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IN-CHAMBER VACUUM GAGE CALIBRATION METHOD AND ITS APPLICATION TO CRYOPUMPING MEASUREMENTS

R. M. Freeman
ARO, Inc.

January 1967

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FOREWORD

The research presented in this report was sponsored by the Arnold Engineering Development Center (AEDC), Air Force Systems Command (AFSC), Arnold Air Force Station, Tennessee, under Program Element 65402234.

Results presented herein were obtained by ARO, Inc. (a subsidiary of Sverdrup & Parcel and Associates, Inc.), contract operator of the AEDC under Contract AF40(600)-1200. The research was conducted from January 1 to August 30, 1965, under ARO Project No. SW3417, and the manuscript was submitted for publication on October 26, 1966.

This technical report has been reviewed and is approved.

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Research Division	Director of Plans and Technology
Directorate of Plans and Technology	

ABSTRACT

This report describes a method of calibrating mass spectrometers and ion gages for pumping speed measurements. Sensitivity factors and pumping speed constants are determined from an analysis of the chamber pressures during gage calibration and pumping speed measurements. The CO_2 sensitivity factor for the ion gage or mass spectrometer is obtained. The pumping speed constants were determined from the gage readings versus gas throughput. The method was applied to both ion gage and mass spectrometer measurements in determining the pumping speed of a 77°K surface for 300°K CO_2, and approximately the same value of pumping speed was obtained for both gages.

CONTENTS

TABLE

APPENDIX

Illustrations

NOMENCLATURE

C	Sticking fraction
F	Clausing factor
I	Gage indication
K	Conductance, ℓ/sec
K_c	Effective conductance, ℓ/sec
P	Pressure, torr
Q	Throughput, torr-ℓ/sec
S	Pumping speed, ℓ/sec
T	Temperature, °K
t	Time, sec
v	Volume, ℓ
β	Pumping speed proportionality constant
γ	Ion gage or mass spectrometer sensitivity factor

SUBSCRIPTS

0	Background conditions, no gas flow
1	Region number 1
2	Region number 2
f	Forepressure on leak
L	Porous plug leak
t	Theoretical

SECTION I
INTRODUCTION

Investigation of cryopumping phenomena has been in progress at the Arnold Engineering Development Center for several years. As a result, the capture coefficient of a large number of gases has been measured.

Instrument calibration is one of the fundamental problems in the investigation of cryopumping phenomena. It is desirable to have an in-chamber instrument calibration. Such a procedure can eliminate systematic errors which may be introduced into the measurements because of differences in conductance, thermal transpiration, and view factors between the calibration apparatus and the test chamber.

This report describes an in-place calibration technique for pressure measuring instruments such as ion gages and mass spectrometers. An analysis is given for a vacuum chamber of a particular geometry — one in which the testing chamber is separated from the conventional pumping system by an orifice. A technique is given to determine the sensitivity factors of the instruments. Also, calibration and pumping speed measurement procedures are analyzed, and a method is suggested such that a constant outgassing rate, vapor pressure, and other chamber background conditions are taken into account.

To demonstrate the method, the instruments were calibrated for CO_2, and the experimental pumping speed of a 77°K cryosurface for 300°K CO_2 was determined.

SECTION II
ANALYSIS

2.1 ION GAGE AND MASS SPECTROMETER CALIBRATION

Consider a vacuum chamber (Fig. 1) of the geometry schematically shown in Fig. 2 which has an orifice, of conductance K_t, connecting the interior test chamber (region 1) with region 2 which is connected to a conventional pumping system. Region 1 contains a spherical cryosurface, a gas addition inlet, and tubulation to an ion gage and mass spectrometer. If only the pumping system in region 2 is being used and gas is introduced into the chamber through the gas inlet, the equilibrium gas flow rate may be expressed

$$\frac{P_t K_L}{T_{in}} = \frac{Q}{T_{in}} = \frac{P_1 K_t}{T_1} - \frac{P_2 K_t}{T_2} \tag{1}$$

where P_1 and P_2 are the absolute steady-state partial pressures in regions 1 and 2 of the gas in the chamber, and K_t is the conductance of the orifice. P_f is the forepressure on the porous plug leak of conductance, K_L (Fig. 3). T_{in}, T_1, and T_2 are the temperatures in the corresponding regions. The term $\frac{P_1K_t}{T_1}$ represents the outflow through the orifice from region 1 and $\frac{P_2K_t}{T_2}$ the backflow from region 2. Equation (1) may be expressed in terms of an effective orifice conductance as

$$\frac{P_f K_L}{T_{in}} = \frac{P_1 K_c}{T_1} = \frac{Q}{T_{in}} \tag{2}$$

where the effective conductance is given by

$$K_c = \left(1 - \frac{P_2}{P_1} \cdot \frac{T_1}{T_2}\right) K_t \tag{3}$$

The theoretical orifice conductance is (Ref. 1)

$$K_t = A_o F \sqrt{\frac{RT_1}{2\pi M}} \tag{4}$$

where

F = clausing factor for short tubes

A_o = area of orifice

Now the pressure terms in Eq. (3) are the absolute partial pressures of the gas in regions 1 and 2 for which the gage is being calibrated. Since the pressures are obtained as gage readings, it is convenient to write Eq. (3) in terms of the gage indication,

$$K_c = \left[1 - \frac{I_2 - I_{20}}{I_1 - I_{10}} \cdot \frac{\gamma_1}{\gamma_2} \cdot \frac{T_1}{T_2}\right] K_t$$

or

$$K_c = \left[1 - \left(\frac{I_2}{I_1 - I_{10}} - \frac{I_{20}}{I_1 - I_{10}}\right) \frac{\gamma_1}{\gamma_2} \, \frac{T_1}{T_2}\right] K_t \tag{5}$$

γ_1, γ_2 are the sensitivity factors ($\gamma = I/P$) for ion gages 1 and 2, respectively; I_{10} and I_{20} are the background ion gage readings for regions 1 and 2. I_{10} and I_{20} represent pressures from constant outgassing rates. The assumption is made that all outgassing rates are constant or negligible.

In any calibration test, some of the terms in Eq. (5) are negligibly small in comparison to others, and simplifications can be made. For example, I_{20} is less than 1/100 of I_1, so the term $\frac{I_{20}}{I_1 - I_{10}}$ can be neglected. Further, the sensitivity factors for identical brands of ion gages are almost the same, so the approximation $\gamma_2/\gamma_1 \cong 1$ can be made. (If there is a 15-percent error in the assumed ratio above, the error induced in K_c is less than 1 percent.) Equation (5) to a good approximation then becomes

$$K_c = \left[1 - \frac{I_2}{I_1 - I_{10}} \cdot \frac{T_1}{T_2}\right] K_t \tag{6}$$

Now, in terms of ion gage or mass spectrometer readings, $P_1 = \frac{I_1}{\gamma} - P_{10}$. Equation (2) is then written,

$$I_1 = \frac{\gamma Q}{K_c} \cdot \frac{T_1}{T_{in}} + I_{10} \tag{7}$$

where I_{10} is the gage indication for $Q = 0$.

