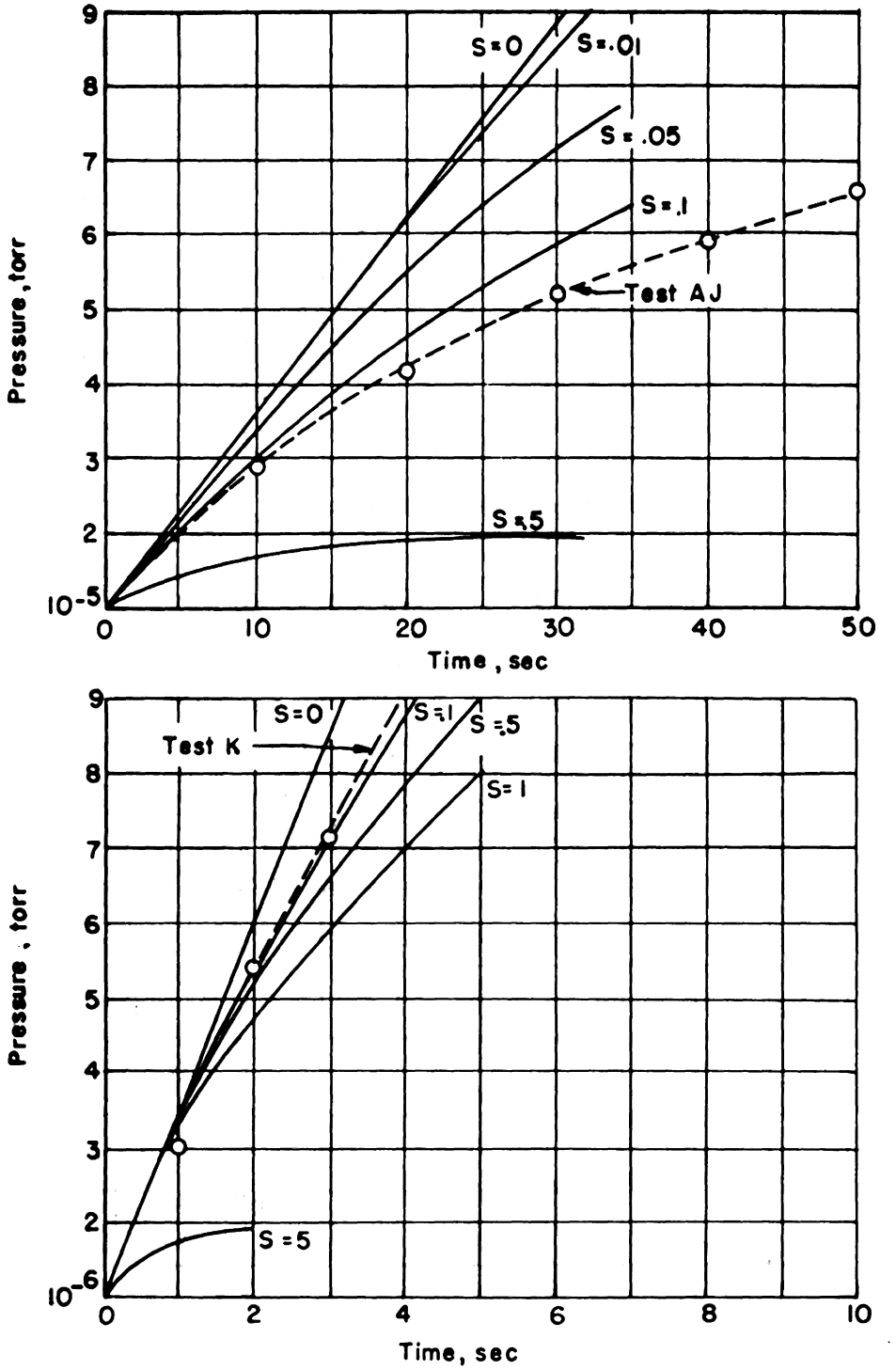


Figure 24.- Estimation of pumping speed (nitrogen).

