NASA TECHNICAL NOTE

NASA TN D-4973

C. 1

NASA TN D-4973

EXPERIMENTAL STUDY IN VACUUM OF THE RATE-OF-RISE OUTGASSING-MEASUREMENT TECHNIQUE

by Gerald L. Gregory

Langley Research Center
Langley Station, Hampton, Va.

EXPERIMENTAL STUDY IN VACUUM OF THE RATE-OF-RISE

OUTGASSING-MEASUREMENT TECHNIQUE

By Gerald L. Gregory

Langley Research Center
Langley Station, Hampton, Va.

EXPERIMENTAL STUDY IN VACUUM OF THE RATE-OF-RISE

OUTGASSING-MEASUREMENT TECHNIQUE*

By Gerald L. Gregory
Langley Research Center

SUMMARY

A parametric study was conducted to determine the capabilities of the rate-of-rise outgassing-measurement technique. In the rate-of-rise technique the rate of pressure rise due to outgassing in a closed volume is a measure of the outgassing rate. The study consisted in experimentally measuring, at room temperature, with the rate-of-rise technique known leak rates of nitrogen, methane, and helium gases in the leak range of 5×10^{-6} to 5×10^{-4} torr-liter-second^{-1} and in the high vacuum range of 10^{-6} to 10^{-3} torr. Nitrogen and methane leak rates were measurable to within an experimental accuracy of ±20 percent of the known leak rate; whereas helium leak rates were measurable to within an accuracy of ±25 percent of the known leak rate. Errors as large as a factor of 10 in the measurement of leak rates were found to exist as the result of ionization-gage pumping. These errors were reduced by reducing gage pumping effects. Within the experimental uncertainty of the investigation for the gases considered, adsorption phenomena were not observed to influence rate-of-rise results.

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. In response to this increased need, more emphasis has been placed on the measurement of outgassing rates. From searches of the scientific literature to find a technique for measuring outgassing rates, several methods are found: namely, the mass-loss method, the throughput method, and the rate-of-rise method. The rate-of-rise method is a dynamic technique and has been used by many experimenters (refs. 1 to 5) to measure outgassing rates. To date, much skepticism surrounds the use of the rate-of-rise technique as a result of errors inherent

---

*The information presented herein is taken in part from a thesis entitled, "Experimental Evaluation of the Rate of Rise Technique for Measuring Outgassing Rates in Vacuum," offered as partial fulfillment of the requirements for the degree of Master of Science in Mechanical Engineering, Virginia Polytechnic Institute, Blacksburg, Virginia, April 1967.

in its application. From the current literature on this technique, it is not possible to ascertain the magnitude or importance of these errors; therefore, the purpose of the present investigation is to determine some of the capabilities and limitations of the rate-of-rise technique by experimentally measuring, at room temperature, known leak rates of nitrogen, methane, and helium gases in the leak range of 5×10^{-6} to 5×10^{-4} torr-liter-second^{-1} for the high vacuum range of 10^{-6} to 10^{-3} torr.

SYMBOLS

The units for the physical quantities used herein are presented primarily in the International System of Units (SI). However, in some instances, the units are given in both the SI system and in the U.S. Customary System.

| K | gage constant, torr^{-1} |

| p | pressure, torr |

| dp/dt | rate of change of pressure, torr-second^{-1} |

| Q | total outgassing rate, torr-liter-second^{-1} |

| Q_{ec} | empty-chamber outgassing rate, torr-liter-second^{-1} |

| Q_L | standard leak rate, torr-liter-second^{-1} |

| Q_s | sample or source outgassing rate, torr-liter-second^{-1} |

| S | pumping speed, liter-second^{-1} |

| t | time, seconds or minutes, as noted |

| T | temperature, $^{\circ}$C |

| V | volume, liters |

RATE-OF-RISE TECHNIQUE

The basis of the rate-of-rise technique is the general flow equation

$$Q = V\frac{dp}{dt} + Sp \tag{1}$$

derived (ref. 6) for a typical vacuum chamber. (See fig. 1.) In this equation, Q is the total outgassing rate in the test chamber, V is the volume of the test chamber, dp/dt is the rate of pressure change in the chamber, S is the pumping speed at exit from the chamber, and p is the chamber pressure.

In the application of the rate-of-rise method, the pumping speed S is made zero by isolating the test chamber from the vacuum source, and the resulting equation is

$$Q = V\frac{dp}{dt} \tag{2}$$

As seen from figure 1, Q in equation (2) is composed of two parts: (a) the test-sample or gas-source outgassing rate, which is the rate desired and (b) the empty-chamber outgassing rate. The empty-chamber outgassing rate is evaluated by applying equation (2) to the test chamber with the test sample or gas source removed. With the empty-chamber outgassing rate evaluated, empty-chamber outgassing contributions may be subtracted from the total observed outgassing rate to obtain the test-sample or gas-source outgassing rate.

In addition, any process which results in a value of S different from zero is a violation of the assumption leading to the derivation of equation (2). Two such processes which can occur frequently during a rate-of-rise measurement are adsorption pumping and ionization-gage pumping. Adsorption may occur on the chamber walls as well as on any test samples that may be present. Both adsorption and gage pumping remove gas from the test chamber and result in a reduction in the observed outgassing rate.

Some investigators (refs. 2, 7, and 8) have reported that adsorption effects introduce large errors into rate-of-rise measurements, whereas others (for example, ref. 9) investigating adsorption in general indicate that significant adsorption may occur only at pressures below 10^{-8} torr. The effects of adsorption in a rate-of-rise measurement have not previously been evaluated because adsorption data in the pressure range of 10^{-3} to 10^{-6} torr were lacking.

It is well documented that the operation of an ionization gage in vacuum results in the pumping of gas molecules. Several investigators (refs. 10 to 15) of this pumping action have reported numerous gage-pumping mechanisms and pumping speeds. Because of the complexity of gage pumping and adsorption mechanisms and because, in many cases, the effects of these processes on rate-of-rise measurements cannot be analytically evaluated, an experimental evaluation of the rate-of-rise technique has been conducted at the NASA Langley Research Center and the results are the subject of this report.

DESCRIPTION OF APPARATUS

Vacuum Facility

A photograph of the vacuum facility used in this investigation is shown in figure 2. The test chamber is constructed of 347 stainless steel with an internal volume of 3.57 liters (not including the gage volume of 0.23 liter). A 4-inch (10-cm) air-operated isolation valve provided rapid isolation of the test chamber. The isolation valve incorporates an elastomer O-ring seal, and the actuation mechanism of the valve was differentially pumped to minimize gas loads from the valve during closure. All other chamber vacuum seals were metallic. A 10-inch (25-cm) instrumentation flange formed the upper part of the test chamber. Figure 3 is a photograph of the test chamber and instrumentation flange. The instrumentation consisted of a pressure-recording system, temperature-recording system, and gas-inlet system.

Pressure-Recording System

The pressure in the test chamber was measured with a Bayard-Alpert ionization gage. The gage was mounted with the grid in the vertical position, and the gage features a nonburnout thoria-coated iridium filament. The output from the gage was recorded on a recording oscillograph. The gage was operated with a standard gage controller, and electron bombardment was used as the gage degassing technique.

Temperature-Recording System

Three chromel-alumel thermocouples were mounted in a commercially available thermocouple feedthrough for measuring the temperature of the inside of the chamber wall. Temperatures were recorded on a multichannel recorder. All outgassing studies were conducted at room temperature ($25^\circ \pm 3^\circ$ C).

Gas-Inlet System

The gas-inlet system provides a means of simulating the outgassing rate of a test specimen which is to be measured with the rate-of-rise technique. Problem areas due to interactions between the test specimen and outgassing species may develop in an actual outgassing study which could not be observed in this investigation. These problem areas are pointed out at appropriate parts of the discussion.

The gas-inlet system consisted of a gas-inlet tube and a gas flowmeter. The gas-inlet tube was constructed of 0.46-cm inside-diameter stainless-steel tubing approximately 0.9 meter long. The inlet tube was silver soldered to a 7-cm vacuum flange which was mounted on the 10-inch (25-cm) instrumentation flange. The inlet tube protruded into the test chamber approximately 24 cm. Figure 4 is a photograph of the part of the gas-inlet

4

tube which protruded into the test chamber. Details of the test section of the gas-inlet tube are shown in figures 4 and 5. This section was formed by sealing the open end of the tube with silver solder and by drilling in the periphery of the tube eight holes of 0.055-cm diameter. The gas-inlet tube was located in the approximate center of the test chamber as shown in figure 5. With the gas-inlet tube constructed in this manner, the gas flowed outward radially from the center of the test chamber in a manner similar to the outgassing from a test specimen.

The leak rates of gas flowing into the test chamber were measured with a gas flow-meter. The operating console of the flowmeter is shown in figure 6. The gas flowmeter is commercially available and of constant-pressure design (ref. 16), 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 chamber. The gas flowmeter sets and measures flow rates in the range of 2×10^{-1} to 5×10^{-6} torr-liter-second^{-1}.

Test Gases

Three test gases were selected for the evaluation of the rate-of-rise technique: nitrogen, methane, and helium. Helium was selected as a test gas to permit the evaluation of the technique for a gas which is not readily pumped by the gage nor adsorbed by the chamber walls. Nitrogen was selected because of the sizable quantity of data in the literature on gage pumping of nitrogen. In addition nitrogen is frequently observed as an outgassing species. Methane permitted an evaluation of the technique for a gas of hydrocarbon composition. The impurity contents of the test gases are given in table 1.

TEST PROGRAM AND PROCEDURES

Introduction

The normal procedure in applying the rate-of-rise technique to the measurement of an outgassing rate is to place the test specimen in a vacuum chamber and then to exhaust the chamber to the desired pressure level. When a measurement is to be taken, a valve is closed to isolate the test chamber and specimen from the pump, and the chamber pressure is allowed to rise. The outgassing rate of the specimen and chamber is calculated from the pressure behavior according to equation (2). Then, the chamber outgassing rate (which can be found from rate-of-rise measurements of the empty chamber) is subtracted from the total (specimen and chamber) outgassing rate to determine the outgassing rate of the test specimen.

For these experimental studies, the outgassing rate of the test specimen is simulated by use of the gas flowmeter and gas-inlet tube. The test program consisted of leaking into the test chamber known gases at specific leak rates and then measuring, at

room temperature, these leak rates with the rate-of-rise technique. Deviations between the known leak values and those measured by the rate-of-rise technique were evaluated in terms of the various phenomena that could cause errors and inaccuracies in the rate-of-rise technique. As a means of evaluating the errors and inaccuracies, various test parameters were varied.

Test Parameters

Five test parameters were varied, as follows:

(1) Gas species

(2) Time interval between gage electron bombardment (degassing) and rate-of-rise measurement (time after gage degas)

(3) Ionization-gage emission current

(4) Pressure range for rate-of-rise measurement

(5) Magnitude of standard leak

Procedures

Prior to the evaluation of the rate-of-rise technique, the test chamber, ionization gage, and gas-inlet tube were baked for approximately 200 hours at 200° C. After the baking was complete, the vacuum chamber was examined for leaks by using a helium leak detector with a sensitivity of 10^{-10} torr-liter-second^{-1}. Leakage through the closure of the air-operated isolation valve was examined to insure that the valve was properly seated. The test program was started only after the leak detector indicated that all leaks present were negligible (less than 10^{-10} torr-liter-second^{-1}). After the chamber was checked for leakage, the liquid nitrogen trap was filled and maintained full throughout the test evaluations.

A generalized test procedure was used for each rate-of-rise measurement and was as follows: The ionization gage was electron bombarded (degassed) for 10 minutes with a grid potential of 800 volts and a grid current of 90 to 100 milliamperes. At the end of gage degassing, the gage was allowed to cool. During this cooling time (5 to 60 minutes, depending on this test parameter), the liquid nitrogen trap was manually overfilled, and the gage emission current was adjusted to test value. At the end of the gage cooling period, the isolation valve was closed and the chamber pressure was allowed to rise a predetermined amount. After the pressure rise was completed (4 to 130 seconds were required for this pressure rise, depending on test parameters), the isolation valve was opened, and the test chamber was returned to the initial pressure that existed prior to valve closure. This sequence of events constituted a rate-of-rise measurement.

The procedures were as follows:

(1) Before the test gas was leaked into the test chamber, a rate-of-rise measurement was taken to identify the empty-chamber outgassing rate. Empty-chamber measurements were taken 20 minutes after the end of gage degassing (that is, the gage was allowed to cool 20 minutes before valve closure) and at 1-milliampere gage emission current. The pressure was allowed to increase from 10^{-7} to 10^{-4} torr and the empty-chamber outgassing rate was typically 8×10^{-7} torr-liter-second^{-1} as determined from the 10^{-7} decade (10^{-7} to 10^{-6} torr) pressure rise.

(2) After the empty-chamber outgassing measurement, a specific leak rate of test gas was set on the gas flowmeter, and the test gas was leaked into the test chamber at this rate. Test gas continuously leaked into the test chamber with only slight adjustments being necessary to the flowmeter to maintain the desired leak rate.

(3) After setting the leak rate on the flowmeter, eight rate-of-rise measurements were made at various times ranging from 5 to 60 minutes after gage degassing. The generalized test procedure was used for each rate-of-rise measurement, and 30 minutes were allowed between the opening of the isolation valve of one measurement and the start of electron bombardment for the next measurement. (In all cases the 30-minute allowance was more than sufficient for the chamber pressure to return to the value which existed prior to valve closure.) For each measurement, pressure-rise data were recorded across one decade of pressure. Gas flowmeter measurements of the set leak rate were made during the time interval between the end of gage degassing and the start of the rate-of-rise measurement.

(4) At the end of these eight rate-of-rise measurements, the leak rate of test gas into the chamber was cut off and approximately 14 hours were allowed for the vacuum system to reach its base pressure (typically 5×10^{-9} torr) before another test sequence was initiated.

The foregoing test procedures were repeated 28 times for the purpose of investigating the test parameters.

PRECISION AND ACCURACY

In general, the uncertainty levels quoted in this section are the maximum expected deviations of the measured parameter indication.

Standard Leak Rate

The standard leak rates were measured with the gas flowmeter with an uncertainty of ±5 percent. Both the physical limitations of the flowmeter and the observation

uncertainties in the actual data measurements were considered. Leak rates are reported in torr-liter-second^{-1} units at 20° C.

Pressure Measurements

The ionization gage was calibrated by a gas flow rate-orifice-conductance calibration system (ref. 16) with an uncertainty level of ±8 percent. Gage constants (torr^{-1}) for nitrogen and methane were obtained directly from a calibration at 10 milliamperes of emission current. The gage constant for helium was calculated from the nitrogen gage constant and the ratio of helium to nitrogen constants reported in the literature (ref. 17). The helium gage constant is estimated to be accurate to ±13 percent. Gage constants for the Bayard-Alpert gage are sensitive to emission current variations (ref. 18) and, on an average, gage constants at 1 milliampere of emission current are 18 percent higher than constants at 10 milliamperes of emission current. The gage constants at 1 milliampere were assumed to be 18 percent higher than the constants obtained by calibration at 10 milliamperes of emission current. From the results reported in reference 18 and the gages and pressure ranges used in these studies, the 10-milliampere and 1-milliampere gage constants are assumed to have the same uncertainty levels. The appropriate gage constant was used for each rate-of-rise measurement, and typical gage constants are shown in table 2.

Because of the lack of the knowledge of the gas composition of the outgassing from the test-chamber walls, the gage constant for nitrogen was assumed to apply in reporting empty-chamber outgassing rates. Empty-chamber outgassing rates are consequently reported in equivalent nitrogen pressure units. For most gas standard leak measurements the empty-chamber outgassing rate was found to be small as compared with the magnitude of the test-gas leak rate. (That is, 10 percent or less of the total gage indication during a rate-of-rise measurement was due to empty-chamber outgassing.) Hence, any uncertainties in the rate-of-rise measurements of the standard leak rates as the result of subtracting the empty-chamber outgassing from the observed rate-of-rise measurements were considered to be negligible unless stated otherwise.