The ion gage or mass spectrometer sensitivity factor can be obtained by fitting a curve to data plotted in the form I_1 versus Q, i. e.,

$$\gamma = K_c \left(\frac{dI_1}{dQ}\right)_{calibrate} \frac{T_{in}}{T_1} \tag{8}$$

2.2 PUMPING SPEED MEASUREMENTS

When dynamic equilibrium is reached during a pumping speed run, the gas flow into the chamber is equal to the gas flow being removed by the cryopumping sphere plus the effective gas flow out of the orifice. Thus,

$$\frac{Q_{in}}{T_{in}} = \frac{Q_{cryopump}}{T_1} + \frac{Q_{orifice}}{T_1} \tag{9}$$

The backflow through the orifice is included in an effective conductance (Eq. (3)), and Eq. (9) reduces to

$$\frac{Q_{in}}{T_{in}} = \frac{P_1\left(\frac{ds}{dt}\right)_{cryopump}}{T_1} + \frac{P_1 K_c}{T_1} \tag{10}$$

With free molecule flow conditions in the chamber, the volumetric pumping rate on a surface area A_S is given by:

$$\left(\frac{dv}{dt}\right)_{cryopump} = C A_s \sqrt{\frac{RT_1}{2\pi M}} = C S_t \tag{11}$$

where C is defined as the ratio of the actual pumping speed to the theoretical strike rate, S_t.

A substitution of Eq. (11) in Eq. (10) yields,

$$P_1 = \frac{T_1}{T_{in}} \frac{Q_{in}}{\left[C S_t + K_c\right]} \tag{12}$$

However, the pressure, P_1, in Eqs. (10) and (12) is an absolute partial pressure of the test gas. So, when Eq. (12) is differentiated and written in terms of the pressure gage reading, the ion gage or mass spectrometer sensitivity factor must be used to obtain the true pressure.

Thus,

$$\frac{1}{\gamma}\left(\frac{dI_1}{dQ}\right)_{cryopump} = \frac{T_1}{T_{in}} \cdot \frac{1}{\left[C S_t + K_c\right]} \tag{13}$$

Combining Eq. (8) with Eq. (13) we get the sticking fraction

$$C = \frac{K_c}{S_t}\left[\frac{\left(\frac{dI_1}{dQ}\right)_{calibrate}}{\left(\frac{dI_1}{dQ}\right)_{cryopump}} - 1\right] \tag{14a}$$

where the orifice conductance is

$$K_c = \left(1 - \frac{I_2}{I_1 - I_{10}}\right) A_o F \sqrt{\frac{RT_1}{2\pi M}} \tag{14b}$$

and the cryosurface pumping speed is

$$S_t = A_S \sqrt{\frac{RT_1}{2\pi M}} \tag{14c}$$

Equations (14a, b, c) are the basis of the data reduction for the present experiment.

SECTION III
EXPERIMENTAL APPARATUS

The vacuum chamber used for the experiments (Figs. 1 and 2) is a stainless steel cylinder whose volume of 362 ℓ can be evacuated with a 6-in. oil diffusion pump and a cryopump. The diffusion pump is equipped with an LN_2-cooled cold trap and is backed by a 400 ℓ/min mechanical pump.

Concentric within the main chamber is a copper shroud 24 in. in diameter, 25 in. in length, and sealed at each end. The entire shroud is wrapped with a continuous piece of 5/8-in. copper tubing. An orifice 0.82 in. in diameter was drilled in the center of the bottom plate of the shroud. The shroud is constructed of 1/8-in. copper sheet.

A 7-in. diameter hollow stainless steel sphere is located in the center of the chamber (Figs. 1 and 2). The sphere is supported by a cylindrical vacuum jacket which encloses the transfer lines for gaseous helium (GHe) or LN_2. The sphere could be filled with LN_2 for the cryopumping experiments from the laboratory supply lines. The effective area of the cryosurface is 970 cm^2.

The gas addition system schematic is shown in Fig. 3. The system consists of a gas reservoir, two pressure gages, and two porous plug leaks arranged in a parallel bank. Each leak is made of a small disk of pressed sintered stainless steel and is mounted in a tube (see Ref. 2 for details of this type of leak). The leaks are arranged so that they can be isolated from the system. The gas addition system can be evacuated with a mechanical pump, which is connected to the system.

The instrumentation for measuring gas pressure inside the chamber consists of a mass spectrometer and two Bayard-Alpert-type ionization gages. The mass spectrometer and one of the ion gages are connected to tubulation which penetrates the copper shroud. The other ion gage is used for pressure measurements outside the shroud.

SECTION IV
EXPERIMENTAL PROCEDURE

The technique described in Section II for calibrating vacuum gages requires an accurately known gas flow rate or throughput, Q. The first step in these experiments was to calibrate the porous plug leaks; i. e., to determine the conductance of each leak. To do this, the pressure in the gas reservoir was adjusted to a particular value. Then, the valves on both sides of the leak to be calibrated were opened, permitting the gas to flow through the porous plug and into the evacuated UHV chamber. The decay in gas reservoir pressure, P_f, was then recorded as a function of time.

The conductance, K_L, of each pressed sintered steel leak was calculated by the following equation.

$$K_L = \frac{v}{t} \ln \frac{P_1}{P_2}$$

The gas throughput is defined as $Q = P_f K_L$, where P_f is the forepressure on the porous plug leak and K_L is the leak conductance.

The leak calibration data and corresponding conductance value are presented in Fig. 4. After the initial calibration of the leaks, it was possible to attain known gas flow rates over a range from 5×10^{-6} to 5×10^{-2} torr ℓ/sec.

The mass spectrometer and ion gage were calibrated simultaneously by flowing gas into the shroud at a known rate and then recording the output current of both gages after steady-state conditions were attained. The equilibrium gas pressures in the chamber were calculated from Eq. (2): $P_1 = Q/K_c$.

The procedures used in making the pumping speed measurements are identical to the calibration procedures except that the cryosphere was maintained at a temperature of 77°K by filling with LN_2. The instrument calibration procedure was carried out immediately before every series of pumping speed measurements. The sphere was precoated with a CO_2 flow rate of about 7×10^{-2} torr ℓ/sec^{-1} for 30 min before the experimental runs to provide a solid CO_2 surface (Ref. 3).