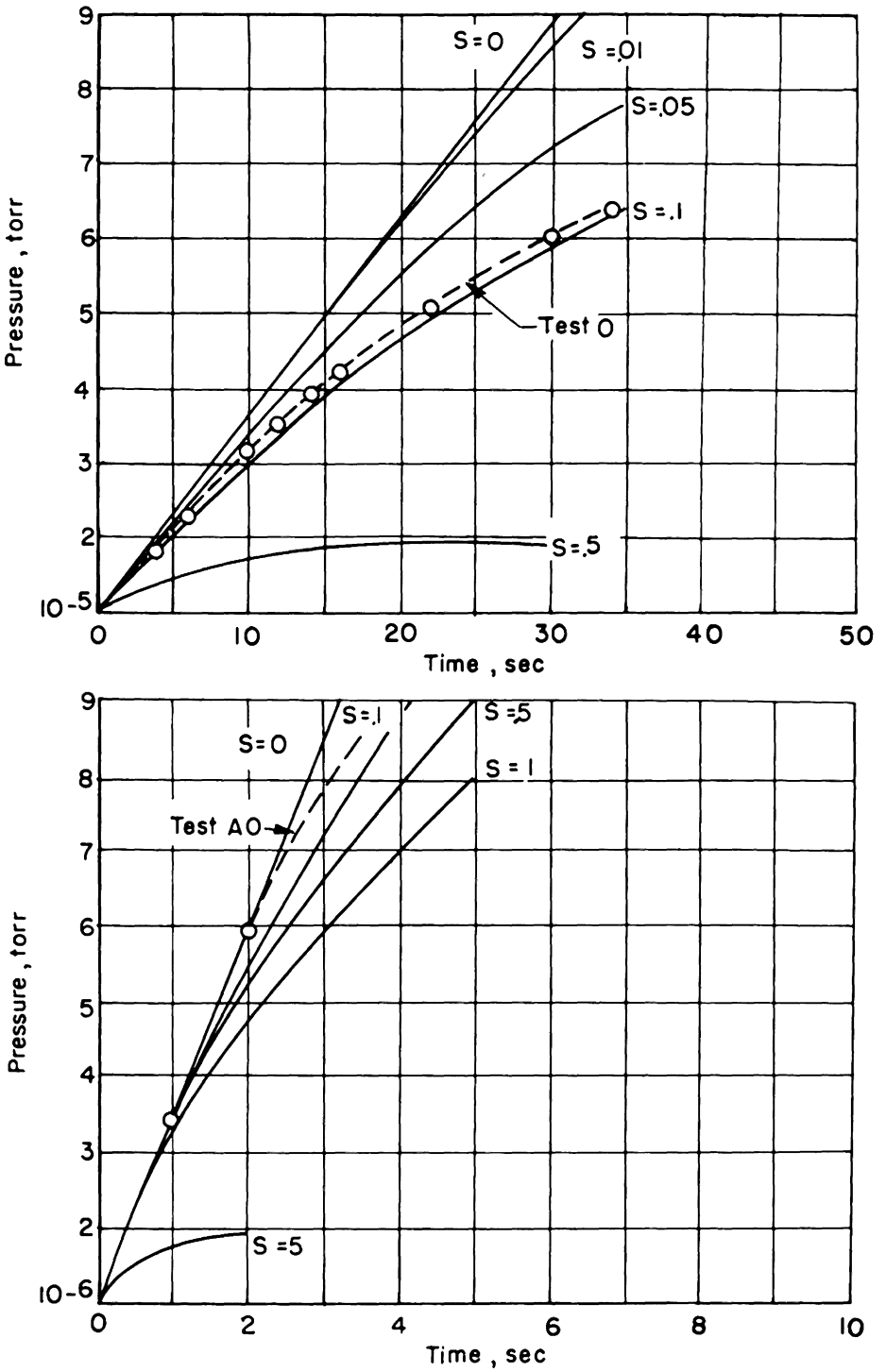


Figure 25.- Estimation of pumping speed (methane).