A criterion was not developed for determining the uncertainties incurred in pressure measurements due to variations in gage cleanliness; however, a technique was developed to minimize gage cleanliness effects. This technique consisted of electron bombardment of the gage prior to each rate-of-rise measurement for 10 minutes with 6 volts on the filament and an 800-volt potential between filament and grid with the stipulation that the grid current reached 90 to 100 milliamperes during this period. With this technique, the gage was assumed to be at the same degree of cleanliness for corresponding times after bombardment (degassing).

Volume Measurement

The volume of the test chamber, including test-gage volume, was measured by a water-fill technique and was found to be 3.8 liters with an estimated uncertainty of 1/2 percent.

Pressure-Recording Oscillograph

The output from the ionization gage was recorded on a recording oscillograph. The oscillograph features a D'Arsonval-type galvanometer with an uncertainty level of less than 1/2 percent of full scale. The recorder has a full-scale (10-cm) rise time of 12 milliseconds and was calibrated daily with a deflection sensitivity of 10 milliamperes per centimeter. From consideration of the limitations of the recorder and the uncertainties in reading slopes from the oscillographs, the pressure-time slopes dp/dt used in the rate-of-rise equation (eq. (2)) were readable to within an accuracy of approximately ± 6 percent.

Overall Accuracy

On the basis of the uncertainties in the parameters just discussed, the outgassing rates are considered to be measurable to within an accuracy of ± 15 percent for nitrogen and methane and within an accuracy of ± 20 percent for helium. If the 5-percent error from the standard leak measurements is considered, the observed values of the simulated outgassing rates should be within ± 20 percent for nitrogen and methane and within ± 25 percent for helium of the standard leak set on the gas flowmeter. Any additional deviation in the rate-of-rise measurements and the standard leak values will be investigated from the standpoint of violation of the assumption that the pumping speed equals zero during a rate-of-rise measurement.

PRESSURE TRACES AND DATA REDUCTION

Pressure Traces

The rate-of-rise equation (eq. (2)) was derived on the assumption that the pumping speed S in equation (1) was zero. In the actual measurement process of the rate of pressure rise, there are certain processes which could violate the assumption that S is zero. In the cases where S is not zero, the error introduced into a rate-of-rise measurement depends on the magnitude of the product Sp (eq. (1)) as compared with the leak rate or outgassing rate being measured. Therefore, S can be so small that the assumption that S is approximately zero appears valid and still large errors may result from the product Sp. In order to determine the effects of the product Sp on the pressure-time traces taken during a rate-of-rise measurement, pressure-time traces

were calculated from equation (1) by assuming three pumping-speed relationships and typical pumping speed and pressure values. The three pumping-speed relationships were (a) $S = 0$, (b) S = Constant, and (c) S varies as an exponential decaying time function. Figure 7 presents three traces which were calculated for particular test conditions and are typical for the range of test parameters being investigated. Curve (a) is the ideal trace where the product Sp is zero. When the pumping speed is other than zero, the calculated pressure-time trace deviates from the linear trace denoted by curve (a). At any particular time, the difference between curve (a) and curve (b) or curve (a) and curve (c) is an indication of the quantity of gas that has been removed from the test volume as a result of the product Sp. It might be noted that after a long valve-closure time, curves (a) and (c) will be parallel.

For the purpose of gaining some insight into the type of pumping that may be taking place during the rate-of-rise measurement, the experimental pressure-time traces were grouped and compared with the calculated traces. Three general types of pressure traces were observed during rate-of-rise measurements and are shown in figure 8. Figure 8(a) is a linear trace which was most frequently observed for helium gas measurements and is similar to the zero pumping speed trace in figure 7. Figure 8(b) shows a nonlinear trace which is similar to the constant pumping speed trace in figure 7. This trace was observed in only a few instances. Figure 8(c) shows a nonlinear trace which does not have a counterpart in the calculated traces. However, if the initial valve closure effects are neglected, it is similar to the constant pumping speed trace in figure 7. This type of trace was most frequently observed for the nitrogen and methane measurements. During the experimental measurements, no traces that were similar to the exponential decreasing time function trace in figure 7 (curve (c)) were observed.

The type of pumping-speed behavior occurring in the test chamber cannot be determined solely by a comparison of observed and calculated pressure traces. However, a comparison of pressure traces in this manner is helpful in supporting any conclusions drawn as to the nature of the pumping process taking place.

Data Reduction

The pressure-time slopes used in equation (2) for the rate-of-rise calculations were arbitrarily taken as the slope which existed between 8 and 10 on the pressure decade (except as noted otherwise) where the measurement was taken. (See fig. 8.) The pressure slopes as read from the oscillograph records were corrected for empty-chamber outgassing and for gage calibration before they were used in equation (2). The gage-calibration equation is

$$\left(\frac{dp}{dt}\right)_{true} = \left(\frac{dp}{dt}\right)_{indicated} \frac{10}{K} \tag{3}$$

where 10 is the manufacturer's assumed gage constant and K is the appropriate gage constant from table 2.

RESULTS AND DISCUSSION

General Remarks

Figures 9, 10, and 11 present the results of rate-of-rise measurements of nitrogen, methane, and helium gas standard leaks, respectively. Each of these plots shows rate-of-rise measurements taken to measure a specific leak rate at a given pressure level. The ordinate and abscissa of each plot are leak rate and time after gage degas, respectively. The dashed line in each plot represents the magnitude of the standard leak rate and is an arithmetic average of the gas flowmeter readings for the measurements. (See "Average standard leak rate" column in tables 3, 4, and 5.) In the case where the results for two emission currents are shown on one plot, the dashed line is the average of flowmeter readings for both sets of measurements and the ± indication following the stated magnitude of the dashed line is the variation of individual standard leak averages from the total average. The pressure scale shown on each plot is the indicated pressure scale (prior to gage calibration) across which the rate-of-rise measurements were taken. The results shown in figures 9, 10, and 11 are tabulated in tables 3, 4, and 5, respectively.

Effects of Time After Gage Degassing

One of the problems associated with the ionization gage is the contamination of the gage elements after prolonged usage, which requires periodic degassing of the gage. Because a newly degassed gage requires a finite time to return to pressure and temperature equilibrium with the system, the effects of the time interval between the end of gage degassing and the time at which rate-of-rise measurements are made (time after degassing) were investigated. These times ranged from 5 to 60 minutes. As can be seen from figures 9, 10, and 11, in most cases, the time after gage degassing had no influence on rate-of-rise measurements.

In figure 10(c) for methane, a variation in rate-of-rise measurements as the result of time after degassing was found to exist. For this case rate-of-rise measurements increase approximately 700 percent between 5 and 20 minutes after degassing and then level off after 20 minutes to about 2.3×10^{-6} torr-liter-second^{-1}. The explanation for this phenomenon is believed to be associated with the behavior of gage pumping speed for methane gas as a function of time and with the magnitude of the Sp product as compared with the standard leak rate. (Throughout the remainder of this text the phrase "high Sp product" carries with it the connotation that the Sp product is a large percentage of the standard leak rate.) It seems plausible that a high Sp product and a gage pumping speed that decreases with time and then saturates to a constant value would account for

the behavior observed in figure 10(c). Unavailability of information concerning the behavior of gage pumping speed with time for methane gas does not permit substantiation of this explanation. However, it is observed that this phenomenon is not present in the other methane measurements of 10 milliamperes where the Sp products are a smaller percentage of the standard leak value. (See table 6 for the magnitude of the methane Sp products.) In any event, for the 10-milliampere measurements of figure 10(c) after 20 minutes, rate-of-rise measurements are not influenced by time after degassing.

In summary, for all but one case, rate-of-rise measurements can be taken at any time from 5 to 60 minutes after gage degassing. For the one exception, it was found that for best results rate-of-rise measurements can be taken from 20 to 60 minutes after gage degassing.

<center>Gage Emission-Current Effects</center>

General comments.- Although various pumping mechanisms and a variety of pumping speeds may exist for gages, it is well documented that gage pumping speed is sensitive to gage emission-current variations. Greenwood (ref. 5), in investigating the outgassing rate of solid propellant fuels with the rate-of-rise technique, showed that known air standard leaks could be more accurately measured by reducing gage emission current. In the present investigation, the leak rates of 5×10^{-6}, 1×10^{-5}, and 5×10^{-5} torr-liter-second^{-1} were measured at two values of gage emission current, 10 milliamperes and 1 milliampere. Parts (a), (b), (c), and (d) of figures 9, 10, and 11 show the results of these measurements. (Note that for helium, figs. 11(a) and 11(b), measurements at only one emission current are reported. The measurements at the other emission currents have been omitted because of an observed malfunction of the gas flowmeter.) In all cases the rate-of-rise measurements at 10 milliamperes show lower values of leak rate than the measurements at 1 milliampere, so that the Sp product is larger at 10 milliamperes. This higher Sp product is the result of the gage having a higher pumping speed at 10 milliamperes. (It is shown subsequently that the pumping mechanism occurring in the test chamber is gage pumping.) The 1-milliampere measurements of figures 9, 10, and 11 represent the most accurate attempt to measure a given leak rate and, except for figure 9(b), the 1-milliampere measurements of the standard leak rates give the most accurate results. In addition the Sp products associated with the 1-milliampere measurements are less than the experimental uncertainty and all 1-milliampere measurements were observed to lie within the experimental uncertainty band, that is, within ±20 percent of the standard leak rate for nitrogen and methane, and within ±25 percent for helium. In order to gain a better understanding of the importance of the Sp product, the Sp products associated with these emission-current measurements are now discussed in some detail for each of the test gases.

Nitrogen.- For nitrogen gas measurements, the high Sp product at a current of 10 milliamperes introduced errors into rate-of-rise measurements as large as a factor of 10, as shown in figure 9(c). In figure 9(c) the Sp product at 10 milliamperes is approximately 90 percent of the standard leak rate being measured (see table 7), whereas for the corresponding 1-milliampere measurements the Sp product is less than 20 percent of the standard leak rate.

Gage pumping speeds (from ref. 15) for nitrogen gas and for a Bayard-Alpert gage of the type used in these investigations are 0.14 and 0.02 liter-second^{-1} at 10-milliampere and 1-milliampere emission current, respectively. The 10-milliampere rate-of-rise measurements reported in figure 9 can be evaluated analytically from equation (1) by considering gage pumping as the gas removal process. In this analytical treatment, the standard leak rate of the test gas is compared with a calculated leak rate based on the average observed $V \frac{dp}{dt}$ as plotted in figure 9 and the calculated Sp product. The Sp term is obtained by assuming S to be gage pumping at a constant value of 0.14 liter-second^{-1} and the chamber pressure p to be that pressure at which dp/dt was read from the experimental pressure traces. From table 7 it is evident that when gage pumping was assumed as the pumping process, the calculated leak rates agreed with the standard leak rates to within the ±20 percent experimental uncertainty. The 1-milliampere nitrogen measurements were evaluated in the manner of table 7 with the result that the analytical leak rates agreed with the standard leak rates. It should be noted that 10-milliampere measurements can result in accurate measurements of the standard leak rates provided the Sp product is small as compared with the leak rate. This result is evident from figure 9(b) where the 10-milliampere measurements give a more accurate measurement of the standard leak rate than the 1-milliampere measurements. The Sp product associated with the 10-milliampere measurements of figure 9(b) is only 15 percent of the standard leak rate.

Methane.- Rate-of-rise measurements of methane resulted in measurements lower than the standard leak rate by as much as a factor of 5. (See fig. 10(c).) In figure 10(c), the Sp product is 60 percent of the standard leak rate. (See table 6.) Note that this measurement is the only one at 10 milliamperes for methane in which the Sp product is greater than the experimental uncertainty. Since values of gage pumping speed for methane were not found in the literature, it is necessary to estimate the gage pumping speed (if gage pumping is assumed to be the only pumping mechanism involved) from the experimental data. This estimation consists in finding what constant value of S in equation (1) will result in pressure traces similar to those observed experimentally. The gage pumping speed at 10 milliamperes of emission current is of the most interest as it results in the larger Sp products. Figure 12 presents a comparison of these pressure traces as discussed for the 10-milliampere measurements of figure 10(c). From

figure 12 the gage pumping speed for methane at 10 milliamperes is estimated to be 0.09 liter-second^{-1}. This value was substantiated by the other 10-milliampere measurements. Also note that this value is reasonable for gage pumping speed, and hence the assumption that the pressure traces of figure 12 are the result of gage pumping appears valid. With this value of pumping speed used, table 6 is constructed and shows that, to within 20 percent of the experimental uncertainty, gage pumping is the only pumping mechanism occurring in the test chamber. In a similar manner, it was confirmed that gage pumping accounted for the behavior of the 1-milliampere measurements of methane standard leak rates.

Helium.- For helium gas the 10-milliampere and 1-milliampere rate-of-rise measurements report approximately the same value of leak rate. (See figs. 11(c) and 11(d).) This result is expected, as the gage pumping speed for helium (ref. 15) at 10 milliamperes is only 0.04 liter-second^{-1}, and hence the Sp products for helium are small at both emission currents. Evaluation of the Sp products for helium gas measurements confirmed that gage pumping was the mechanism occurring in the test chamber.

Concluding remarks on gage emission current.- The effect of gage emission current on rate-of-rise measurements of gas standard leaks is that increasing gage emission current results in increasing the magnitude of the gage pumping speed. This effect, in turn, increases the magnitude of the Sp products associated with the measurements and results in lower reported values of leak rate. The magnitude of the Sp product as compared with the leak value to be measured controls the degree of accuracy of rate-of-rise measurements. The absolute value of the gage pumping speed itself is not the criterion for determining the accuracy of a leak-rate measurement, as it was shown that 10-milliampere and 1-milliampere measurements resulted in equally valid leak rates provided the Sp product was small.

<center>Pressure Effects</center>

The effects of the pressure at which rate-of-rise measurements are taken can be seen by a comparison of the nitrogen measurements at 10 milliamperes in figures 9(b) and 9(c). Figure 13 presents such a comparison. The test environments for these measurements are similar except for the pressure scale across which the rate-of-rise pressure traces were recorded. The pressure values shown in figure 13 are the pressures (true) at which dp/dt was read from the experimental pressure traces. The leak rates reported at the higher pressure (6.54×10^{-5} torr) are approximately a factor of 10 lower than the standard leak rate, whereas the lower pressure measurements (1.10×10^{-5} torr) are of the same magnitude as the standard leak rate. Therefore, the error reported by the 6.54×10^{-5} torr measurements is substantially decreased by reducing the pressure at which the rate-of-rise measurements are made. Since the ionization gage was operated at 10 milliamperes for both sets of measurements, the phenomena observed in figure 13

14

cannot be attributed to a change in the magnitude of the gage pumping speed. However, as seen from table 7, the Sp product of the 6.54×10^{-5} torr measurements is substantially higher (90 percent of the standard leak rate) than that for the 1.10×10^{-5} torr measurements (15 percent of the standard leak rate). In addition, table 7 does confirm that gage pumping is the pumping process that is responsible for this phenomenon; therefore, the larger Sp product associated with the higher pressure measurements is the result of the gage pumping more gas at the higher pressure level and thus is not as observable at the lower pressure level. The conclusion that gage pumping is the controlling mechanism is further corroborated by figures 14 and 15 in which typical experimental traces obtained for the 6.54×10^{-5} and 1.10×10^{-5} torr measurements, respectively, are compared with calculated pressure traces. The experimental and calculated pressure traces are noted to agree well throughout the trace time. The calculated pressure-time traces for these figures were obtained by integration of equation (1) in which S was assumed to be a constant at 0.14 liter-second^{-1} and all other parameters were considered similar to those used in the experimental investigation. The same type of pressure effects are observed for the methane gas measurements. (See figs. 10(b) and 10(c).) Figure 16 is a comparison of the 10-milliampere measurements from these two plots. In this case the higher pressure measurements report leak rates that are a factor of 5 lower than the standard leak rate. (Disregard the 5-, 10-, and 15-minute data points for the high pressure measurements, as their behavior has been attributed to effects of time after degassing.) The phenomenon observed in figure 16, again, is attributed to the gage pumping more gas at a higher pressure level and can be verified by table 6 and by a comparison of the experimental and calculated pressure traces as was done for nitrogen. (This comparison of pressure traces is not presented as part of this text.)