SECTION V
RESULTS AND DISCUSSION

By use of Eq. (8), the sensitivity factors of the region 1 pressure gage can be determined from the slope of the curve in Fig. 5 or 6. The ion gage values for CO_2 gas compare favorably with published values (Ref. 4). Each individual gage will vary, however, because of such factors as slight differences in location of elements within the gage, condition of the filament, and type of read-out used with the gage.

The values of $(dI/dQ)_{calibrate}$ for the ion gage and mass spectrometer may be obtained as the slopes of the lines in Figs. 7 and 8. The time rate of change of sensitivity factors was found to be negligible. Gage calibration data were taken immediately before each pumping speed test. The instrument reading caused by background pressure, outgassing, and/or other conditions in the chamber can also be determined from Figs. 5 and 6 as the y-intercepts on the plots of instrument reading (y-axis) versus calculated gas pressure.

The pumping speed constants $(dI_1/dQ)_{cryopump}$ for the ion gage and mass spectrometer may be obtained from Figs. 9 and 10 by taking the slopes. The average pumping speeds of the 77°K sphere, obtained from the ion gage data and mass spectrometer data, were 7.55×10^3 ℓ/sec and 7.83×10^3 ℓ/sec, respectively (see Table I). The corresponding average sticking fractions for CO_2 were 0.82 and 0.85 for the ion gage and mass spectrometer data (Table I). The data were taken over a pressure range of 1×10^{-6} to 6×10^{-6} torr.

The random error in these experiments is rather small, as shown by the close fit of the data to a straight line in Figs. 5 through 10.

In a report to be published shortly (Ref. 5), a sticking fraction of 0.99 for 300°K CO_2 on a 77°K concave cryosurface is reported. This sticking fraction, although much larger than those reported here, was calculated from data taken for a radiation load, 50 percent of the radiation from the 300°K chamber walls. However, it was noted that when the cryosurface of the same experimental apparatus above was irradiated by a hot tungsten filament, the sticking fraction was reduced significantly (15 percent) by this heat load. Thus, it appears that the sticking fraction value is influenced by the amount and possibly by the frequency of the radiation incident upon the cryosurface. Thus, it is emphasized that the sticking fractions listed in this report are those for which the cryosphere was exposed to walls at a temperature of 300°K.

SECTION VI
CONCLUSIONS

The technique described in Section II presents a simple and useful way in which to determine the sensitivity factors for ion gages and mass spectrometers and, thereby, to determine the pumping speed of a cryopump for data taken with these gages. The ion gage sensitivity factor and the mass spectrometer sensitivity factor are the slopes of the lines in the plots of gage reading versus calculated gas pressure. The value

TABLE I
TABULATED STICKING FRACTION, SENSITIVITY FACTOR, AND GAGE CALIBRATION FACTORS

Ion Gage Data					Mass Spectrometer Data				
C	γ	$\left(\frac{dI_1}{dQ}\right)_{Cal}$, sec/torr-$\ell$	$\left(\frac{dI_1}{dQ}\right)_{Pump}$, sec/torr-$\ell$	$\left(1 - \frac{I_2}{I_1 - I_{10}}\right)^{\ddagger}$	C	γ, amp/torr	$\left(\frac{dI_1}{dQ}\right)_{Cal}$, $\frac{\text{amp-sec}}{\text{torr-}\ell}$	$\left(\frac{dI_1}{dQ}\right)_{Pump}$, $\frac{\text{amp-sec}}{\text{torr-}\ell}$	$\left(1 - \frac{I_2}{I_1 - I_{10}}\right)^{\ddagger}$
0.87	1.395	5.45×10^{-2}	1.734×10^{-4}	0.91	0.88	8.48×10^{-3}	3.31×10^{-4}	1.045×10^{-6}	0.91
0.88	1.34	5.23×10^{-2}	1.65×10^{-4}	0.91	0.90	13.05×10^{-3} (div/torr)	5.09×10^{-4} $(\frac{\text{div-sec}}{\text{torr-}\ell})$	1.57×10^{-6} $(\frac{\text{div-sec}}{\text{torr-}\ell})$	0.91
0.76*	1.266	4.40×10^{-2}	1.81×10^{-4}	0.95	0.81*	7.45×10^{6}	2.59×10^{5}	0.998×10^{3}	0.95
0.78*	1.10	3.81×10^{-2}	1.53×10^{-4}	0.95	0.82*	7.61×10^{6}	2.64×10^{5}	1.005×10^{3}	0.95

*These values were calculated from data taken in the UHV chamber with the following modifications. The theoretical orifice conductance was increased to 30.2 ℓ/sec from 28.1 ℓ/sec for 300°K CO_2, and different Veeco® ion gages and a Veeco mass spectrometer were used.

‡This column is the effect of back pressure on the orifice conductance.

of γ for CO_2 is the nominal value and attests to the validity of the experiment. The value of γ for the mass spectrometer is primarily a function of the current and voltage setting for the mass spectrometer.

The technique assures that vapor pressures, outgassing rates, and any other background conditions are taken into account. The combined effect of the above conditions can be determined from the gage reading at zero inflow. The resulting value of the pumping speed for 300°K CO_2 was 8.1 ℓ/sec-cm^2 yielding a sticking fraction of 0.85. The method is quite useful for in-place gage calibrations and pumping speed measurements in systems of the same basic physical configuration as described.

Since it is apparent that a radiative heat load can change the pumping speed of a cryosurface, it would be of interest to explore this effect further. This effect could possibly be the explanation for some of the variation between reported values of the sticking fraction for 300°K CO_2. Thus, it is emphasized that the sticking fractions reported are calculated from data taken when the 77°K cryosurface was exposed to radiation from 300°K copper walls.