Ionization Gage Pumping

There are several different mechanisms by which gas is pumped by an ionization gage during its normal operation. These mechanisms include ionic pumping and various types of adsorption pumping. The effects of these pumping mechanisms on a rate of rise measurement is that the observed value of outgassing rate is lower than the true value. Gage pumping can be decreased by operating the ionization gage at reduced emission currents. To investigate the effects of gage pumping on rate of rise outgassing measurements, leak rate-test gas combinations were evaluated at two values of gage emission currents, 10 ma and 1 ma. Comparison of test results at the two emission currents will evaluate the effects and importance of gage pumping on rate of rise measurements. Figures 13 through 21 show the results of 10 ma and 1 ma test series taken at a particular leak rate and for a given test gas. As seen from these figures, the outgassing rates as measured at 1 ma are higher than those measured at 10 ma, indicating that gage pumping is reduced by reducing gage emission current. In some of the 1 ma test series the measured leak rates lie above the ±20 percent error band. This is due to the gage constant at 1 ma being different from that at 10 ma. The gage constant was determined by calibrating the gage for nitrogen at 10 ma emission. The 1 ma test data presented in figures 13 through 21 were plotted using the 10 ma gage constant. Melfi and Kern[28] report that for a Bayard Alpert gage the gage constant may be as much as 18 percent higher at 1 ma than at 10 ma emission. If a negative correction of the order of 18 percent is applied to the 1 ma test series data, then these test measurements fall

within the 20 percent measurement error band. Therefore, failure to calibrate the ionization gages at 1 ma emission current resulted in the 1 ma test series measurements falling outside of the measurement error band. Kern in his investigations conducted limited experimentation on the type of test gages used for this investigation, but sufficient data was not available to apply the gage constant correction to the 1 ma data.

In the discussion of the shape of the pressure traces, it was concluded that for the four test series under discussion that a constant pumping speed process was removing gas from the test volume at a rate of approximately, 0.1 to 0.2 liter-sec^{-1}. In the case of N_2 gas, literature reports a gage pumping speed of the order of 0.1 to 0.2 liter-sec^{-1}. From Byvik's[29] work on the pumping speed of the Veeco RG 75 gage, it is plausible that the pumping speed of the gage for N_2 is constant for trace lengths of the order of 150 seconds. Comparing the reported pumping speed behavior of the gage for N_2 with that pumping speed observed in tests K and AJ, it is concluded that gage pumping was the source of gas removal resulting in the decade error in outgassing rates. By similar considerations of the gage pumping speed behavior for helium and methane in conjunction with the observed outgassing measurements, it is concluded that ionization gage pumping was the only mechansim observed to be removing gas from the isolated test volume. The deviation of the measured outgassing rates from the known leak rate and hence the ability of the rate of rise

technique to measure a leak rate was dependent on three factors: the measurement error, failure to calibrate ionization gages at 1 ma, and ionization gage pumping.

Evaluation of the Ability of the Rate of Rise Technique to Measure Outgassing Rates

All leak rate-test gas combinations were measurable within the ±20 percent measurement error band. Some measured outgassing rates fell outside of this error band, but were found to be the result of failure to calibrate the gage at 1 ma emission current or the result of ionization gage pumping. Adsorption effects were not encountered in the evaluation of the rate of rise technique and are believed to be important only at the lower pressure levels.

From the experimental evaluation of the rate of rise technique the following procedures are suggested when using the rate of rise technique:

(1) The outgassing from the test specimen should be the primary influence on the pressure behavior.

(2) The pressure scale over which the pressure rise data is to be taken should be such that the trace time required to cover the pressure scale is as short as possible, but not less than the response time of the system and instrumentation. This minimization of the trace time reduces the effects of gage pumping.

(3) Additional reduction of the effects of gage pumping can be obtained by reducing the gage emission current. In many cases due to valve closure effects or possibly adsorption

effects when encountered, guideline 2 may not be feasible and reduction of gage emission current will be the only means of reducing the effects of gage pumping.

(4) Time after gage bombardment at which rate of rise measurements are taken may or may not be a factor influencing outgassing measurements, but should be investigated for each outgassing experiment. This will insure that time after gage bombardment is not a factor or; if shown to be a factor, will reveal the time at which the outgassing measurements should be taken.

X. SUMMARY

It has been shown that there is a definite need for the knowledge of outgassing rates of a variety of materials. With this need there has developed an increasing interest in the techniques available for the measurement of outgassing rates.

A review of the literature indicated that no one technique was acceptable as a standard technique, but there were three techniques which are used frequently to measure outgassing rates. Of these techniques, the rate of rise method is simple and easy to apply but is used with skepticism due to the errors which manifest themselves in the application of the technique. The literature showed that the rate of rise technique has been used with various degrees of successfulness and with varying detail as to the accuracy of application. An experimental evaluation of the rate of rise technique was not found in the literature.