It might be mentioned here that pressure effects are not observed in the 1-milliampere measurements of figures 8(b), 8(c), 9(b), and 9(c), because for these measurements the pumping speed of the gage is sufficiently small that higher pressures than those investigated for the particular test conditions would have to be considered before the pressure effect can be observed.

Because of the low value of the Sp product and since only 1-milliampere measurements are presented in figure 11(b), the effects of pressure on helium rate-of-rise measurements were not ascertained. However, it is reasonable to assume that the pressure effects for helium are similar to those observed for nitrogen and methane.

The effects of pressure on rate-of-rise measurements of the standard leak rates can be summarized by saying that increasing the pressure at which rate-of-rise measurements are taken results in the ionization gage pumping more gas. This effect, in turn, increases the magnitude of the Sp product and results in lower values of leak rates.

Effects of Standard Leak Magnitude

Five values of standard leak rates were investigated to evaluate the sensitivity of rate-of-rise measurements to the magnitude of the leak rate: 5×10^{-6}, 1×10^{-5}, 5×10^{-5}, 1×10^{-4}, and 5×10^{-4} torr-liter-second^{-1}. The absolute value of the standard leak rate did not influence the ability of the rate-of-rise technique to measure the given leak rate. All but one (standard leak rate of 5×10^{-6} torr-liter-second^{-1} for helium) of the leak-rate—test-gas combinations were measured to within the respective experimental uncertainty bands. However, it was observed that the relative magnitude of the standard leak as compared with the Sp product and the empty-chamber outgassing rate affected the rate-of-rise results. In cases where the Sp product was a large percentage of the leak rate being measured, the reported values of leak rates were as much as a factor of 10 lower than the standard leak rate. In general, the desired accuracy of a rate-of-rise measurement depends upon the acceptable magnitude (as compared with the leak rate being measured) of the Sp product. For these investigations an acceptable magnitude of the Sp product was found to be the value of the experimental uncertainties.

For all but one of the standard leak measurements, the empty-chamber contributions to the rate-of-rise measurements are approximately 10 percent or less of the total measurement. The one exception is the 10-milliampere measurement for helium shown in figure 11(a). For these measurements the relative magnitudes of the empty-chamber outgassing and the standard leak rate and the low sensitivity of the gage for helium are such that 30 percent of the gage indication during these measurements is the result of empty-chamber outgassing. As a result, the reported values of leak rate are 42 percent lower than the standard leak value. This is well outside of the ±25 percent experimental uncertainty. The 17-percent (42 - 25) discrepancy could not be justified by considering the Sp product for these measurements. However, this 17-percent discrepancy is believed to be due to additional inaccessible uncertainties introduced into the reported values as a result of the high percentage of empty-chamber outgassing contributions and to the fact that the true gage constant for the empty-chamber outgassing is not known. In addition, part of this discrepancy is attributed to gage pumping of the empty-chamber outgassing gas species. In general this problem was not encountered when the empty-chamber outgassing rate contributes less than 10 percent of the total gage indication. A word of caution is extended here; for best results it is advisable to have empty-chamber outgassing contributions to the rate-of-rise measurement less than 10 percent in order to insure that additional inaccessible uncertainties are not introduced.

Summary of Gage Pumping Effects

As seen from equation (1) and the data already presented, the accuracy to which a specific leak rate is measured depends largely upon the magnitude of the Sp product as

compared with the magnitude of the leak rate. In these investigations, Sp products as large as 90 percent of the standard leak rate were observed, and in these cases, rate-of-rise measurements were found to be as much as a factor of 10 lower than the true leak rate. The source of the Sp product for these investigations was ionization-gage pumping. This was verified in two ways: (1) Comparisons of experimentally obtained rate-of-rise pressure-time traces with calculated pressure-time traces (based on gage pumping) agree to well within experimental uncertainty. (2) Discrepancies between observed values of $V \frac{dp}{dt}$ and known standard leak rates were accounted for by the Sp product associated with the ionization gage. In each case, the Sp product was reduced by reducing either the gage pumping speed or the pressure at which rate-of-rise measurements were taken (or both). It has been shown that standard leak rates can be determined to within the experimental accuracy even in the cases where the Sp product is large, but for these cases the behavior of the pumping mechanism as well as its magnitude must be known in order to make the appropriate corrections to the experimental data. In the event that these pumping mechanisms are unknown, a practical upper limit on the magnitude of the Sp product is the value of the experimental uncertainty.

Adsorption Effects

In every leak-rate measurement, the discrepancy between the standard leak rate and the rate-of-rise measurement was attributed to gage pumping and experimental uncertainty. Therefore, within the limits of the accuracy of these measurements no adsorption phenomena were observed during this rate-of-rise investigation.

In the case of an actual outgassing investigation, adsorption effects may be introduced as the result of gas interactions with the surface of the test specimen. In addition, rate-of-rise measurements at lower pressure levels and for other gas species as well as in test chambers of materials other than stainless steel may introduce adsorption effects.

CONCLUSIONS

The experimental study of the rate-of-rise technique for measuring nitrogen, methane, and helium gas standard leak rates at room temperature in the leak range of 5×10^{-6} to 5×10^{-4} torr-liter-second^{-1} and for a pressure range of 10^{-6} to 10^{-3} torr resulted in the following conclusions:

(1) Standard leak rates of nitrogen and methane are measurable to within ±20 percent. All helium leak rates except for the leak rate of 5×10^{-6} torr-liter-second^{-1} were measurable to within ±25 percent of the standard leak rate. (The experimental uncertainty associated with the nitrogen and methane measurements is ±20 percent and the experimental uncertainty associated with helium is ±25 percent.)

(2) The Sp product (where S is the magnitude of the pumping process removing gas from the closed test chamber and p is the pressure at which this pumping occurs) is the primary influence on rate-of-rise measurement accuracy.

(3) Rate-of-rise measurements can be influenced by ionization-gage pumping which would result in an error of as much as a factor of 10 in the reported values of standard leak rates.

(4) Errors due to ionization-gage pumping are experimentally minimized by reducing the gage pumping speed (that is, by reducing gage emission current) and/or by decreasing the pressure at which rate-of-rise measurements are taken.

(5) Errors due to ionization-gage pumping can be analytically evaluated so that accurate leak-rate values may be obtained, provided the gage pumping characteristics are known and appropriate corrections are applied to the experimental data.

(6) For the test gases investigated, adsorption phenomena are not observed to be influencing rate-of-rise measurements of gas standard leak rates.

Langley Research Center,
National Aeronautics and Space Administration,
Langley Station, Hampton, Va., August 29, 1968,
124-09-19-01-23.

REFERENCES

1. Lennert, A. E.; Trolinger, J. D.; and Shipp, J. I.: Ion Engine Outgassing Tests Under Simulated Launch Conditions. AEDC-TDR-63-196, U.S. Air Force, Oct. 1963. (Available from DDC as AD 433898.)

2. Rogers, Kenneth W.: An Investigation of Factors Influencing the Outgassing Rate. USCEC Rep. 77-101, Eng. Center, Univ. of Southern California, Sept. 30, 1963.

3. Schrank, M. P.; Benner, F. C.; and Das, D. K.: Theoretical and Experimental Study to Determine Outgassing Characteristics of Various Materials. AEDC-TDR-64-53, U.S. Air Force, Mar. 1964. (Available from DDC as AD 433484.)

4. Mimura, Takuo; Anagnostou, Evelyn; and Colarusso, Paul E.: Thermal Radiation Absorptance and Vacuum Outgassing Characteristics of Several Metallic and Coated Surfaces. NASA TN D-3234, 1966.

5. Greenwood, Lawrence R.: Outgassing Rates of Two Solid Rocket Fuels in Vacuum. NASA TN D-3790, 1967.

6. Greenwood, Lawrence Ronald: Determination of the Outgassing Rate of Two Solid Propellant Rocket Fuels in a Vacuum. M. S. Thesis, Virginia Polytech. Inst., 1965.

7. Markley, F.; Roman, R.; and Vosecek, R.: Outgassing Data for Several Epoxy Resins and Rubbers for the Zero Gradient Synchrotron. 1961 Transactions of the Eighth Vacuum Symposium and Second International Congress, Vol. 1, Luther E. Preuss, ed., Macmillan Co., 1962, pp. 78-85.

8. Shipp, J. I.; and Trolinger, J. D.: Analysis of Methods for Determining Outgassing Rates. AEDC-TDR-64-180, U.S. Air Force, Dec. 1964.

9. Wightman, James P.; and Mugler, John P., Jr.: The Adsorption of Argon, Helium, and Nitrogen on Stainless Steel at Room Temperature in the Pressure Range 10^{-8} to 10^{-7} Torr. Paper presented at 152nd National Meeting, Amer. Chem. Soc. (New York, N.Y.), Sept. 11-16, 1966.

10. Hobson, J. P.: The Pumping of Nitrogen by a Bayard-Alpert Ionization Gauge in an Ultra-High-Vacuum System. Vacuum, vol. 11, no. 1, 1961, pp. 16-24.

11. Varnerin, L. J., Jr.; and Carmichael, J. H.: Ionic Pumping Mechanism of Helium in an Ionization Gauge. J. Appl. Phys., vol. 26, no. 6, June 1955, pp. 782-783.

12. Cobic, B.; Carter, G.; and Leck, J. H.: Pumping of Argon, Nitrogen and Hydrogen in a Bayard-Alpert Gauge. Brit. J. Appl. Phys., vol. 12, no. 8, Aug. 1961, pp. 384-389.

13. Carter, G.; and Leck, J. H.: A Study of the Mechanism of Ion Pumping for the Noble Gases. Proc. Roy. Soc. (London), ser. A, vol. 261, no. 1306, May 16, 1961, pp. 303-315.

14. Robinson, N. W.; and Berz, F.: Initial Pumping and Recovery of Ionization Gauges. Vacuum, vol. 9, no. 1, 1959, pp. 48-53.

15. Byvik, Charles E.; and Bradford, James M.: Pumping of Common Gases by Ionization Gauges Used in Space Simulation Facilities. AIAA/IES/ASTM Space Simulation Conference, Sept. 1966, pp. 209-214.

16. Hayward, W. H.; and Jepsen, R. L.: A Simple High Vacuum Gauge Calibration System. 1962 Transactions of the Ninth National Vacuum Symposium of the American Vacuum Society, George H. Bancroft, ed., Macmillan Co., c.1962, pp. 459-462.

17. Marton, C.: Advances in Electronics and Electron Physics. Vol. 17, Acad. Press, 1962.

18. Melfi, Leonard T., Jr.; and Kern, Frederick A.: Variations in Gage Constant as a Function of Emission Current in an Unshielded Open-End Grid Bayard-Alpert Ionization Gage. NASA TN D-3811, 1967.

TABLE 1.- IMPURITY CONTENT OF TEST GASES

Test gas	Impurity	Concentration, parts per million
Helium	Air	20
	Others	480
Nitrogen (Seaford grade)	Oxygen	20
	Hydrogen	500
	Argon	600
	Water	(a)
Methane (c.p. grade)	Ethane	2200
	Nitrogen	7900
	Propane	160
	Carbon dioxide	100
	Oxygen + Argon	50
	Water	3

[a] 0.002 percent by weight at 55° C dewpoint.

TABLE 2.- TYPICAL IONIZATION GAGE CONSTANTS

Gas species	Gage constant, K, torr^{-1}		Uncertainty, percent
	at 10 mA	at 1 mA	
Helium	1.61	1.90	±13
Nitrogen	8.06	9.51	±8
Methane	13.70	16.17	±8

TABLE 3.- TEST RESULTS FOR NITROGEN

Figure	Average standard leak rate, Q_L, torr-liter-sec^{-1}	Empty-chamber outgassing rate, Q_{ec}, torr-liter-sec^{-1}	Emission current, mA	Pressure range, torr	Measurement method	Leak rate, μtorr-liter-sec^{-1}, at time (min) after gage degas							
						5	10	15	20	30	40	50	60
9(a)	4.97×10^{-6}	5.64×10^{-7}	10	10^{-6}	Gas flowmeter	4.94	4.89	4.75	4.85	5.03	5.23	5.01	5.03
					Rate of rise	2.83	3.21	3.27	3.19	3.08	3.21	3.21	3.39
9(a)	5.49×10^{-6}	5.68×10^{-7}	1	10^{-6}	Gas flowmeter	5.32	5.36	5.58	5.55	5.63	5.51	5.43	5.53
					Rate of rise	4.73	4.95	5.22	5.42	5.72	5.53	5.66	5.44
9(b)	1.02×10^{-5}	5.95×10^{-7}	10	10^{-6}	Gas flowmeter	10.1	10.4	10.1	10.2	10.0	10.3	10.1	10.2
					Rate of rise	10.1	10.0	10.0	9.98	9.99	9.99	9.96	9.98
9(b)	1.04×10^{-5}	6.77×10^{-7}	1	10^{-6}	Gas flowmeter	10.4	10.4	10.4	10.4	10.4	10.6	10.3	10.3
					Rate of rise	12.2	12.5	11.9	12.9	12.5	12.5	12.9	11.5
9(c)	1.01×10^{-5}	8.44×10^{-7}	10	10^{-5}	Gas flowmeter	10.1	9.90	10.1	10.1	10.2	10.1	10.1	9.82
					Rate of rise	0.800	0.990	1.13	1.02	1.04	1.05	1.90	0.990
9(c)	1.01×10^{-5}	7.64×10^{-7}	1	10^{-5}	Gas flowmeter	10.2	10.1	10.1	10.1	10.1	10.2	10.1	10.0
					Rate of rise	7.60	7.26	7.77	7.52	8.45	8.77	8.53	7.93
9(d)	4.97×10^{-5}	9.67×10^{-7}	10	10^{-5}	Gas flowmeter	50.2	49.4	50.2	49.0	49.7	49.2	50.0	49.8
					Rate of rise	46.8	42.3	43.6	41.8	43.2	42.8	42.2	44.1
9(d)	5.14×10^{-5}	8.86×10^{-7}	1	10^{-5}	Gas flowmeter	51.7	51.3	51.2	51.5	51.5	51.4	51.6	50.8
					Rate of rise	53.6	56.1	60.0	56.1	60.6	59.1	53.6	51.2
9(e)	1.00×10^{-4}	1.06×10^{-6}	1	10^{-4}	Gas flowmeter	100	100	100	101	99.0	100	101	100
					Rate of rise	91.0	94.3	97.6	94.3	92.7	95.1	99.2	97.6
9(f)	5.10×10^{-4}	5.67×10^{-7}	1	10^{-4}	Gas flowmeter	514	505	505	508	523	508	508	510
					Rate of rise	510	503	493	513	545	503	509	514