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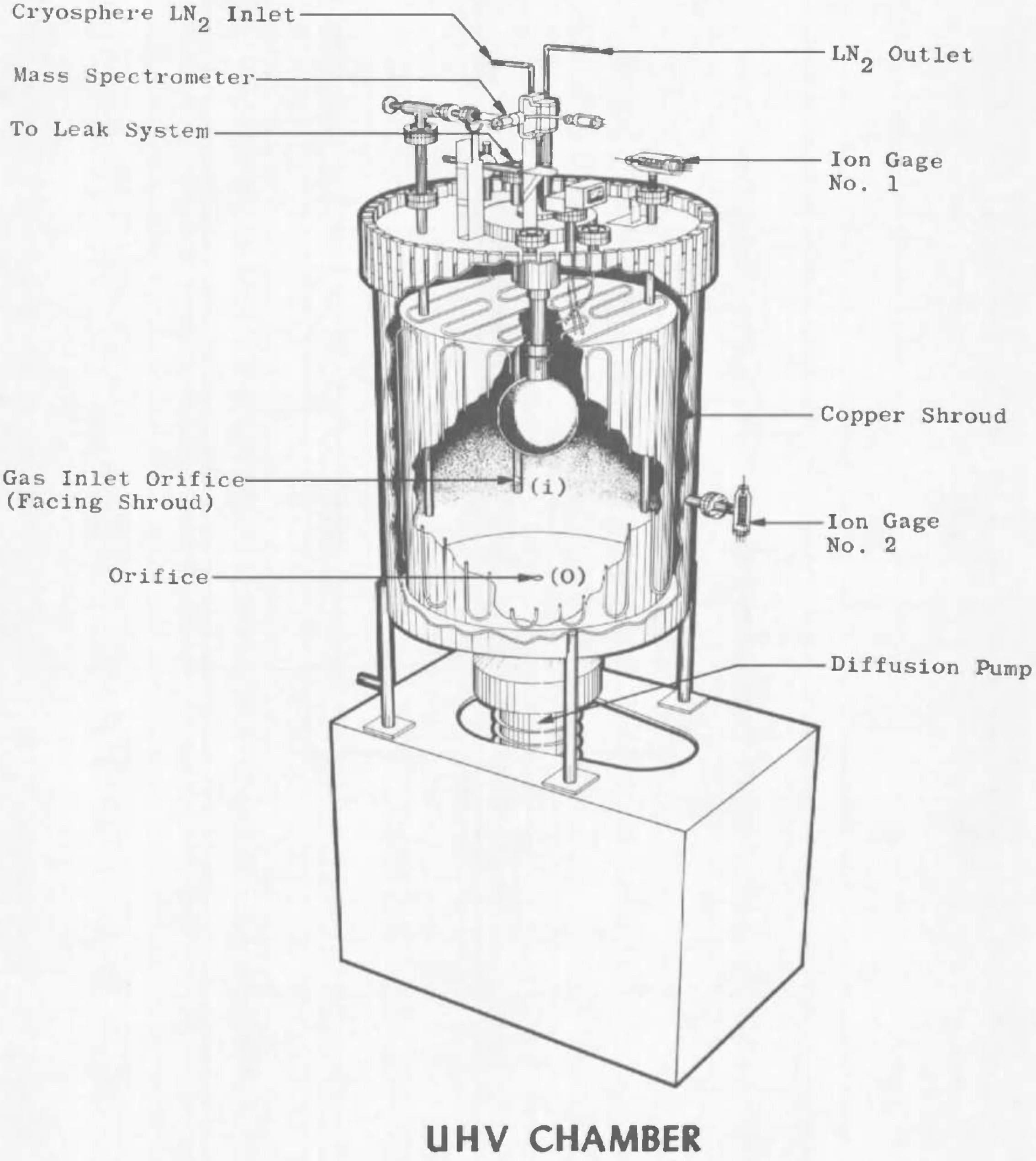


Fig. 1 UHV Chamber

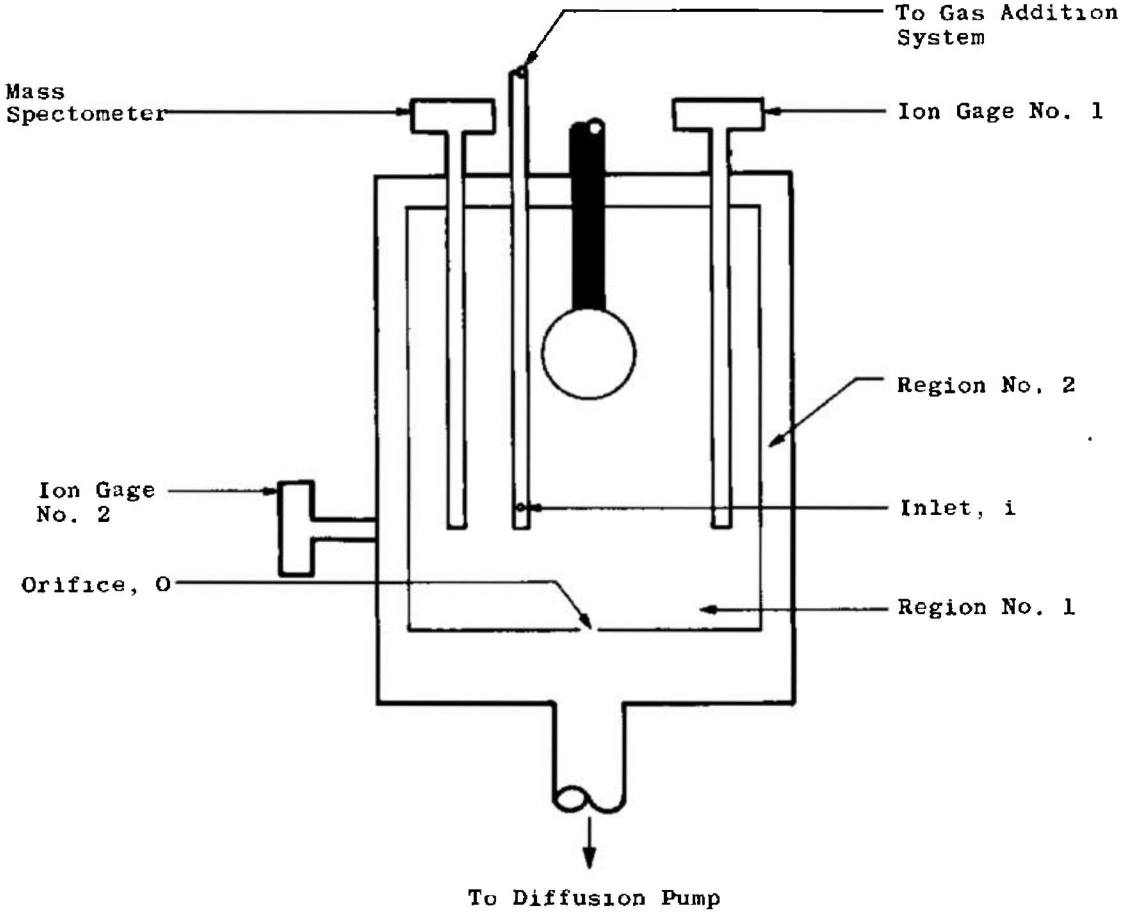


Fig. 2 UHV Chamber Schematic (Internal Configuration)