A test program was conducted to evaluate the rate of rise technique by measuring known leak rates of known gases. The resulting measurements were examined from the viewpoint of errors. It was determined that the measurement error was ±20 percent of the known leak rate. An additional error was found in the form of ionization gage pumping, and in some cases resulted in a decade error in outgassing measurements. The errors due to gage pumping were found to be minimized if proper care was used in the experimental procedures.

In general the rate of rise technique was adaptable to the measurement of outgassing rates in the range of 5×10^{-6} torr-liter-sec^{-1} to 5×10^{-4} torr-liter-sec^{-1} for helium, nitrogen, and methane gas.

XI. CONCLUSIONS

The experimental evaluation of the rate of rise technique resulted in the following conclusions:

(1) The rate of rise technique of measuring outgassing rates is applicable to the measurement of outgassing rates with an accuracy of ±20 percent in the range of outgassing rates from 5×10^{-6} torr-liter-sec^{-1} to 5×10^{-4} torr-liter-sec^{-1} and for gases similar to helium, nitrogen, and methane.

(2) Large errors may result from a rate of rise outgassing measurement if the proper procedures are not followed.

(3) Adsorption is not a factor influencing rate of rise measurements in the range of outgassing rates and for the test gases investigated. In the case of test chambers constructed of materials other than stainless steel, adsorption may be an influencing factor.

(4) Ionization gage pumping does influence rate of rise outgassing measurements. A long time (50 to 120 second) pressure trace combined with a gage pumping speed of the order of 0.1 liter-sec^{-1} can introduce errors as large as a decade in outgassing measurements.

(5) Errors due to gage pumping can be minimized by proper selection of trace time and/or by reduction of gage emission current.

XII. RECOMMENDATIONS

From the results of the evaluation of the rate of rise technique the following are suggested for future investigation:

(1) To evaluate the rate of rise technique for chemically active gases, such as oxygen, and for hydrocarbon gases of large molecular weight.

(2) To investigate the gage phenomena observed in the empty system outgassing pressure traces.

(3) To investigate the adsorption properties of various gases on stainless steel at pressures of 10^{-5} torr and lower. Such an investigation would determine at what conditions adsorption would affect rate of rise measurements.

(4) To establish a criteria for the determination of ionization gage cleanliness such that the degree of cleanliness of a gage would be known at any time during its operation.

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XVI. APPENDICES

APPENDIX A

List of Equipment

1. Mechanical Pump. Welch Scientific Company, Chicago Illinois, Model 8700-2, 5 CFM, NASA 233312. The pump was used as a roughing and holding pump on the vacuum facility.

2. Mechanical Pump. Welch Scientific Company, Chicago, Illinois, Model 8700-2, 5 CFM, NASA 126033. The pump was used to differentially pump the slide valve and as a roughing pump for the gas flow meter.

3. Diffusion Pump. National Research Company, Newton 61, Massachusetts, Model H54-750, Type 0161, NASA 120489, pumping capacity 750 liters/sec., pumping fluid DC 705. The diffusion pump was used as the main vacuum pump.

4. Water Baffle. National Research Company, Newton 61, Massachusetts, Model HW0318, 4-inch size. The baffle was used to aid in minimizing backstreaming of diffusion pump oil.

5. Liquid Nitrogen Trap. National Research Company, Newton 61, Massachusetts, Model HN0315-4, 4-inch size, consumption rate of 120 cc, hour^{-1}. The liquid nitrogen trap was used to aid in minimizing backstreaming of diffusion pump oil.

6. Liquid Nitrogen Level Control. Johns and Frame Company, Livermore, California, Model SL2. The level controller was used to maintain the liquid nitrogen level in the liquid nitrogen trap.

7. Liquid Nitrogen Dewar. Standard Air Company, Inc., Brooklyn, New York, capacity of 100 liters. The dewar was used as a reserve supply of liquid nitrogen for the liquid nitrogen trap.

8. Slide Valve. National Research Company, Newton 61, Massachusetts, Model HC4-A, air operated, 4-inch size. The valve was used to isolate the test volume from the diffusion pump.

9. Valve. Nuclear Products Company, Cleveland 10, Ohio, Model 4BG, stainless steel. The valve was used as maintenance valve on gas inlet tube, to isolate the test volume during flow meter maintainence.