TABLE 4.- TEST RESULTS FOR METHANE

Figure	Average standard leak rate, Q_L, torr-liter-sec^{-1}	Empty-chamber outgassing rate, Q_{ec}, torr-liter-sec^{-1}	Emission current, mA	Pressure range, torr	Measurement method	Leak rate, μtorr-liter-sec^{-1}, at time (min) after gage degas							
						5	10	15	20	30	40	50	60
10(a)	5.42×10^{-6}	6.74×10^{-7}	10	10^{-6}	Gas flowmeter	5.42	5.40	5.34	5.45	5.45	5.50	5.38	5.40
					Rate of rise	4.30	4.08	4.17	4.17	4.35	4.36	3.96	4.21
10(a)	5.42×10^{-6}	6.67×10^{-7}	1	10^{-6}	Gas flowmeter	5.46	5.36	5.49	5.47	5.42	5.33	5.45	5.36
					Rate of rise	5.88	5.96	6.13	6.17	5.76	5.76	5.67	5.49
10(b)	1.00×10^{-5}	8.36×10^{-7}	10	10^{-6}	Gas flowmeter	10.1	10.1	10.1	10.1	10.0	10.0	10.0	10.0
					Rate of rise	9.20	9.20	8.70	9.20	9.20	8.93	8.70	9.20
10(b)	1.01×10^{-5}	9.27×10^{-7}	1	10^{-6}	Gas flowmeter	10.1	10.0	10.1	10.0	10.0	10.2	10.1	10.0
					Rate of rise	9.59	9.92	10.5	10.5	10.1	10.1	10.2	9.92
10(c)	9.44×10^{-6}	9.51×10^{-7}	10	10^{-5}	Gas flowmeter	9.32	9.55	9.50	9.59	9.00	9.57	9.52	9.46
					Rate of rise	0.340	1.09	1.78	2.25	2.18	2.40	2.31	2.40
10(c)	1.04×10^{-5}	6.46×10^{-7}	1	10^{-5}	Gas flowmeter	10.3	10.3	10.3	10.4	10.5	10.7	10.5	10.3
					Rate of rise	9.43	10.1	10.1	10.3	10.1	9.76	10.1	9.76
10(d)	4.92×10^{-5}	7.97×10^{-7}	10	10^{-5}	Gas flowmeter	49.2	49.4	49.3	49.4	48.8	48.8	49.1	49.2
					Rate of rise	41.5	40.6	39.2	41.2	38.4	39.8	40.9	40.2
10(d)	4.92×10^{-5}	7.86×10^{-7}	1	10^{-5}	Gas flowmeter	49.2	49.4	49.2	49.0	49.2	49.0	49.1	49.2
					Rate of rise	52.5	49.9	50.8	51.6	49.5	51.6	49.4	49.8
10(e)	1.07×10^{-4}	5.10×10^{-7}	1	10^{-4}	Gas flowmeter	105	106	106	107	107	107	109	108
					Rate of rise	90.2	93.5	93.5	93.5	92.7	94.3	91.8	93.5
10(f)	4.70×10^{-4}	7.31×10^{-7}	1	10^{-4}	Gas flowmeter	475	472	472	470	470	466	-----	467
					Rate of rise	412	442	442	435	435	429	-----	432

TABLE 5.- TEST RESULTS FOR HELIUM

Figure	Average standard leak rate, Q_L, torr-liter-sec^{-1}	Empty-chamber outgassing rate, Q_{ec}, torr-liter-sec^{-1}	Emission current, mA	Pressure range, torr	Measurement method	Leak rate, μtorr-liter-sec^{-1}, at time (min) after gage degas							
						5	10	15	20	30	40	50	60
11(a)	5.34×10^{-6}	6.05×10^{-7}	10	10^{-6}	Gas flowmeter	5.36	5.36	5.33	5.34	5.29	5.35	5.42	5.38
					Rate of rise	3.11	3.79	3.31	3.61	2.74	2.73	2.81	2.56
11(b)	9.77×10^{-6}	7.19×10^{-7}	1	10^{-6}	Gas flowmeter	9.83	9.82	9.83	9.73	9.80	9.70	9.75	9.70
					Rate of rise	10.9	13.0	13.0	12.8	11.8	7.95	8.53	7.05
11(c)	1.06×10^{-5}	7.51×10^{-7}	10	10^{-5}	Gas flowmeter	10.6	10.5	10.6	10.8	10.4	10.6	10.6	10.7
					Rate of rise	8.92	8.42	7.22	7.92	7.22	8.62	8.22	8.62
11(c)	1.01×10^{-5}	7.22×10^{-7}	1	10^{-5}	Gas flowmeter	10.0	10.0	10.0	10.0	10.2	10.1	10.0	10.1
					Rate of rise	8.77	8.61	8.77	8.69	8.77	8.20	8.77	9.10
11(d)	5.25×10^{-5}	9.02×10^{-7}	10	10^{-5}	Gas flowmeter	53.5	52.5	52.0	52.2	52.5	52.3	53.0	51.8
					Rate of rise	46.5	44.1	46.5	44.3	45.5	43.6	44.3	45.7
11(d)	5.15×10^{-5}	9.27×10^{-7}	1	10^{-5}	Gas flowmeter	51.2	51.6	51.6	51.8	51.5	50.8	51.8	51.5
					Rate of rise	48.2	47.7	45.3	48.2	42.6	46.7	41.9	44.5
11(e)	1.05×10^{-4}	8.15×10^{-7}	1	10^{-4}	Gas flowmeter	103	106	103	104	105	104	105	105
					Rate of rise	115	110	109	114	114	116	114	116
11(f)	5.11×10^{-4}	7.63×10^{-7}	1	10^{-4}	Gas flowmeter	515	514	507	511	500	511	510	518
					Rate of rise	531	564	540	550	525	545	530	540

TABLE 6.- ANALYTICAL CORROBORATION OF GAGE PUMPING EFFECTS FOR 10-MILLIAMPERE METHANE MEASUREMENTS

$$[S = 0.09 \text{ liter-sec}^{-1}]$$

Figure	Standard leak rate, Q_L, torr-liter-sec⁻¹ (*)	True pressure, p, torr	Sp product, torr-liter-sec⁻¹	Sp × 100, percent Q	$V\frac{dp}{dt}$ (experimental), torr-liter-sec⁻¹ (**)	Leak rate (analytical), $V\frac{dp}{dt} + Sp$, torr-liter-sec⁻¹	Percentage deviation, $\dfrac{Q - \left(V\frac{dp}{dt} + Sp\right)}{Q} \times 100$
10(a)	5.42×10^{-6}	6.5×10^{-6}	5.85×10^{-7}	11	4.20×10^{-6}	4.79×10^{-6}	12
10(b)	1.00×10^{-5}	6.3×10^{-6}	5.67×10^{-7}	6	9.04×10^{-6}	9.61×10^{-6}	4
10(c)	9.44×10^{-6}	6.5×10^{-5}	5.85×10^{-6}	62	2.31×10^{-6}	8.16×10^{-6}	14
10(d)	4.92×10^{-5}	6.5×10^{-5}	5.85×10^{-6}	12	4.02×10^{-5}	4.61×10^{-5}	6

*Value from table 4.

**Average of eight experimental values from table 4.

TABLE 7.- ANALYTICAL CORROBORATION OF GAGE PUMPING EFFECTS FOR 10-MILLIAMPERE NITROGEN MEASUREMENTS

$$[S = 0.14 \text{ liter-sec}^{-1}]$$

Figure	Standard leak rate, Q_L, torr-liter-sec⁻¹ (*)	True pressure, p, torr	Sp product, torr-liter-sec⁻¹	Sp × 100, percent Q	$V\frac{dp}{dt}$ (experimental), torr-liter-sec⁻¹ (**)	Leak rate (analytical), $V\frac{dp}{dt} + Sp$, torr-liter-sec⁻¹	Percentage deviation, $\dfrac{Q - \left(V\frac{dp}{dt} + Sp\right)}{Q} \times 100$
9(a)	4.97×10^{-6}	1.10×10^{-5}	1.54×10^{-6}	31	3.17×10^{-6}	4.73×10^{-6}	5
9(b)	1.02×10^{-5}	1.10×10^{-5}	1.54×10^{-6}	15	1.00×10^{-5}	1.15×10^{-5}	13
9(c)	1.01×10^{-5}	6.54×10^{-5}	9.15×10^{-6}	91	1.11×10^{-6}	1.03×10^{-5}	2
9(d)	4.97×10^{-5}	1.10×10^{-4}	1.54×10^{-5}	31	4.34×10^{-5}	5.88×10^{-5}	18

*Value from table 3.

**Average of eight experimental values from table 3.
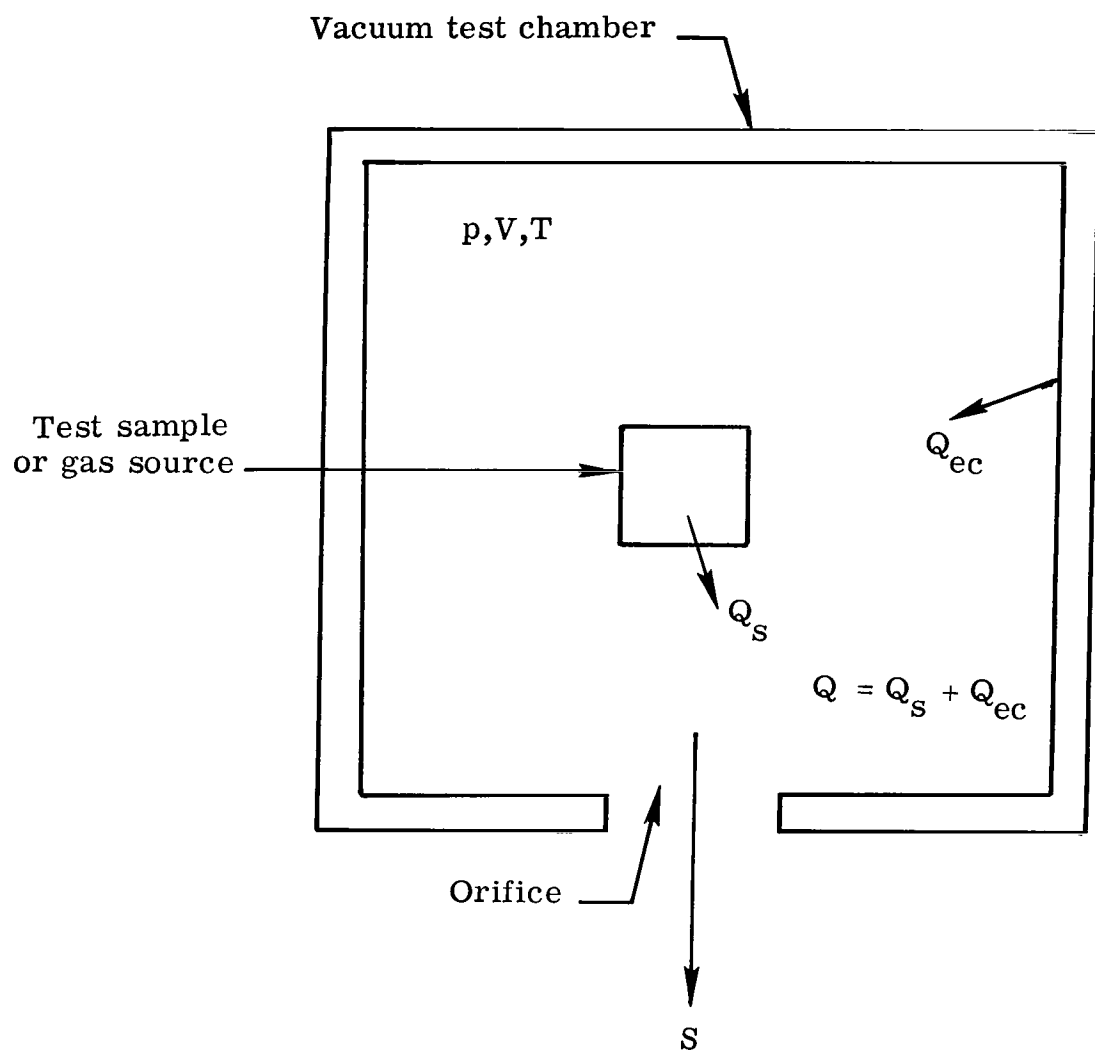
Figure 1.- System configuration for the outgassing equation.
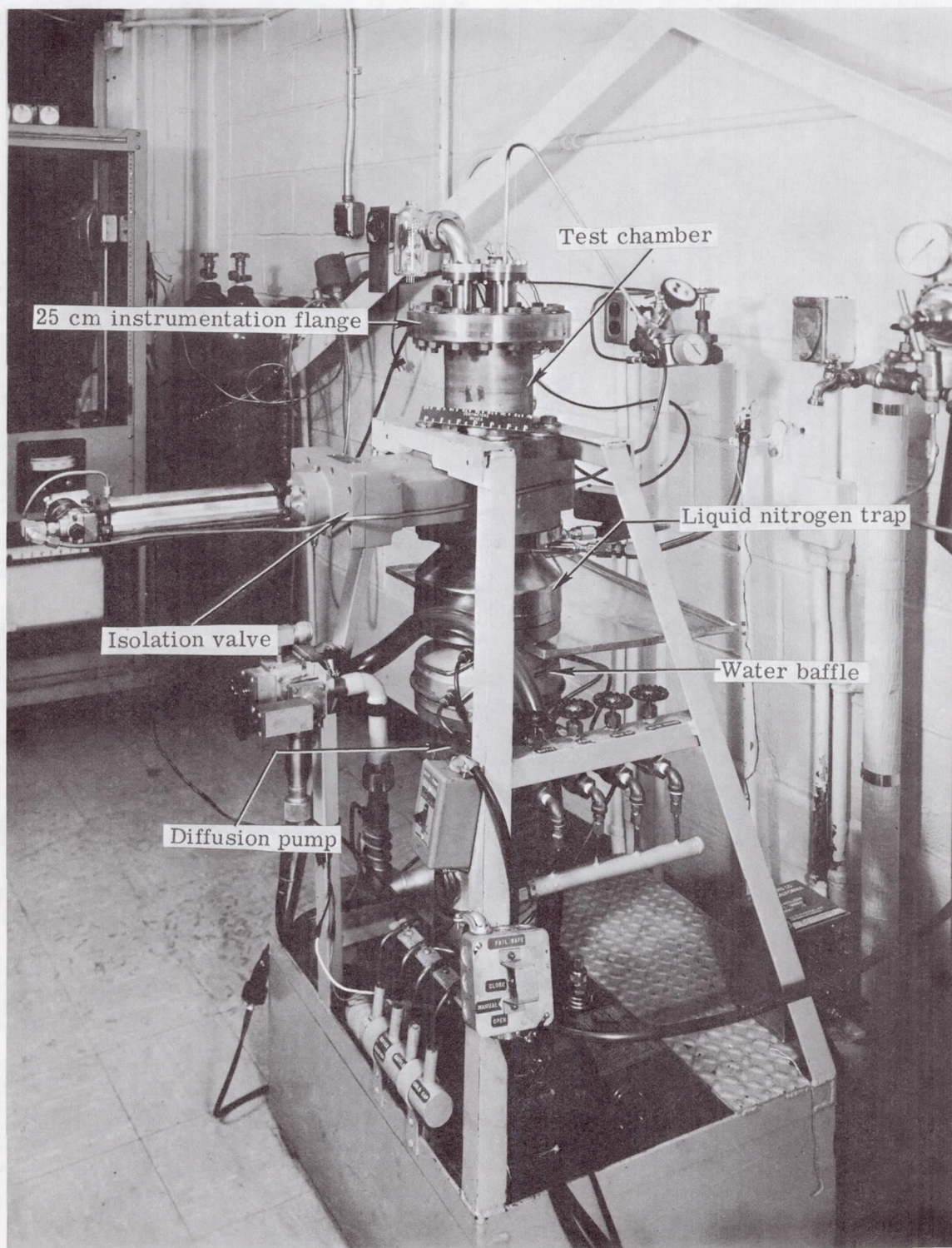
Figure 2.- Outgassing test apparatus.