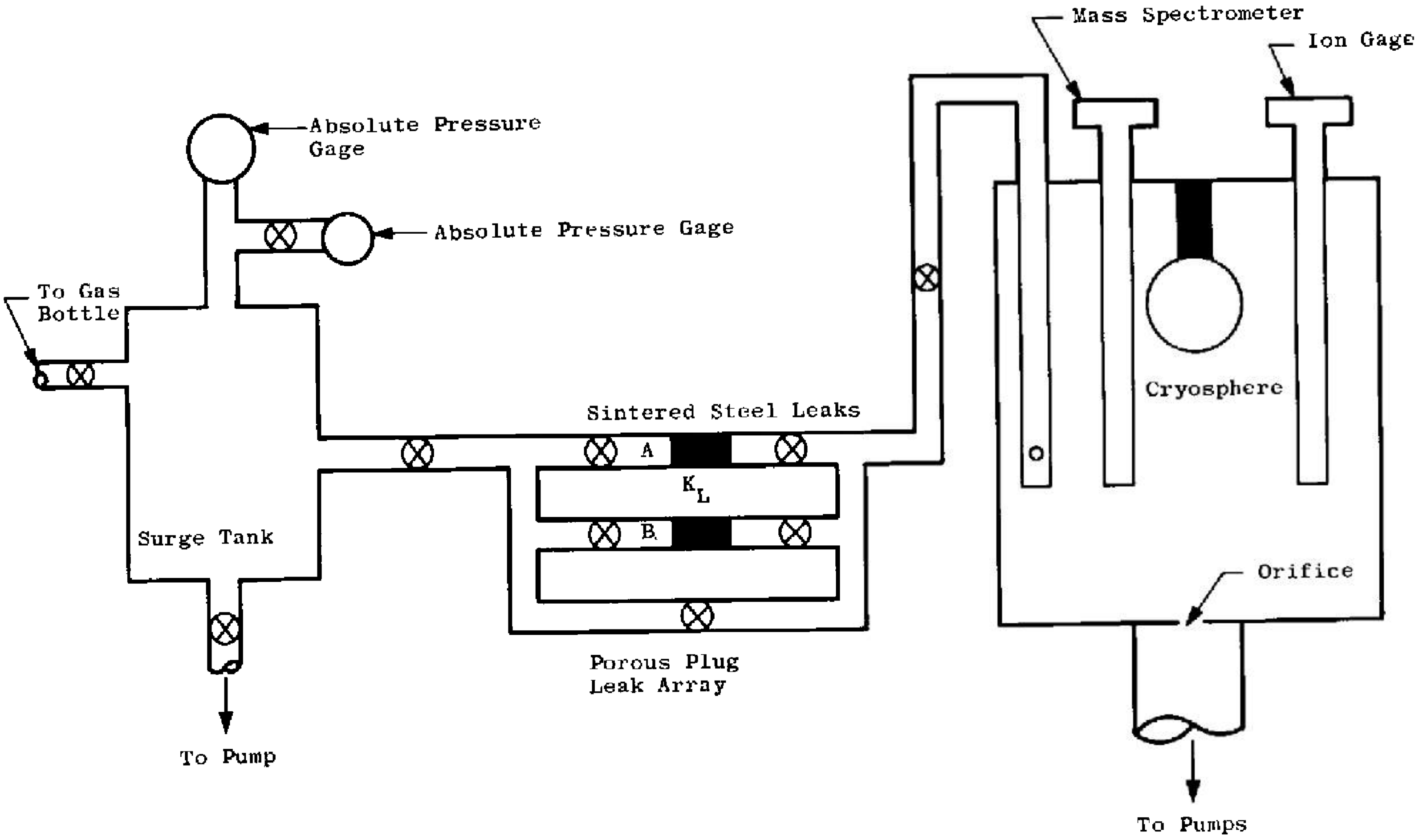
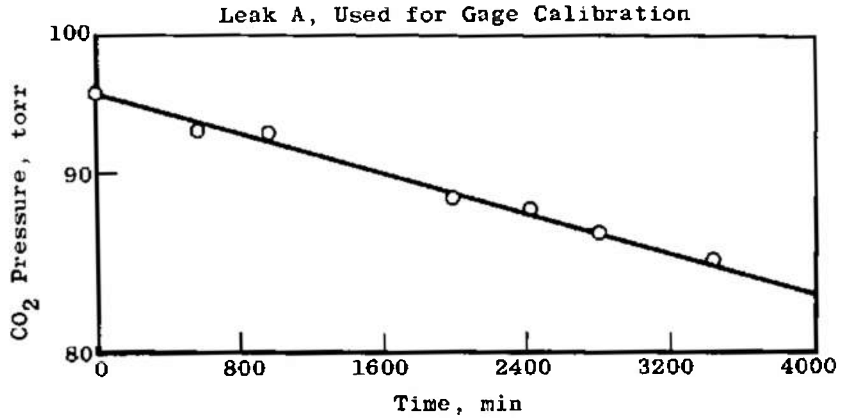
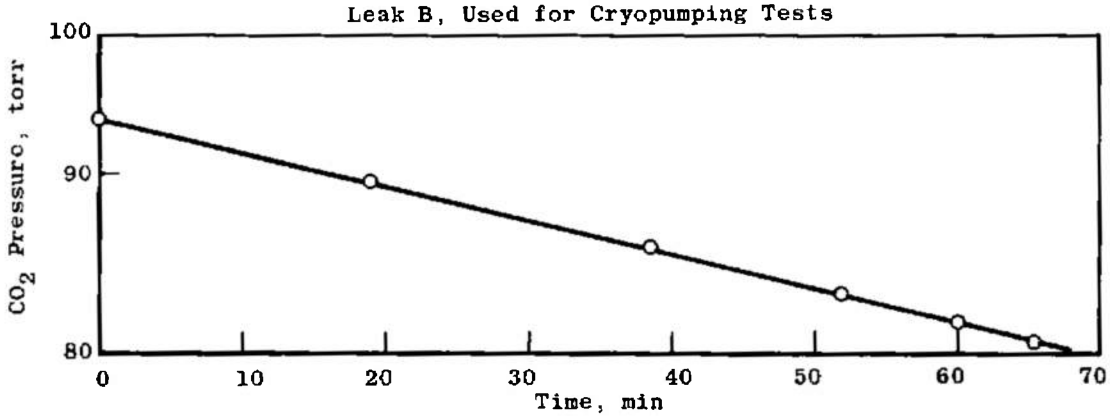