10. Gage Controller. Hastings-Raydist, Inc., Hampton, Virginia, Serial 338, NASA 122056, range 0-1 atmosphere in microns of mercury. The controller was used to measure foreline pressure.

11. Ionization Gage Controller. Veeco Vacuum Corporation, New Hyde Park, Long Island, New York, type RG 21X, NASA 125592 and NASA 125593, range 10^{-3} to 2×10^{-10} torr. The ionization gage controllers were used as power supplies for the operation of the ionization gages.

12. Ionization Gage. Veeco Vacuum Corporation, New Hyde Park, Long Island, New York, type RG 75, pyrex tubulation, thoria-coated iridium filament, range 2×10^{-10} to 10^{-3} torr, NASA 434519, NASA 418C18, and NASA 414T40. The ionization gages were used to measure the pressure in the test volume.

13. Sanborn Recorder. Sanborn Company, Wlathan, Massachusetts, Model 150, NASA 105854. The recorder was used to record the pressure changes in the test volume by recording the output of the ionization gage as a function of time.

14. Recording Oscillograph. Bristol Company, Waterbury, Connecticut, model IPG560, range 0-1000 F. The recorder was used to record the output of the thermocouples instrumenting the test volume.

15. Thermometer. Taylor Instrument Company, Asheville, North Carolina, Mercury thermometer, 0-212F, 2F increments. The thermometer was used to measure the temperature of the test gases being leaked into the test volume.

16. Gas Flow Meter. Varian Associates, Palo Alto, California, Model 9735000, range 5×10^{-6} to 2×10^{-1} torr-liter-sec^{-1}. The flow meter was used to measure the flow rate of test gases into the test volume.

17. Leak Detector. Veeco Vacuum Corporation, New Hyde Park, Long Island, New York, Series MS-9, uses helium as tracer gas. The leak detector was used to check the vacuum facility for possible leaks.

18. Power Supply. The Instrument Research Division, National Aeronautics and Space Administration, Langley Research Center, Hampton, Virginia. The power supply was used to electron bombard the ionization gages.

19. Test Chamber. The Heavy Machine Shop, National Aeronautics and Space Administration, Langley Research Center, Hampton, Virginia. The test chamber was constructed of 347 stainless steel with an internal volume of approximately 3.5 liters.

20. Gas Inlet Tube. Space Vacuum Laboratory, National Aeronautics and Space Administration, Langley Research Center, Hampton, Virginia. The gas inlet tube was constructed of 347 stainless steel and was used to admit the test gas into the test volume with a radial directional flow.

21. Thermocouple Feedthrough. Varian Associates, Palo Alto, California, Model 954-5015. The feedthrough was used to admit thermocouples into the test volume for the recording of chamber wall temperatures.

22. Methane Gas. Air Products and Chemical, Inc., Allentown, Pennsylvania, CT grade, supplied in 200 cubic foot quantities. The methane gas was used as a test gas for the evaluation of the rate of rise technique.

23. Nitrogen Gas. Southern Oxygen Corporation, Hampton, Virginia, Seaford grade, supplied in 200 cubic foot quantities. The nitrogen gas was used as a test gas for the rate of rise evaluations.

24. Helium Gas. Air Reduction Company, Richmond, Virginia, supplied in 200 cubic foot quantities. The helium gas was used as a test gas for the evaluation of the rate of rise technique.

25. Pressure regulator. Matheson Company, Inc., East Rutherford, New Jersey, models 19-580, 19-590, 19-350. The pressure regulators were used to control the supply pressure of the reserve test gas going to the gas flow meter.

APPENDIX B

Derivation of the Leak Rate Equation

In this appendix, equation 7.3 which was used to determine the flow rate of test gas into the test volume will be derived. Leak rate, as is outgassing rate, can be defined to be the time derivative of the product of pressure and volume, or in equation form

$$L = d/dt\ (PV) \tag{B-1}$$

Performing the differentiation,

$$L = P\,\frac{dV}{dt} + V\,\frac{dP}{dt} \tag{B-2}$$

Equation B-2 is the basis of the measurement of leak rates. The Varian gas flow meter employs a constant pressure concept resulting in a simplification of equation B-2; that is, $dP/dt = 0$ and

$$L = P\ dV/dt \tag{B-3}$$

Therefore the measurement of the change in volume of a quantity of gas at a constant pressure determines the leak rate of that gas. The Varian flow meter utilizes a variable volume to maintain a constant pressure during leak rate measurement. The variable volume is obtained by using a constant area piston of variable displacement. The expression dV/dt in equation B-3 is replaced by a time average change in volume,

$$\frac{dV}{dt} = \frac{A(X_2 - X_1)}{t_2 - t_1} \tag{B-4}$$

where:

A = Area of displacement piston, mm^2

X_2 = Final displacement of piston, mm

X_1 = Initial displacement of piston, mm

t_2 = Time corresponding to x_2, sec

t_1 = Time corresponding to x_1, sec

Referencing the leak rate to a standard temperature of 20° C (527.67R) and incorporating equation B-4, the leak rate equation becomes

$$L = \frac{P_o A(X_2 - X_1)\ 527.67}{(t_2 - t_1)\ T_R} \times 10^{-6} \qquad \text{(B-5)}$$

where A, X_2, X_1, t_2 and t_1 have previously been defined and

L = Leak rate, torr-liter-sec^{-1}

P_o = Reference pressure, torr

T_R = Room temperature, R

527.67 = Leak rate reference temperature, R

APPENDIX C

Leak Rate Accuracy

An equation for the error involved in the measurement of a leak rate can be developed by considering the definition of leak rate

$$L = P\, dV/dt + V\, dP/dt \tag{C-1}$$

Replacing dV/dt by its time average and developing a similar time average for dP/dt results in

$$L = P \frac{V_p}{t_2 - t_1} + V \frac{P_V}{t_2 - t_1} \tag{C-2}$$

where V_p is change in volume at constant pressure and P_V is the change in pressure at constant volume. Taking the total differential of both sides of equation C-2 and letting $t_1 = 0$ and $t_2 = t$ gives

$$dL = \frac{V_p}{t} dP + \frac{P}{t} dV_p + \frac{P_v}{t} dV + \frac{V}{t} dP_v - \left(\frac{PV_p + VP_V}{t^2} \right) dt \tag{C-3}$$

or

$$\Delta L = \frac{V_p}{t} \Delta P + \frac{P}{t} \Delta V_p + \frac{P_V}{t} \Delta V + \frac{V}{t} \Delta P_V - \left(\frac{PV_p + VP_V}{t^2} \right) \Delta t \tag{C-4}$$

Dividing equation C-4 by equation C-2 with $t_1 = 0$ and $t_2 = t$ gives

$$\frac{\Delta L}{L} = \frac{V_p \Delta P + P \Delta V_p + P_V \Delta V + V \Delta P_V - \frac{\Delta t}{t} \left(PV_p - VP_v \right)}{PV_p + VP_V} \tag{C-5}$$

Since the flow meter is of constant pressure design, then $P_V = 0$ and $\Delta P_V = 0$ and substituting P_o for P gives

$$\frac{\Delta L}{L} = \frac{\Delta P_o}{P_o} + \frac{\Delta V_p}{V_p} - \frac{\Delta t}{t} \qquad \text{(C-6)}$$

Equation C-6 gives a relationship for the deviation of the measured leak rate in terms of the deviation of the three measured parameters: pressure, volume, and time. The largest deviation occurs when Δt is negative and hence the leak rate accuracy equation becomes

$$\frac{\Delta L}{L} = \left(\frac{\Delta P_o}{P_o} + \frac{\Delta V_p}{V_p} + \frac{\Delta t}{t} \right) \times 100 \qquad \text{(C-7)}$$

where:

$\Delta L/L$ = Maximum error in leak rate, percent

ΔP_o = Uncertainty in pressure measurement

P_o = Pressure measurement

ΔV_p = Uncertainty in volume measurement

Δt = Uncertainty in time measurement

t = Time measurement

Equation C-7 is equation 8.1 which was used to determine the accuracy of each leak rate measurement.