L-66-6350.1
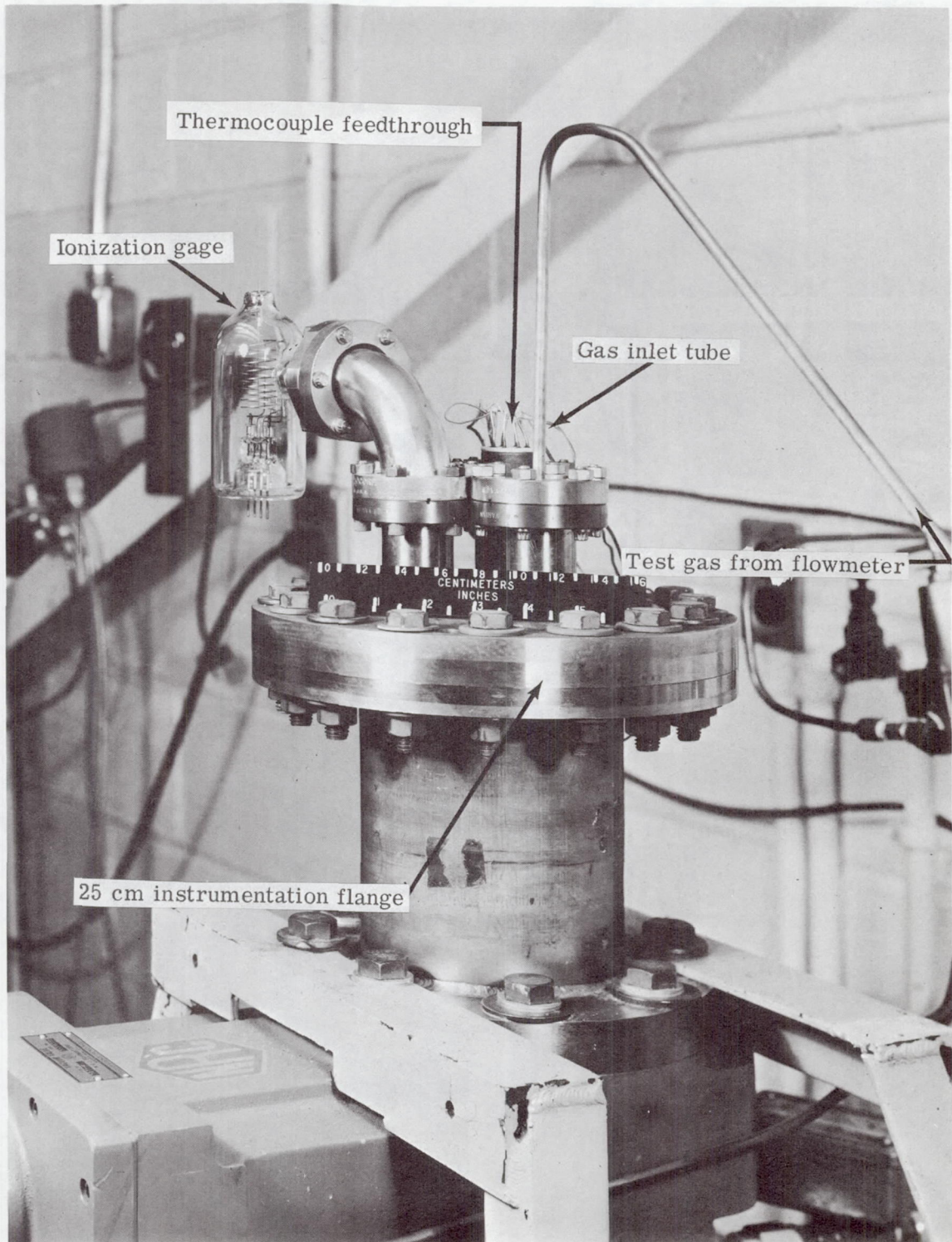
Figure 3.- Test chamber.

L-66-6349.1
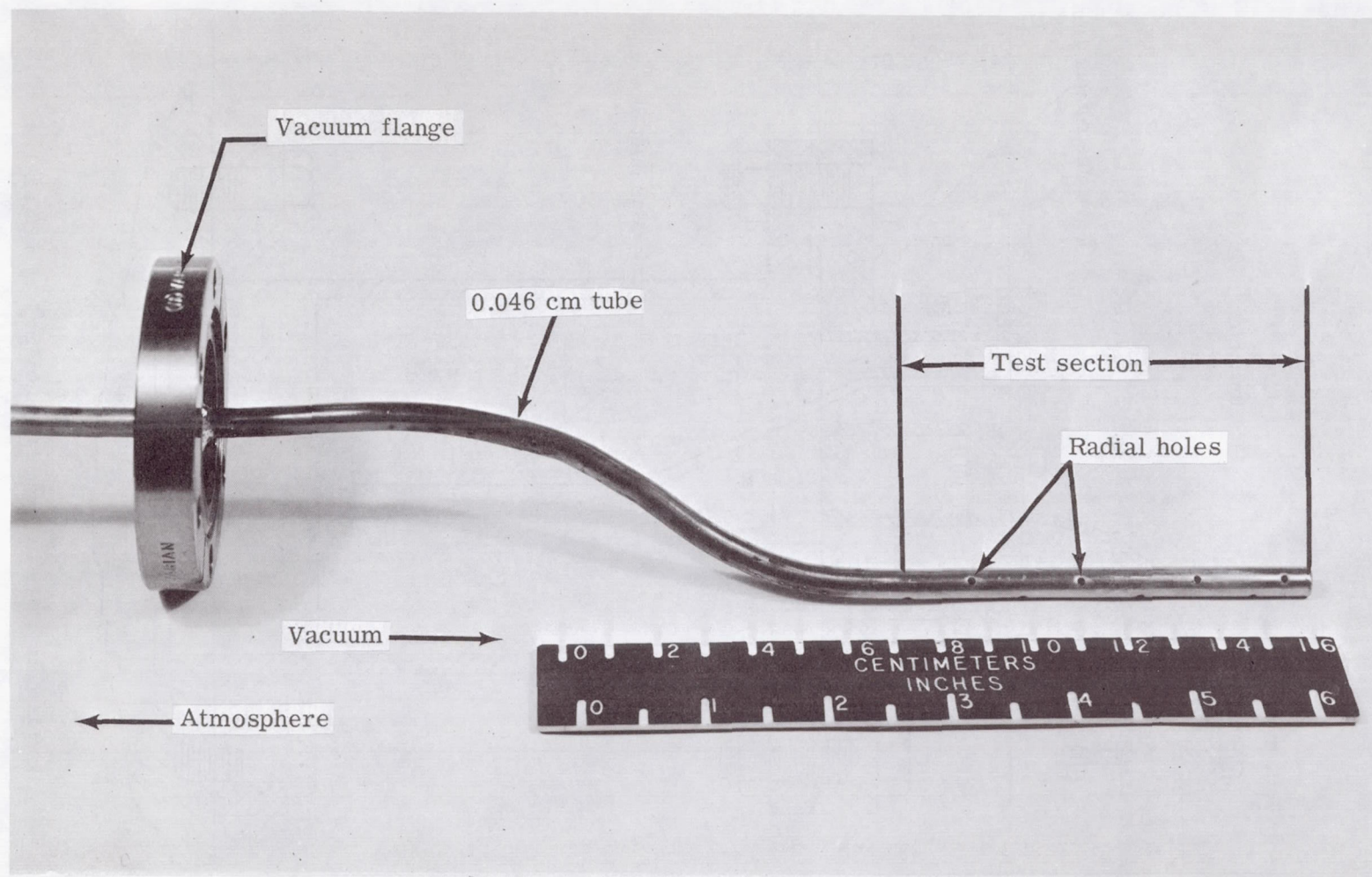
Figure 4.- Gas inlet tube.

L-66-6347.1

Ionization gage

5.08 cm

To flowmeter

Diam = 3.49 cm

5.72 cm

25 cm instrumentation flange

Diam = 1.76 cm

18.42 cm

10.8 cm

Diam = 3.15 cm

1.32 cm

1.32 cm

Gas inlet tube

10.16 cm

19.56 cm

Diam = 13.51 cm

Isolation valve
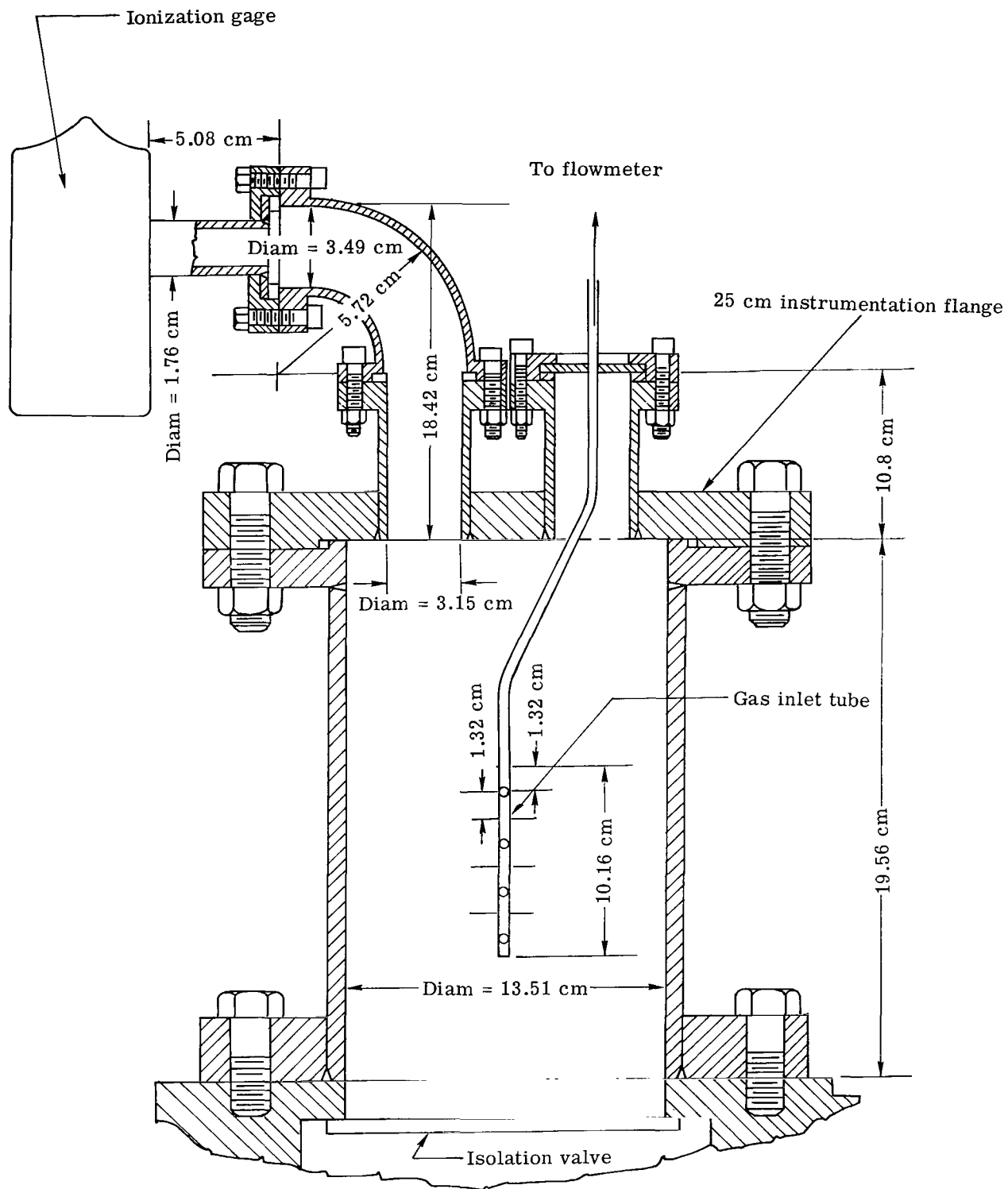
Figure 5.- Cross section of test chamber. (Dimensions are in cm; not to scale.)
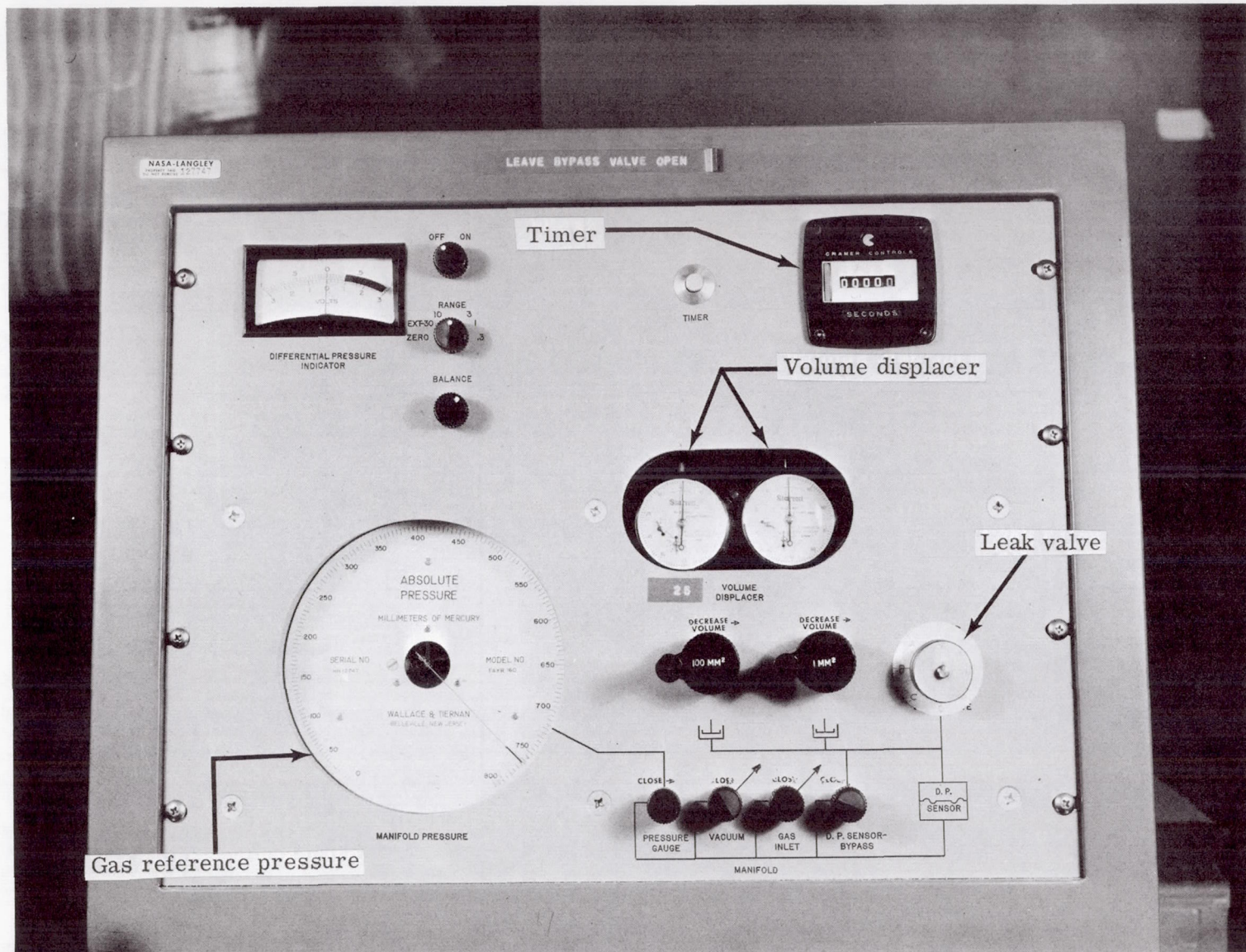
Figure 6.- Gas flowmeter operating console.