Fig. 3 UHV Gas Addition System Schematic


Leak A, Used for Gage Calibration

$K_A = \frac{v}{t} \ln (P_1/P_2)$ Where $v = 9.145$ ℓ

$K_A = 5.50 \times 10^{-6}$ ℓ/sec


Leak B, Used for Cryopumping Tests

$K_B = \frac{v}{t} \ln (P_1/P_2)$ Where $v = 9.555$ ℓ

$K_B = 3.68 \times 10^{-4}$ ℓ/sec

Fig. 4 Sintered Steel Leak Calibration

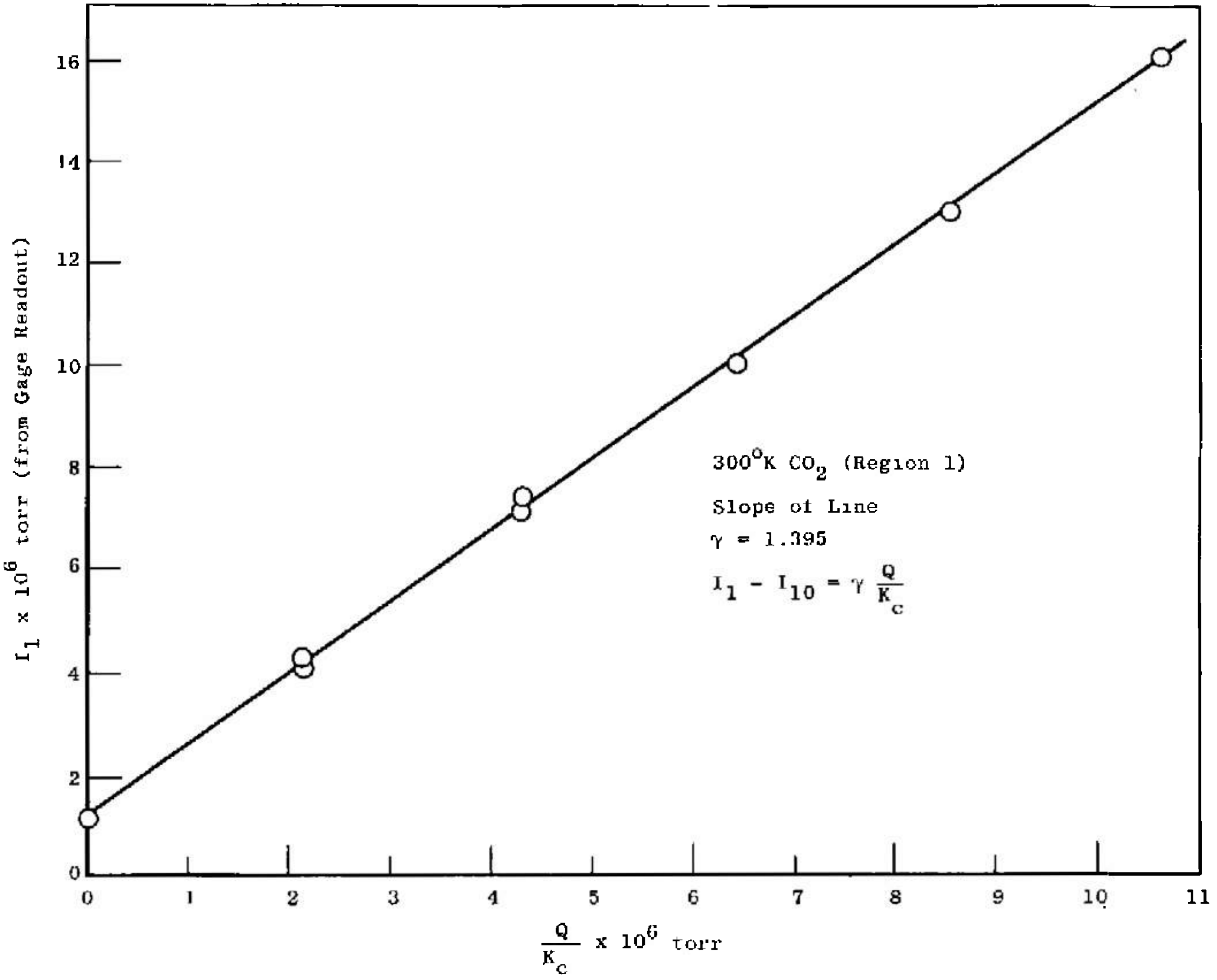