APPENDIX D

Rate of Rise Outgassing Rate Accuracy

An equation for the error involved in the measurement of an outgassing rate by the rate of rise technique can be developed by considering the rate of rise equation 7.2

$$Q = V\,\Delta P/\Delta t$$

or

$$Q = V\,\frac{P_V}{t_2 - t_1} \tag{D-1}$$

where:

Q = Outgassing rate, torr-liter-sec^{-1}

V = Volume, liter

P_V = Pressure change at constant volume, torr

t_1 = Initial time, sec

t_2 = Final time, sec

and by forming the total differential of Q with V, V_p, t_2, and t_1 as independent variables.

$$dQ = \frac{dQ}{dV}\,dV + \frac{dQ}{dP_V}\,dP_V + \frac{dQ}{dt_1}\,dt_1 + \frac{dQ}{dt_2}\,dt_2 \tag{D-2}$$

Then by obtaining the required partial derivatives from equation D-1 and substituting into equation D-2, gives

$$\Delta Q = \frac{P_V}{t_2 - t_1}\,\Delta V + \frac{V}{t_2 - t_1}\,\Delta P_V + \frac{VP_V}{(t_2 - t_1)^2}\,\Delta t_1 - \frac{VP_V}{(t_2 - t_1)^2}\,\Delta t_2 \tag{D-3}$$

Dividing equation D-3 by equation D-1 and noting that the maximum error occurrs when all terms are positive, results in

$$\Delta Q/Q = \frac{\Delta V}{V} + \frac{\Delta P_V}{P_V} + \frac{\Delta t_1 + \Delta t_2}{t_2 - t_1} \qquad \text{(D-4)}$$

Equation D-4 relates the error in the measurement of an outgassing rate to the error of the observed quantities. ΔP_V consists of two terms; ΔP_a and ΔP_b. ΔP_a is the error in pressure measurement due to the uncertainties resulting from gage calibration, where as ΔP_b is the error due to the uncertainties in reading the pressure trace. Therefore equation D-4 for the maximum error in a measurement of an outgassing rate becomes

$$\frac{\Delta Q}{Q} = \left(\frac{\Delta V}{V} + \frac{\Delta P_a + \Delta P_b}{P_2 - P_1} + \frac{\Delta t_1 + \Delta t_2}{t_2 - t_1} \right) \times 100 \qquad \text{(D-5)}$$

where:

$\Delta Q/Q$ = Maximum error in outgassing rate, percent

$P_2 - P_1$ = Pressure difference over which $\Delta P/\Delta t$ was read

ΔP_a = Uncertainty in reading pressure trace

$t_2 - t_1$ = Time difference over which $\Delta P/\Delta t$ was read

Δt_1 = Uncertainty in initial time measurement

Δt_2 = Uncertainty in final time measurement

ΔV = Uncertainty in chamber volume measurement

V = Volume of test chamber

This equation is equation 8.2 which was used to determine the accuracy of the measured outgassing rates.

EXPERIMENTAL EVALUATION OF THE RATE OF RISE TECHNIQUE FOR MEASURING OUTGASSING RATES IN VACUUM

By

Gerald Lee Gregory

ABSTRACT

The subject investigation deals with an experimental evaluation of the rate of rise technique of measuring outgassing rates in vacuum. There is an increasing need for the knowledge of outgassing rates of various materials and therefore there has been an increasing interest in the techniques available for the measurement of outgassing rates. Several measurement techniques for outgassing rates are reported in the literature and among these is the rate of rise method. The treatment of the rate of rise technique in the literature is contradictory and inadequate. Many opinions are offered in the literature as to the suitability or unsuitability of the rate of rise technique to measure outgassing rates. However, these opinions are not based on sound technical background as a technical evaluation of the technique is not presently available. Therefore the purpose of this investigation was to provide a sound technical evaluation of the rate of rise technique.

The evaluation consisted of the measurement of known outgassing rates for known gases by the rate of rise technique in a vacuum chamber. Three test gases, helium, nitrogen, and methane, were investigated in the range of outgassing rates of 5×10^{-6} to 5×10^{-4} torr-liter-sec^{-1}. The investigations were conducted at room temperature with a maximum measurement error of ±20 percent. The rate of rise technique was found

applicable to the measurement of outgassing rates in the range investigated and for the test gases studied. Errors of the order of a decade in the measured outgassing rate were reported in some cases. These errors were the result of ionization gage pumping during the rate of rise measurements and could be made negligible by the proper selection of pressure rise time and/or by a reduction in the gage emission current. Adsorption was found not to be a factor influencing rate of rise measurements in the range investigated. From the results of the evaluation of the rate of rise technique several general guidelines were established to insure accurate results from a rate of rise investigation.