L-66-6346.1
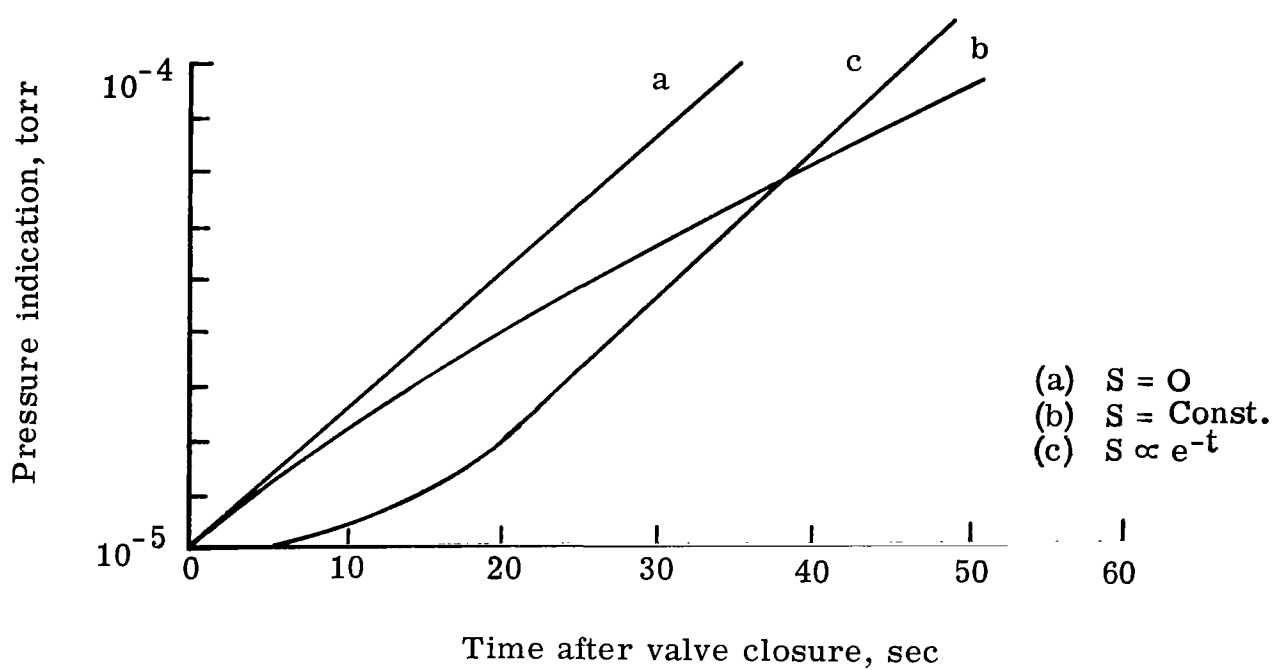
Figure 7.- Calculated rate-of-rise pressure traces. Leak rate, 1.00×10^{-5} torr-liter-sec^{-1}; test volume, 3.8 liters.
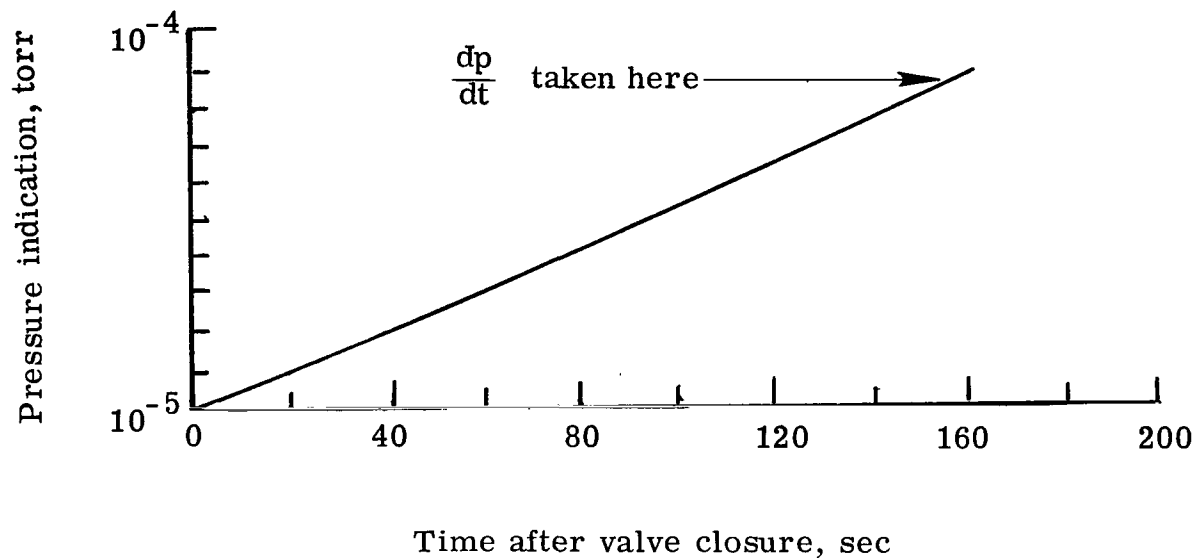
(a) Linear trace. Test gas, helium; standard leak rate, 1.06×10^{-5} torr-liter-sec^{-1}; emission current, 10 mA; pressure scale, 10^{-5} torr.
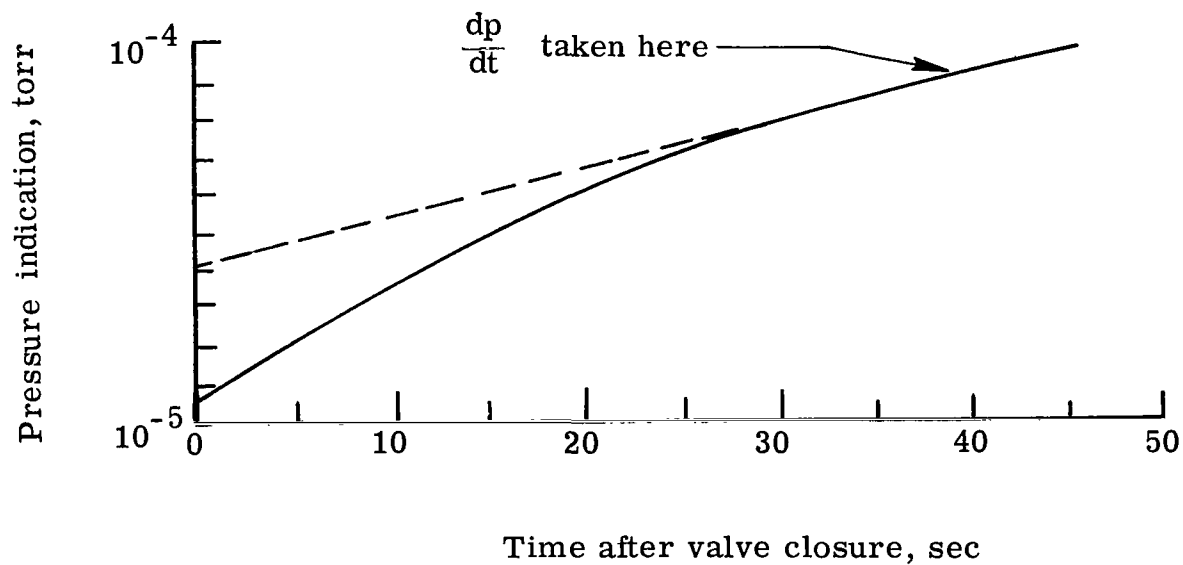
(b) Nonlinear trace. Test gas, methane; standard leak rate, 9.44×10^{-6} torr-liter-sec^{-1}; emission current, 10 mA; pressure scale, 10^{-5} torr.

Figure 8.- Observed rate-of-rise pressure traces.
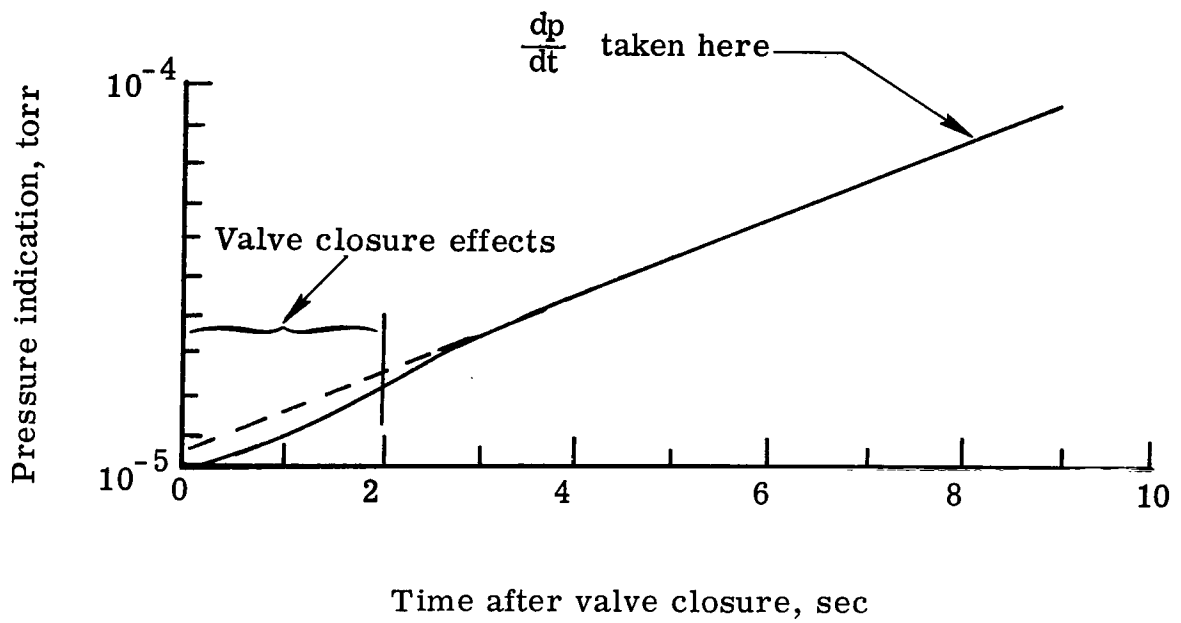
(c) Nonlinear trace. Test gas, nitrogen; standard leak rate, 4.97×10^{-5} torr-liter-sec^{-1}; emission current, 10 mA; pressure scale, 10^{-5} torr.

Figure 8.- Concluded.

34
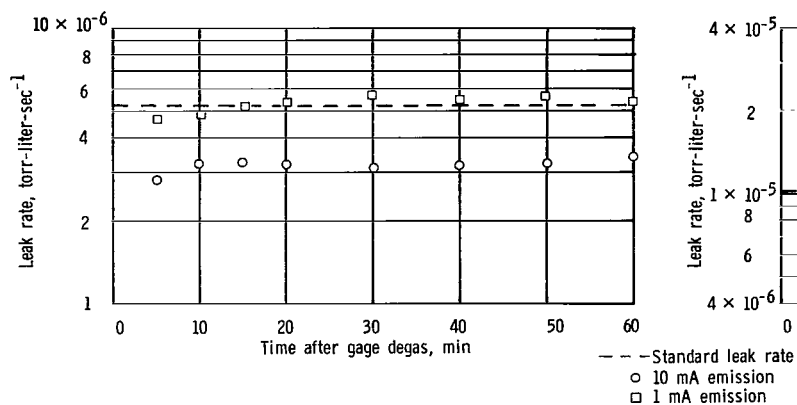
(a) Average leak rate, 5.23×10^{-6} (± 0.26) torr-liter-sec^{-1}; pressure scale, 10^{-6} torr.
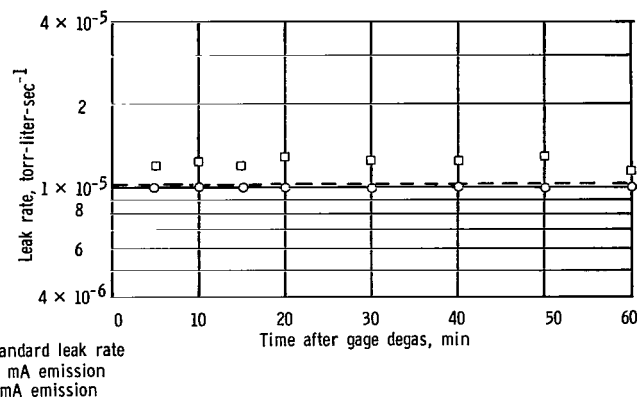
(b) Average leak rate, 1.03×10^{-5} (± 0.01) torr-liter-sec^{-1}; pressure scale, 10^{-6} torr.

— — —Standard leak rate
o 10 mA emission
□ 1 mA emission
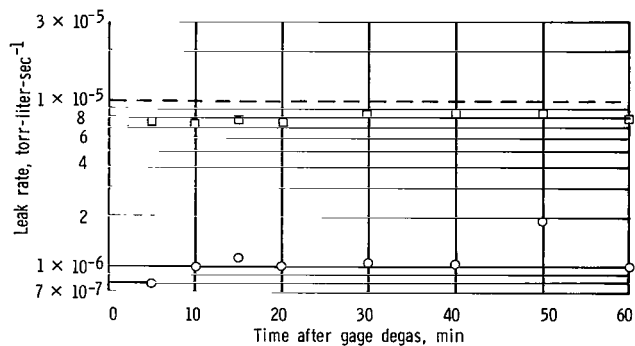
(c) Average leak rate, 1.01×10^{-5} (± 0.0) torr-liter-sec^{-1}; pressure scale, 10^{-5} torr.
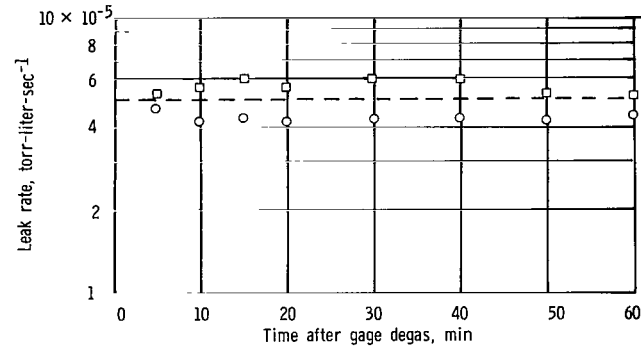
(d) Average leak rate, 5.05×10^{-5} $\left(\begin{smallmatrix}+0.09\\-0.08\end{smallmatrix}\right)$ torr-liter-sec^{-1}; pressure scale, 10^{-5} torr.
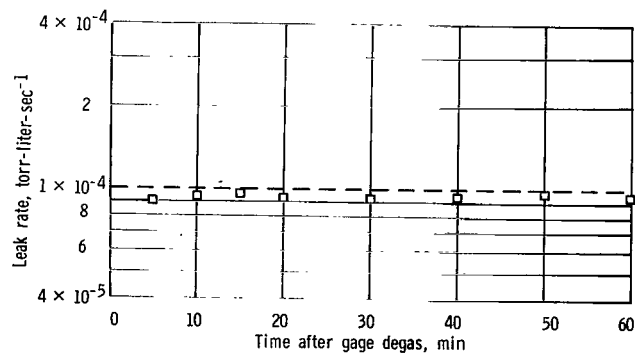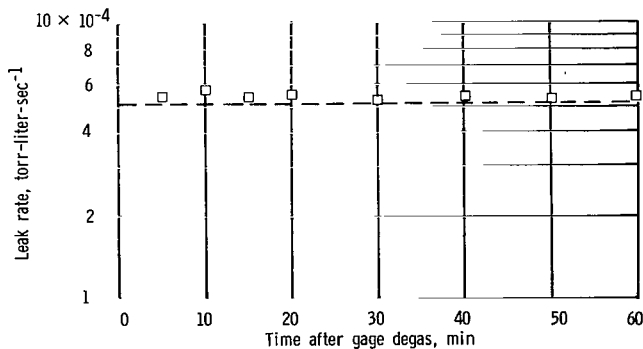
(e) Average leak rate, 1.00×10^{-4} torr-liter-sec^{-1}; pressure scale, 10^{-4} torr.
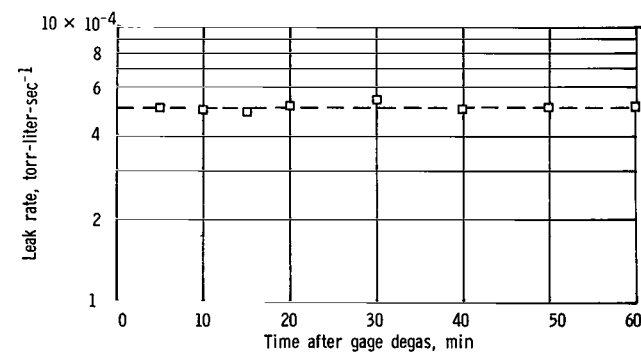
(f) Average leak rate, 5.10×10^{-4} torr-liter-sec^{-1}; pressure scale, 10^{-4} torr.