Fig. 5 Ion Gage Calibration Sensitivity Factor

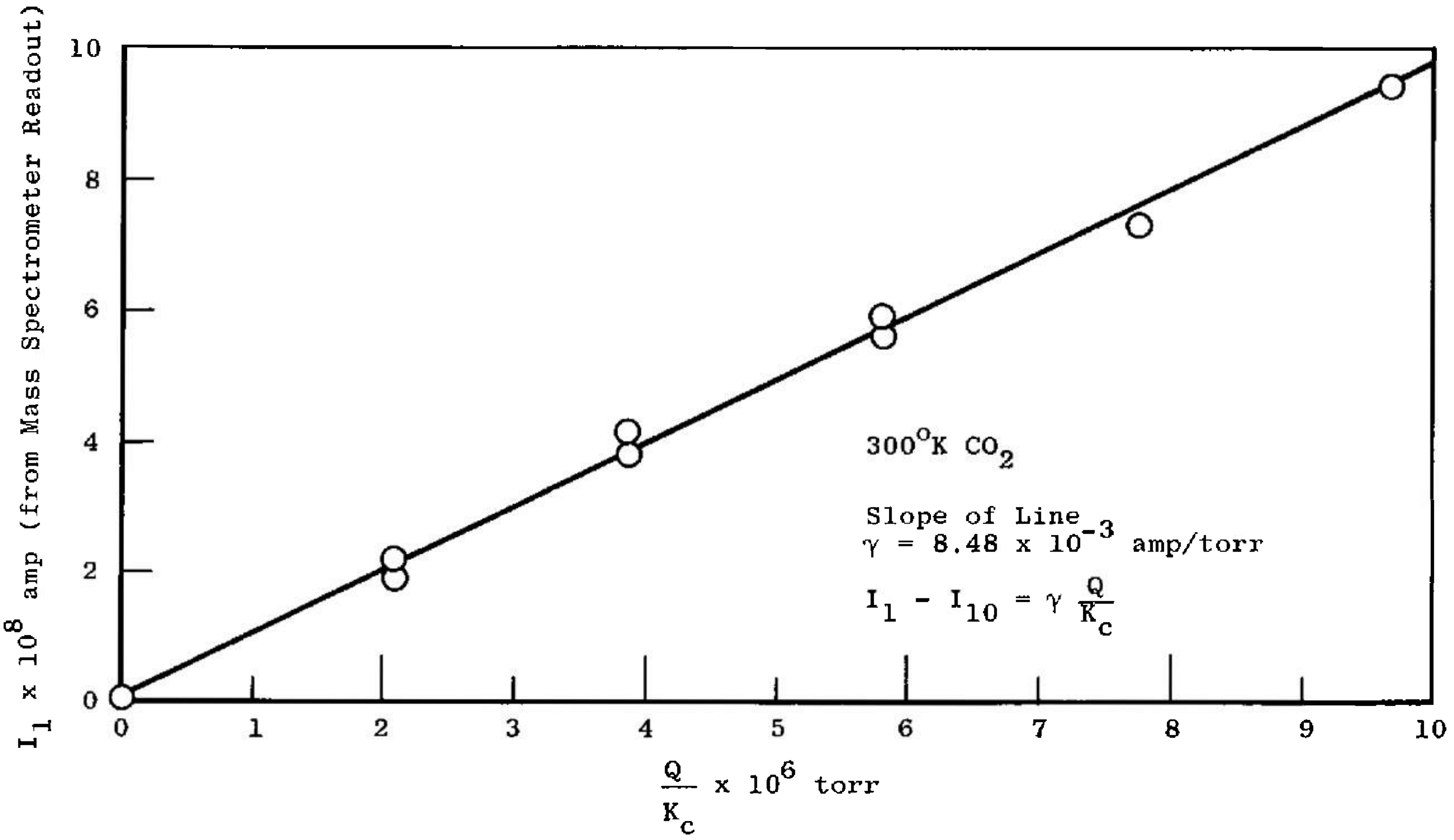


Fig. 6 Mass Spectrometer Calibration Sensitivity Factor

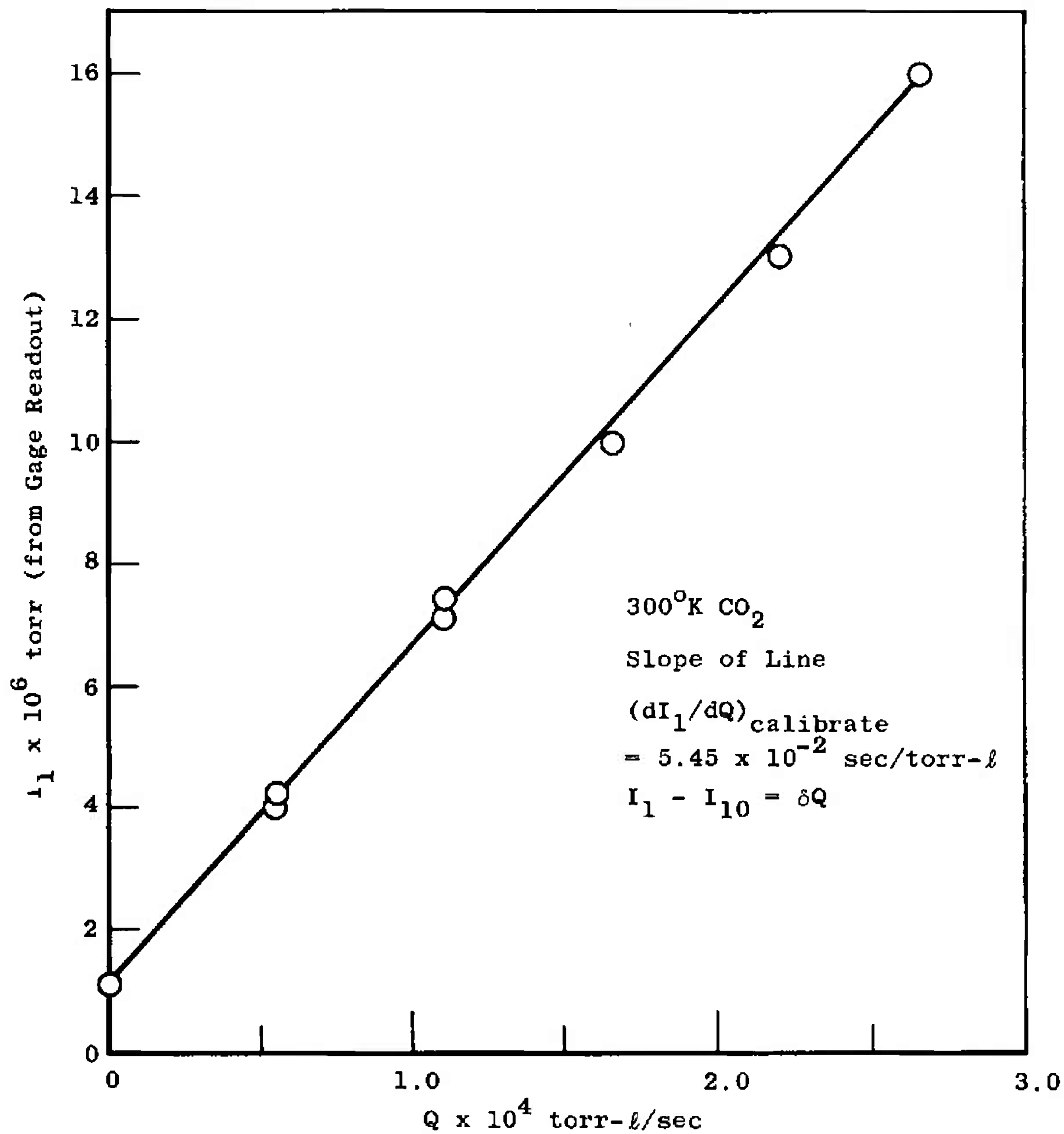


Fig. 7 Ion Gage Calibration

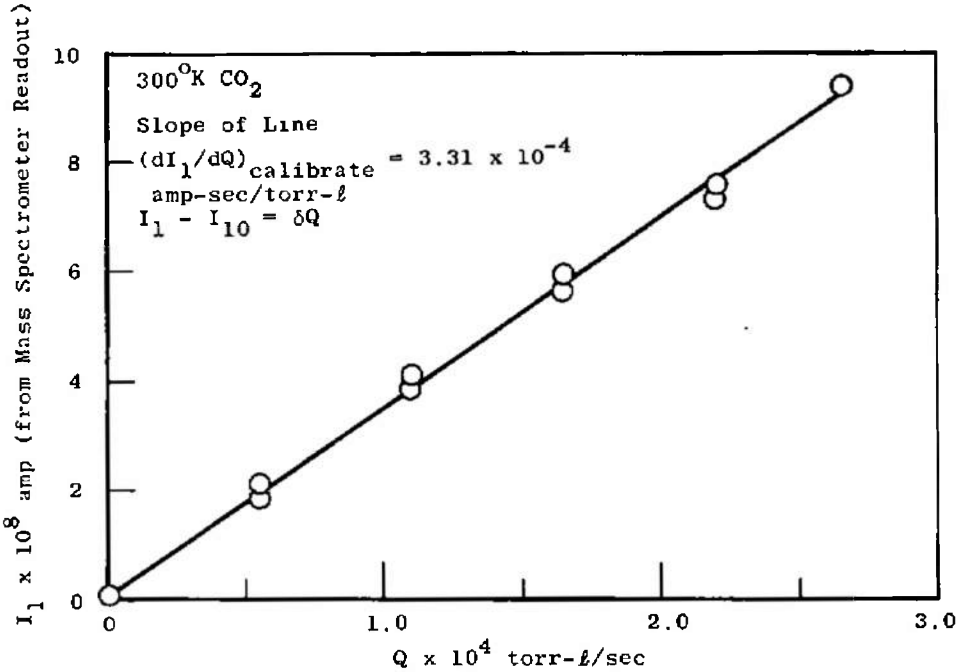


Fig. 8 Mass Spectrometer Calibration

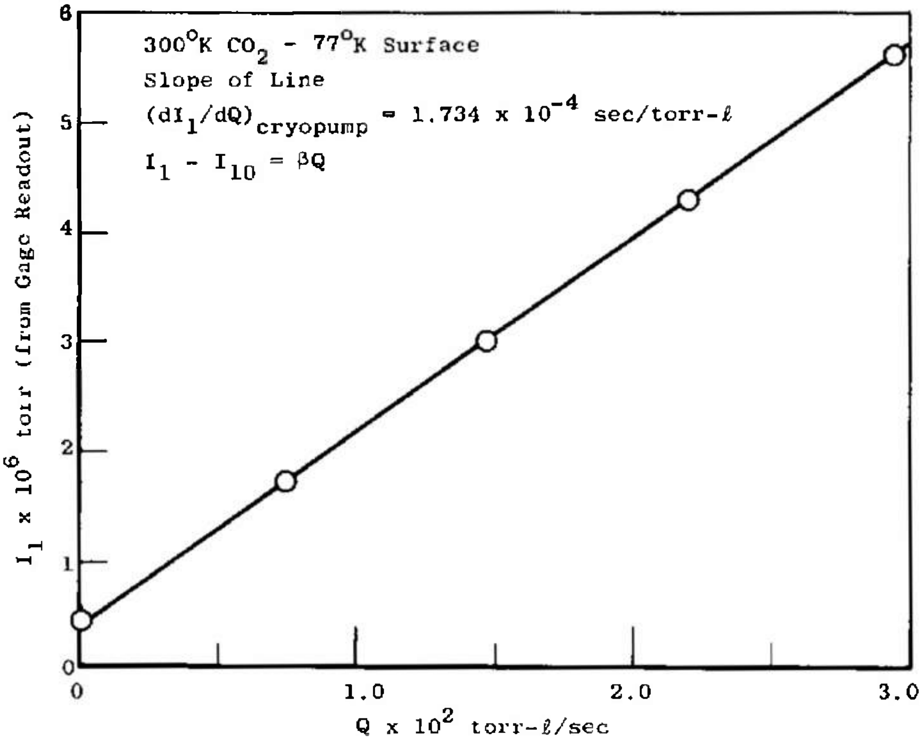


Fig. 9 Ion Gage Pumping Speed