Figure 9.- Nitrogen test results.

35
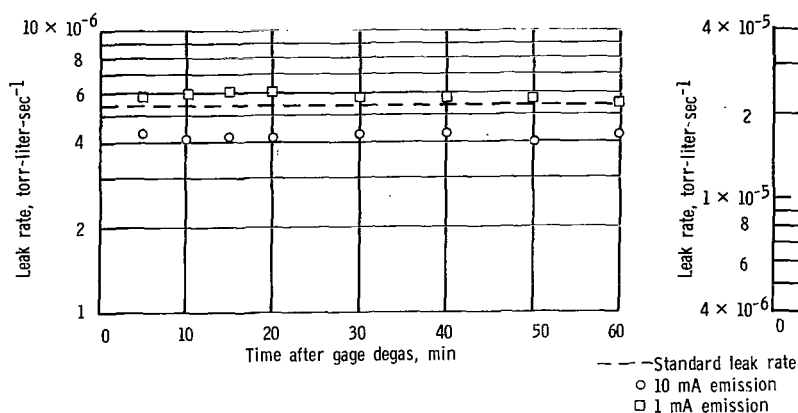
(a) Average leak rate, 5.42×10^{-6} (± 0.0) torr-liter-sec^{-1}; pressure scale, 10^{-6} torr.
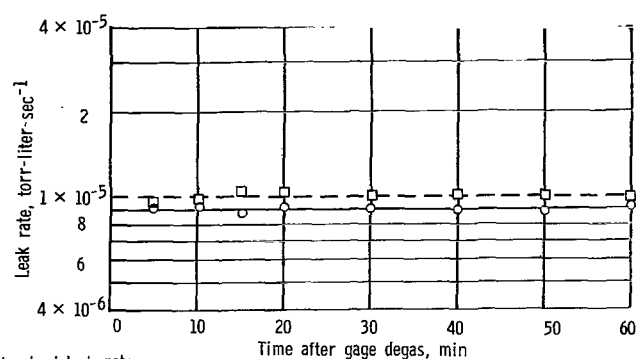
(b) Average leak rate, 1.00×10^{-5} $\binom{+0.01}{-0.00}$ torr-liter-sec^{-1}; pressure scale, 10^{-6} torr.

— —Standard leak rate
○ 10 mA emission
□ 1 mA emission
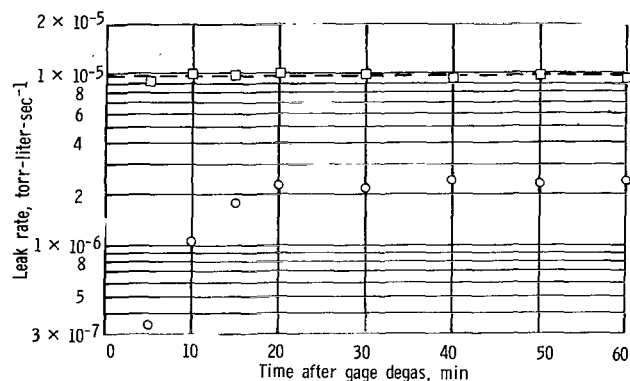
(c) Average leak rate, 9.74×10^{-6} (± 0.28) torr-liter-sec^{-1}; pressure scale, 10^{-5} torr.
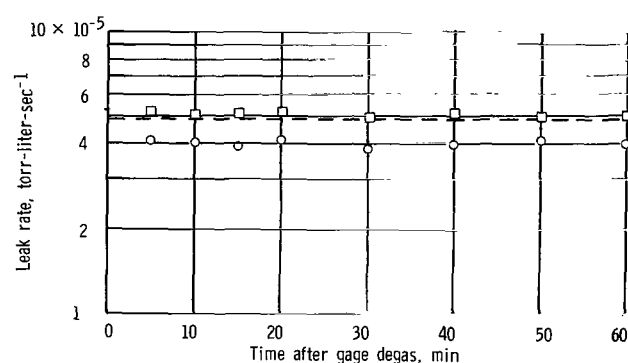
(d) Average leak rate, 4.92×10^{-5} (± 0.0) torr-liter-sec^{-1}; pressure scale, 10^{-5} torr.
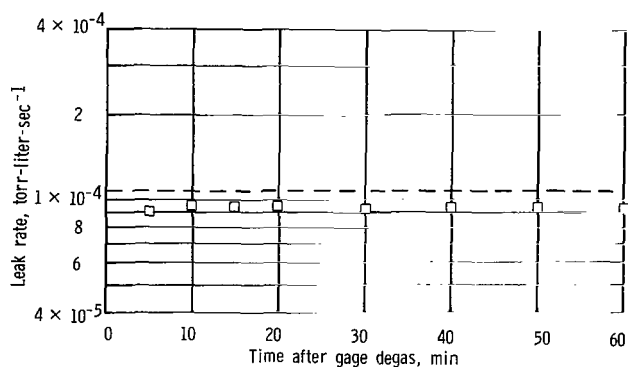
(e) Average leak rate, 1.07×10^{-4} torr-liter-sec^{-1}; pressure scale, 10^{-4} torr.
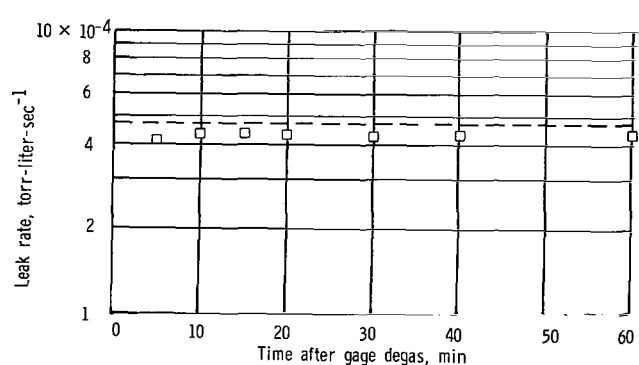
(f) Average leak rate, 4.70×10^{-4} torr-liter-sec^{-1}; pressure scale, 10^{-4} torr.

Figure 10.- Methane test results.

36
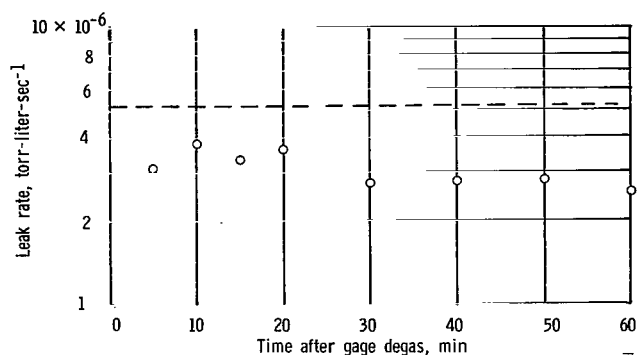
(a) Average leak rate, 5.34 × 10⁻⁶ torr-liter-sec⁻¹; pressure scale, 10⁻⁶ torr.
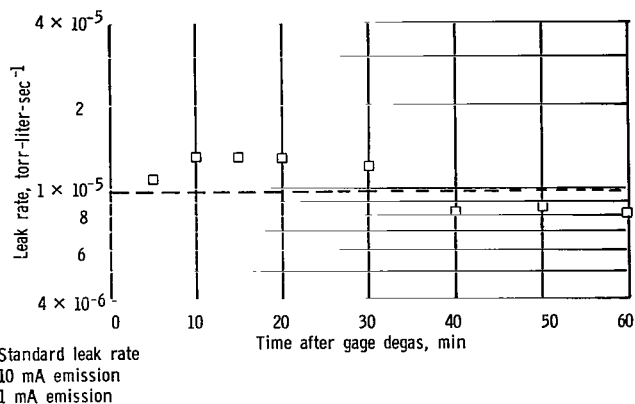
(b) Average leak rate, 9.77 × 10⁻⁶ torr-liter-sec⁻¹; pressure scale, 10⁻⁶ torr.
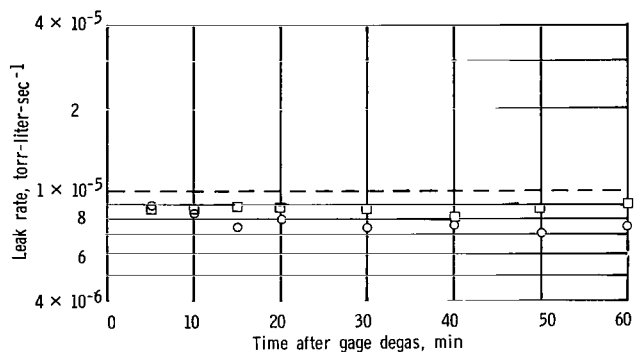
(c) Average leak rate, 1.03 × 10⁻⁵ $\left(\begin{smallmatrix}+0.03\\-0.02\end{smallmatrix}\right)$ torr-liter-sec⁻¹; pressure scale, 10⁻⁵ torr.
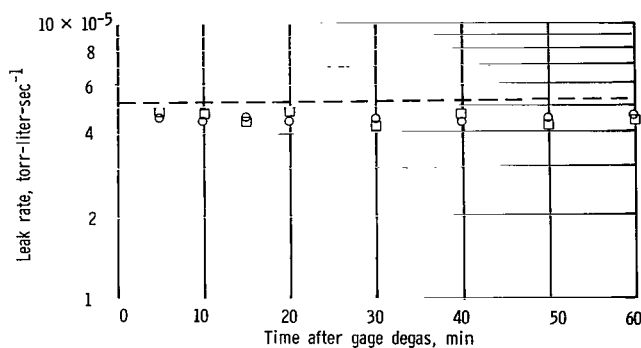
(d) Average leak rate, 5.20 × 10⁻⁵ (±0.05) torr-liter-sec⁻¹; pressure scale, 10⁻⁵ torr.
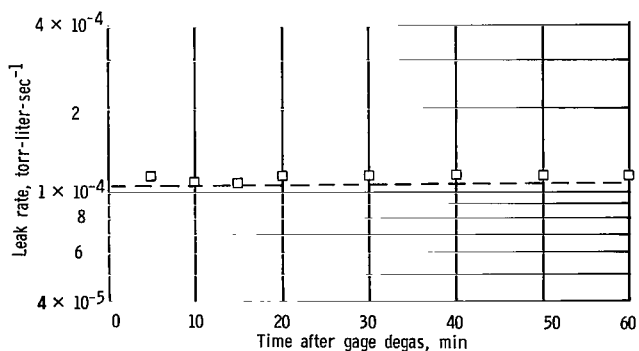
(e) Average leak rate, 1.05 × 10⁻⁴ torr-liter-sec⁻¹; pressure scale, 10⁻⁴ torr.

(f) Average leak rate, 5.11 × 10⁻⁴ torr-liter-sec⁻¹; pressure scale, 10⁻⁴ torr.

Figure 11.- Helium test results.

37
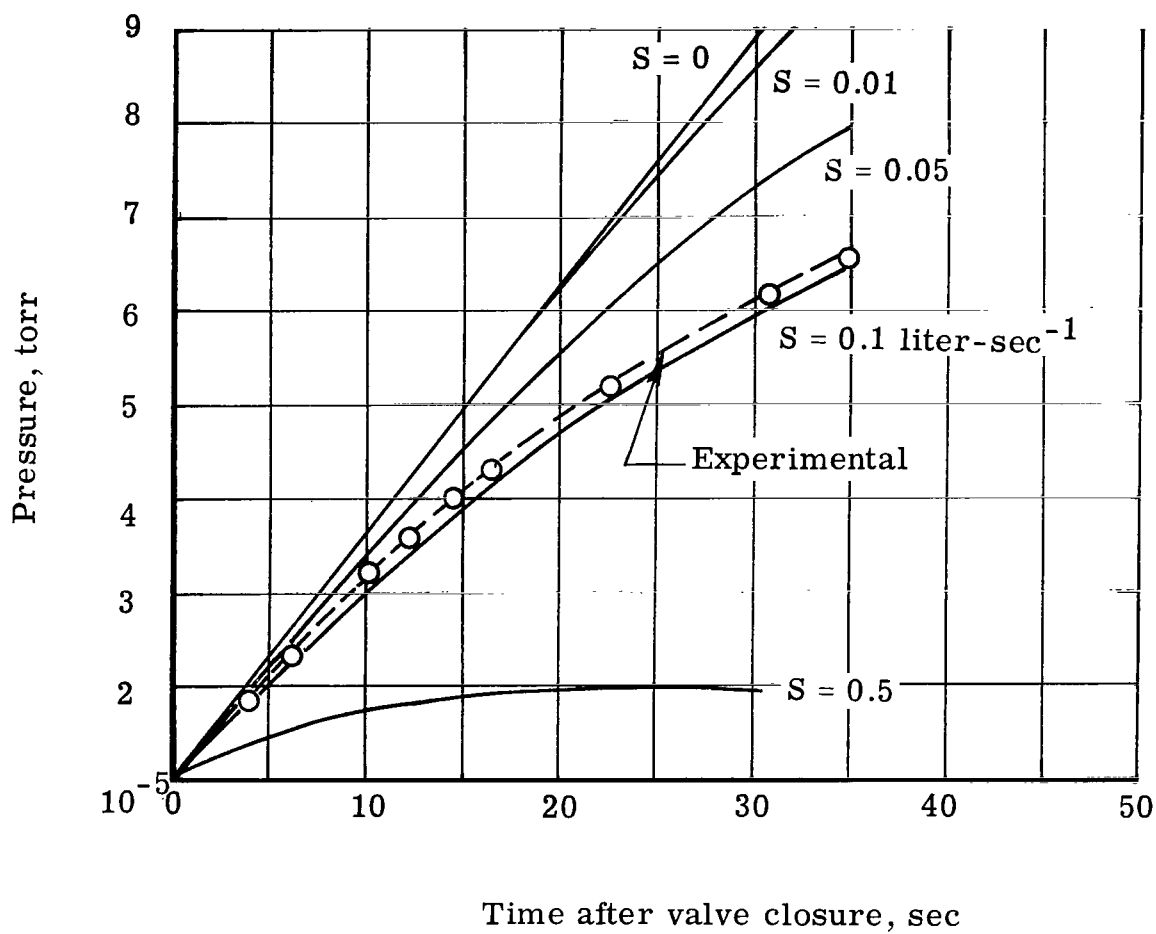
Figure 12.- Estimation of gage pumping speed for methane.
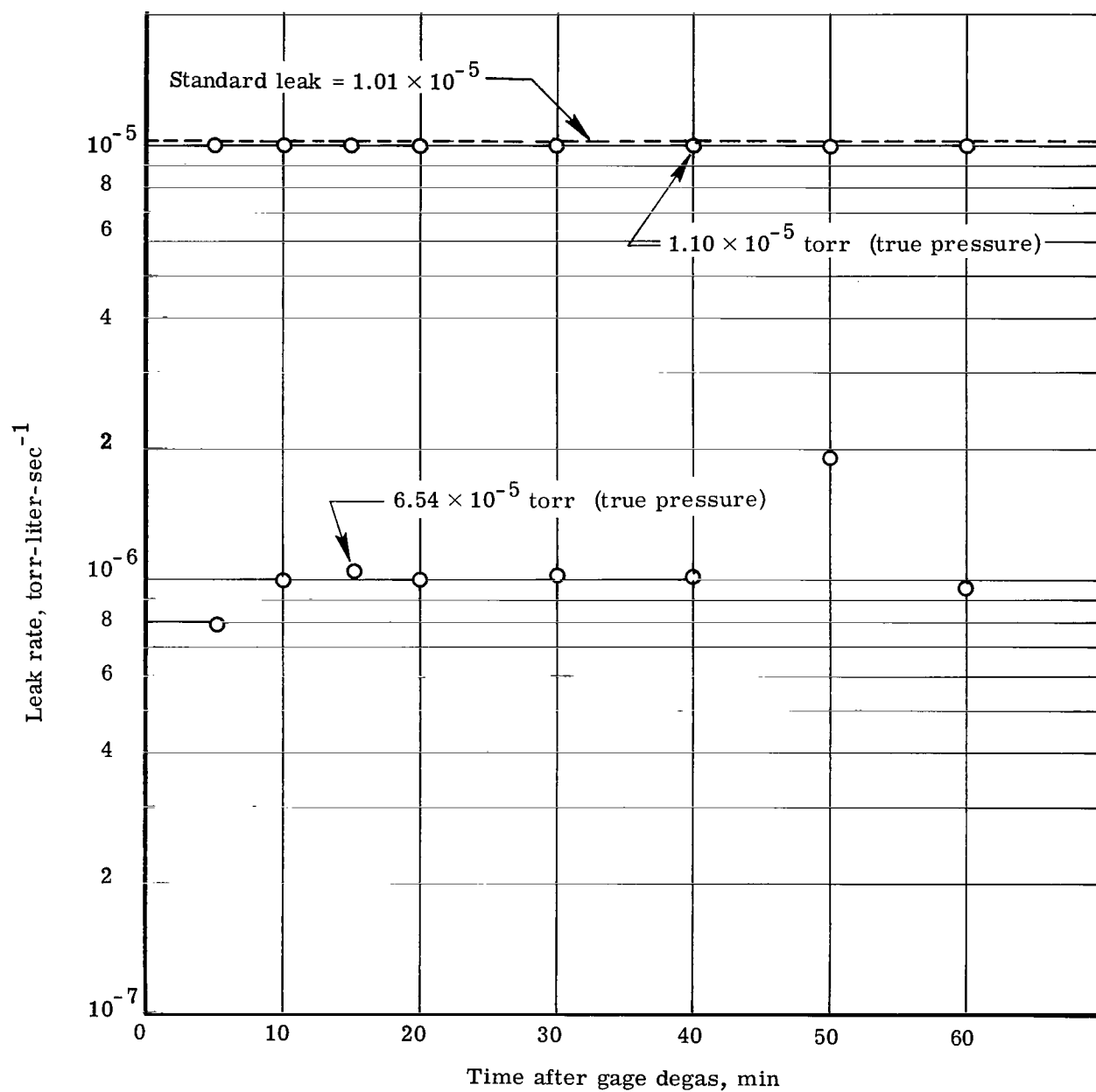
Figure 13.- Effects of pressure on rate-of-rise measurements for nitrogen. Average standard leak, 1.01×10^{-5} torr-liter-sec^{-1}; emission current, 10 mA.
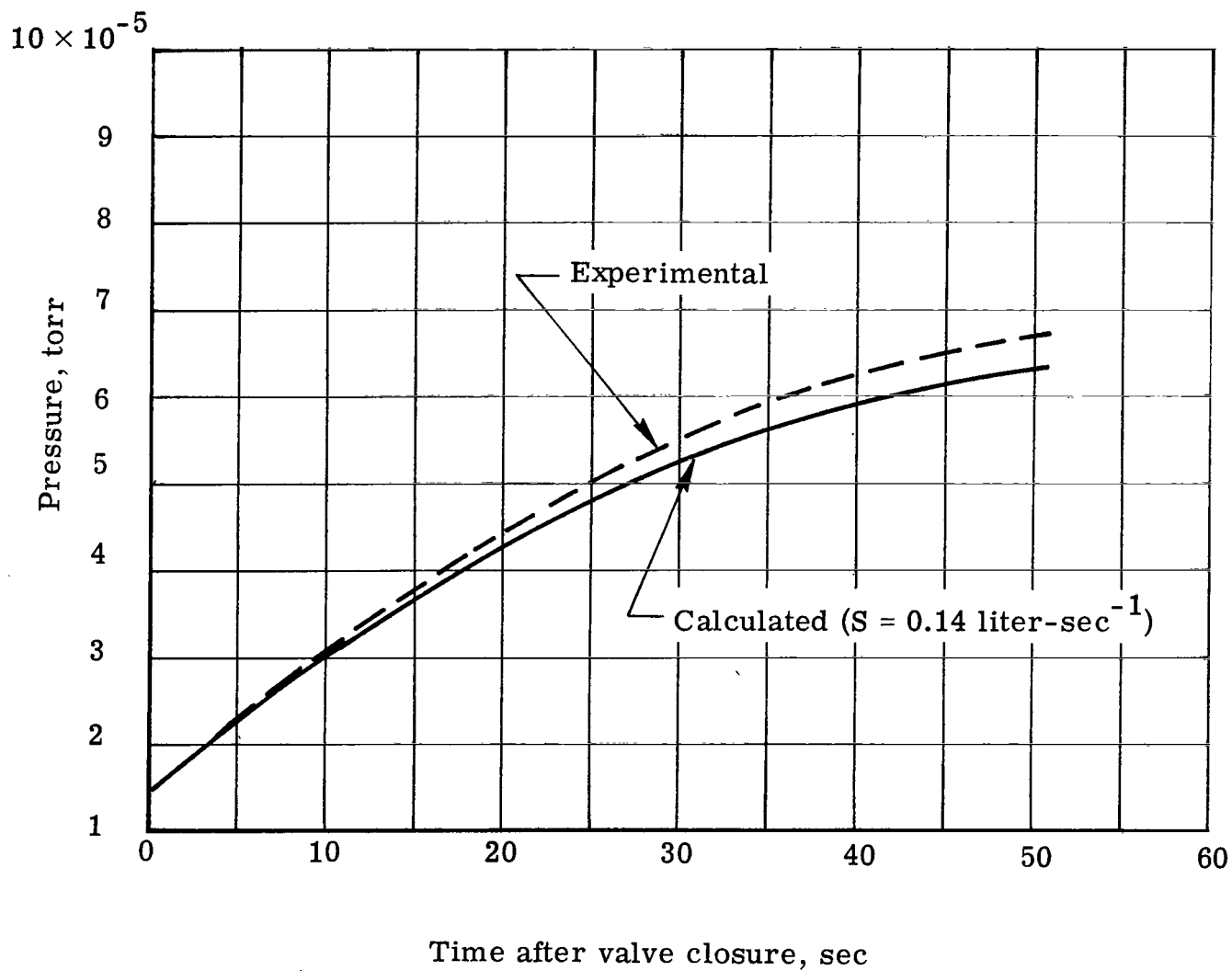
Figure 14.- Typical pressure trace obtained for 10-milliampere measurements of figure 9(c). Standard leak rate, 1.01×10^{-5} torr-liter-sec^{-1}; volume, 3.8 liters.
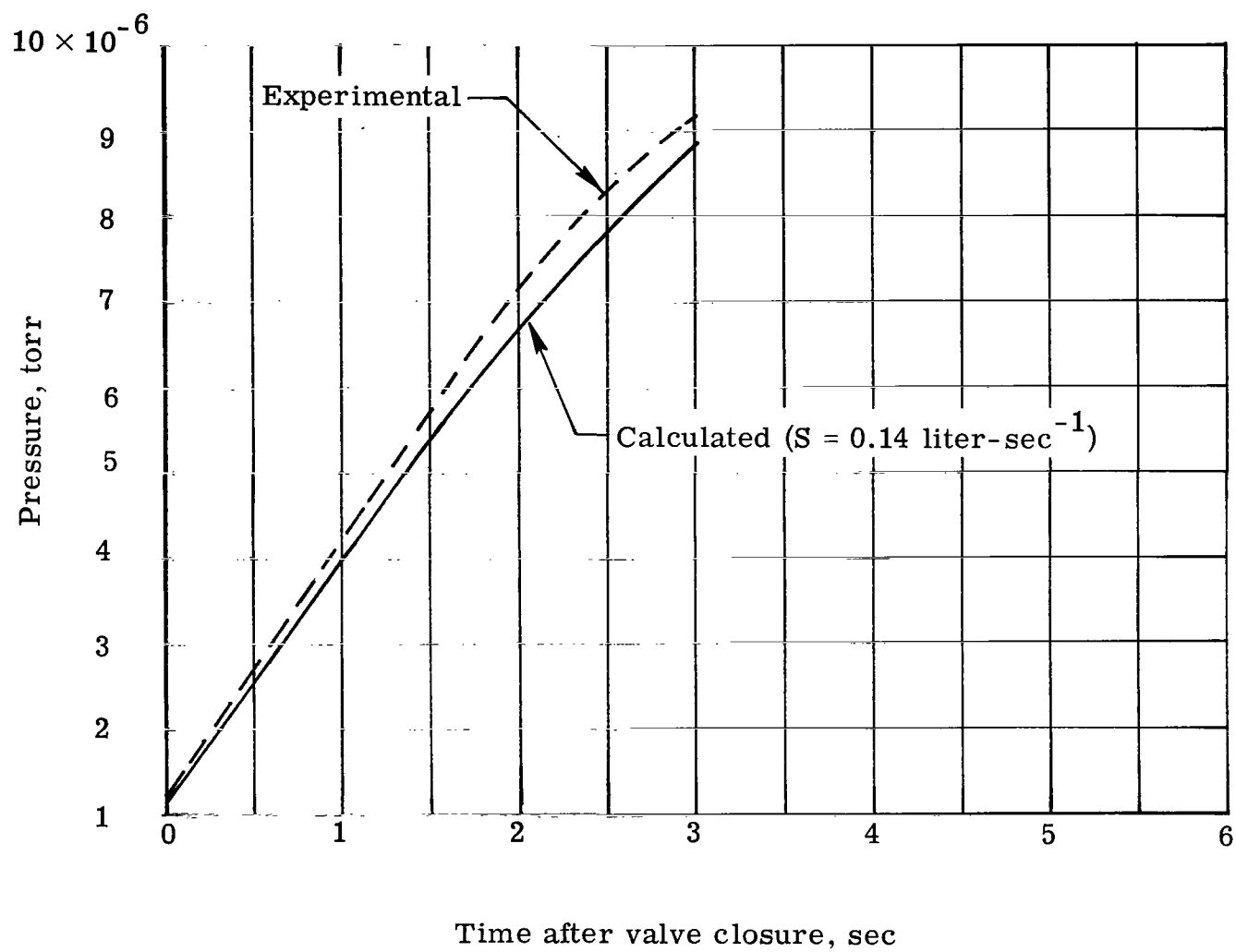
Figure 15.- Typical pressure trace obtained for 10-milliampere measurements of figure 9(b). Standard leak rate, 1.03×10^{-5} torr-liter-sec^{-1}; volume, 3.8 liters.
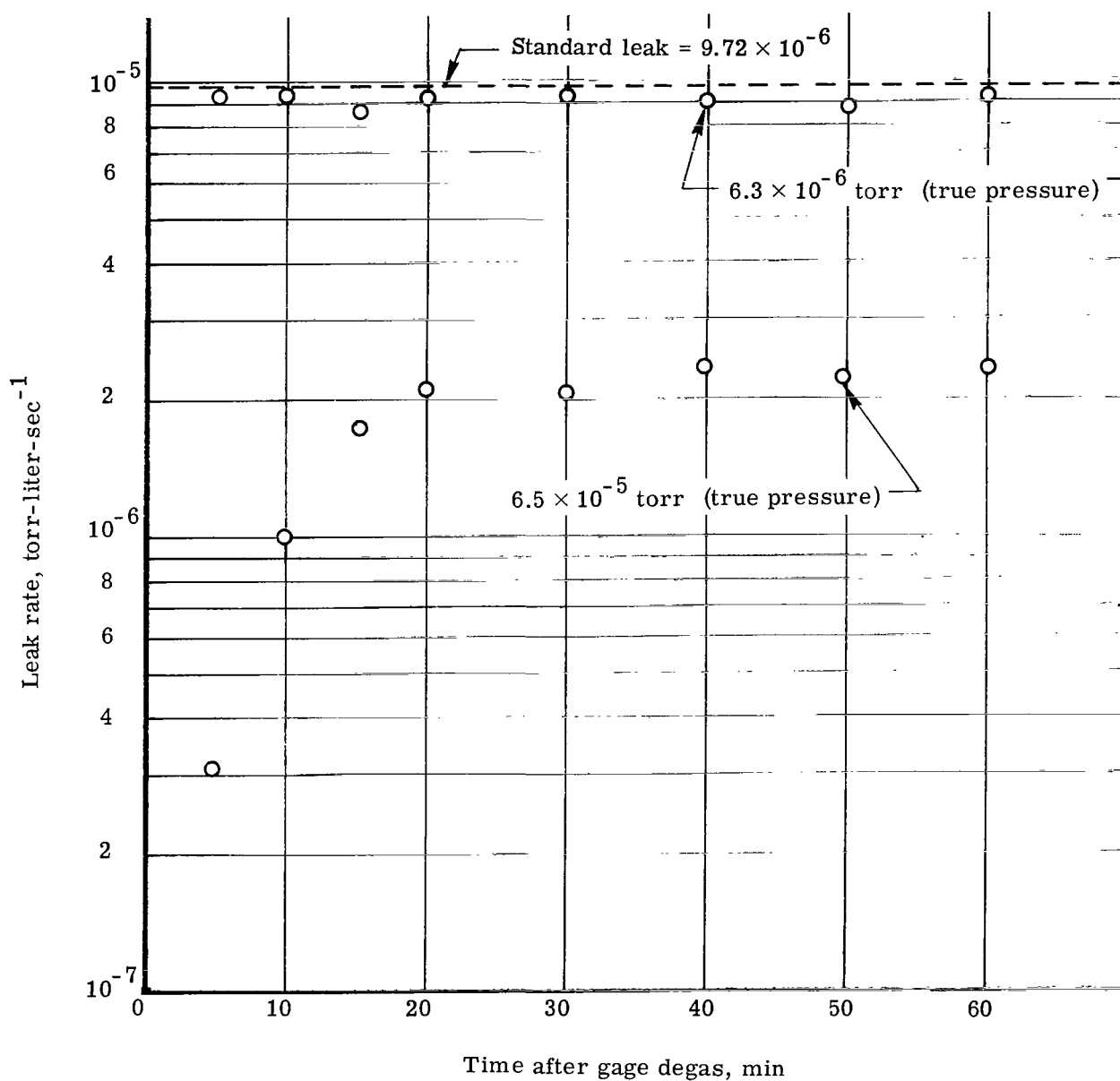
Figure 16.- Effect of pressure on rate-of-rise measurements for methane. Average standard leak rate, 9.72×10^{-6} torr-liter-sec^{-1}; emission current, 10 mA.

"The aeronautical and space activities of the United States shall be
conducted so as to contribute . . . to the expansion of human knowl-
edge of phenomena in the atmosphere and space. The Administration
shall provide for the widest practicable and appropriate dissemination
of information concerning its activities and the results thereof."

— NATIONAL AERONAUTICS AND SPACE ACT OF 1958

NASA SCIENTIFIC AND TECHNICAL PUBLICATIONS

TECHNICAL REPORTS: Scientific and
technical information considered important,
complete, and a lasting contribution to existing
knowledge.

TECHNICAL NOTES: Information less broad
in scope but nevertheless of importance as a
contribution to existing knowledge.

TECHNICAL MEMORANDUMS:
Information receiving limited distribution
because of preliminary data, security classifica-
tion, or other reasons.

CONTRACTOR REPORTS: Scientific and
technical information generated under a NASA
contract or grant and considered an important
contribution to existing knowledge.

TECHNICAL TRANSLATIONS: Information
published in a foreign language considered
to merit NASA distribution in English.

SPECIAL PUBLICATIONS: Information
derived from or of value to NASA activities.
Publications include conference proceedings,
monographs, data compilations, handbooks,
sourcebooks, and special bibliographies.

TECHNOLOGY UTILIZATION
PUBLICATIONS: Information on technology
used by NASA that may be of particular
interest in commercial and other non-aerospace
applications. Publications include Tech Briefs,
Technology Utilization Reports and Notes,
and Technology Surveys.

Details on the availability of these publications may be obtained from:

SCIENTIFIC AND TECHNICAL INFORMATION DIVISION

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Washington, D.C. 20546