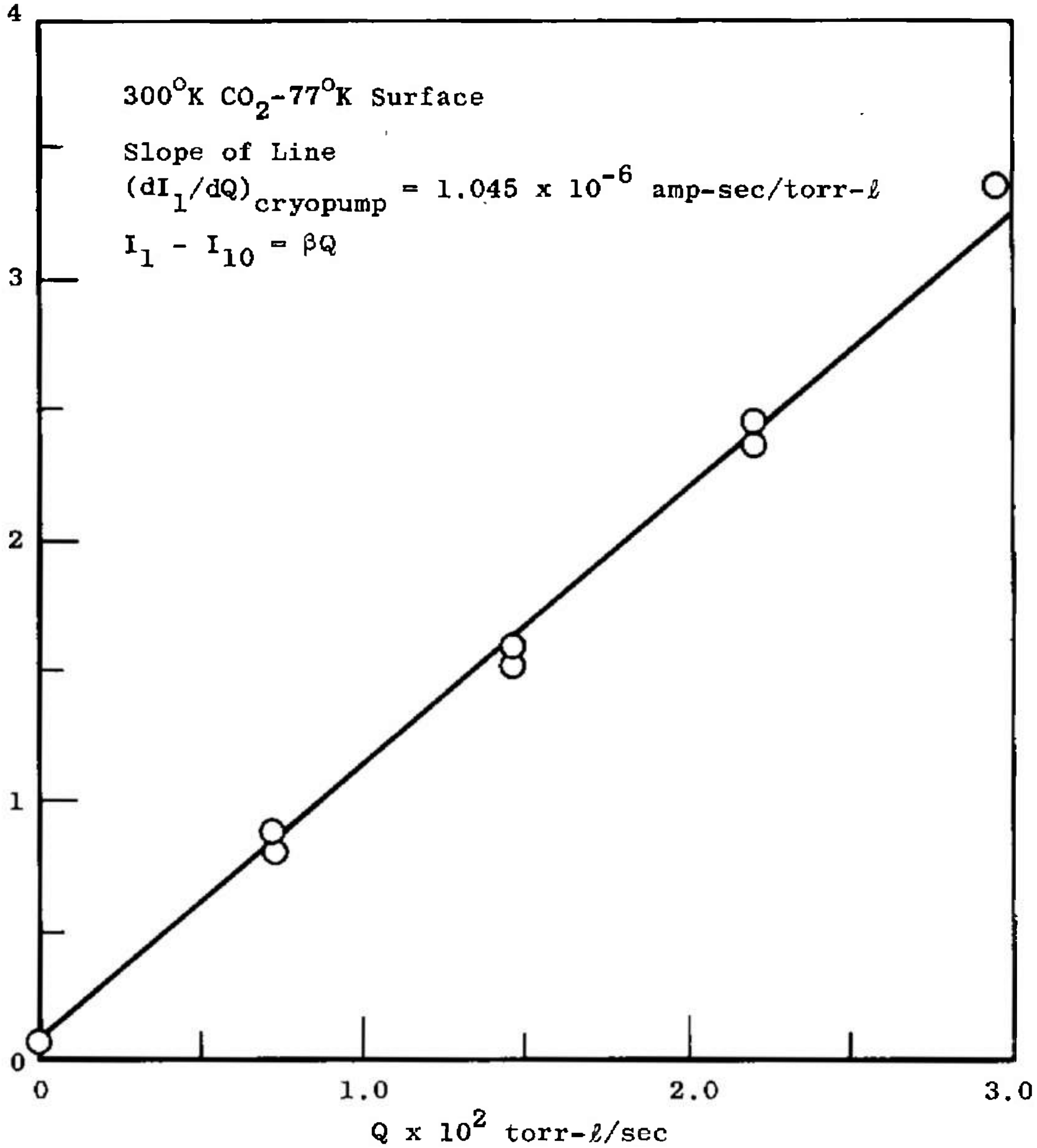


Fig. 10 Mass Spectrometer Pumping Speed

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13 ABSTRACT

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vacuum						
gage						
cryopumping						
4 spectrometers						
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17-5

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