FACILITY FORM 602

N67 16673
(ACCESSION NUMBER)

135
(PAGES)

CR-54934
(NASA CR OR TMX OR AD NUMBER)

(THRU)

1
(CODE)

17
(CATEGORY)

NASA-CR-54934

GPO PRICE $

CFSTI PRICE(S) $

Hard copy (HC) 3.00

Microfiche (MF) 1.95

ff 653 July 65

CONTAMINATION EXPOSURES OF COLUMBIUM AND TANTALUM ALLOY SPECIMENS

FINAL REPORT

Period: February 25, 1965 to June 15, 1966

R. B. FLEMING

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
CONTRACT NAS 3-4169

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

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Prepared By
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Technical Management
NASA - Lewis Research Center
R. A. Lindberg

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

FOREWORD

The work described herein was performed under National Aeronautics and Space Administration Contract NAS 3-4169. Experimental work was performed at the General Electric Company Research and Development Center in Schenectady, New York, by Messrs. W. S. Knapp and K. H. Malmstrom; project engineering and report preparation were performed by Dr. R. B. Fleming. The program was administered for the General Electric Company by Dr. J. W. Semmel, Jr., Manager, Materials and Processes, Space Power and Propulsion Section, Missile and Space Division in Cincinnati, Ohio. Mr. R. A. Lindberg acted as Project Manager for the National Aeronautics and Space Administration, and Mr. C. A. Barrett of the Lewis Research Center contributed significantly to technical aspects of the program.

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1. INTRODUCTION AND SUMMARY

The program described by this report was conducted to determine the oxidation rate, or gettering rate for oxygen, of columbium, zirconium, and tantalum alloy specimens under conditions of low pressures and high temperatures.

A total of 15 exposures were conducted during the program, with oxygen partial pressures at levels ranging from about 10^{-5} torr to 5×10^{-8} torr, and temperatures at levels of 885°, 950°, 980°, and 1100°C.

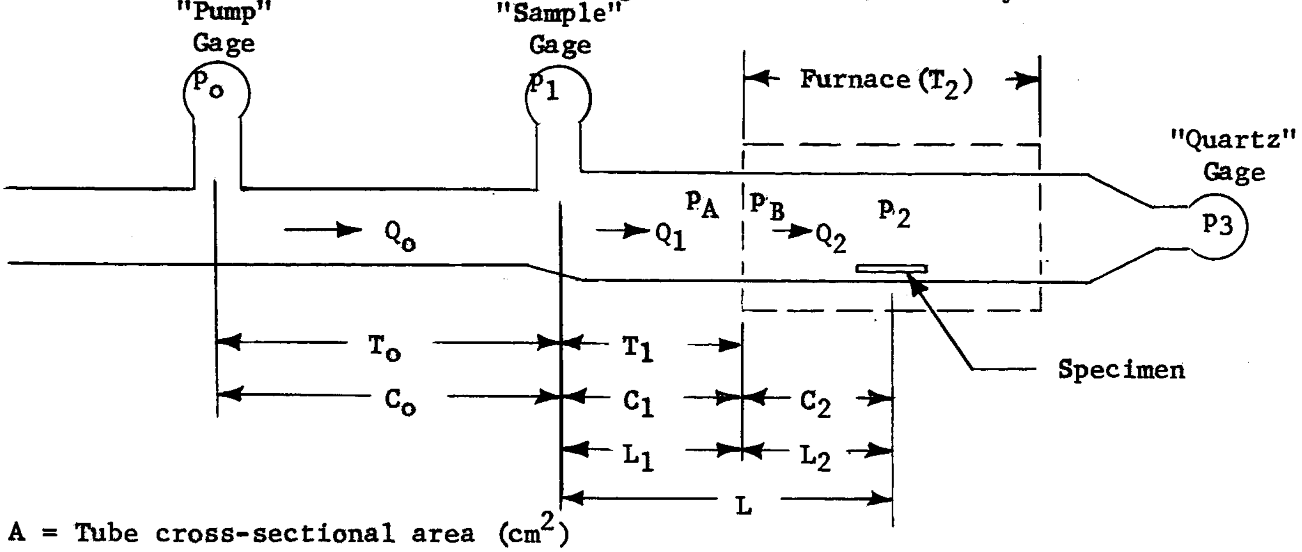
The gettering rates were established by the method of measuring the pressure drop across a known flow conductance, from which the oxygen gas mass flow rate (equal to the gettering rate) can be computed. A knowledge of the pressure directly over the specimen, plus knowledge of the gettering rate, can then be used to calculate the "sticking factor" (that fraction of total molecules striking the specimen, which do not rebound). The sticking factor is the item of primary interest in these experiments.

In addition to the 15 exposures, the program included several experiments which were conducted to determine various pressure gage characteristics, and an analysis was made of pressure distribution throughout the system.

The scope of the investigation was confined to exposing the specimens to the various environments and documenting the data and experimental conditions. Subsequent evaluation of the exposed specimens by chemical and metallurgical techniques, and reduction of data, were conducted at the Lewis Research Center of the National Aeronautics and Space Administration. The work performed by the General Electric Company was done at G.E.'s Research and Development Center in Schenectady at the request of the Space Power and Propulsion Section, Missile and Space Division.

2. NOMENCLATURE

The sketch below and the following list will define the symbols used.



A = Tube cross-sectional area (cm^2)

B = A constant; see Section 5

C = Conductance, defined by $Q = C \triangle p$ (liters/sec)

D = Internal tube diameter (cm)

G = Mass flux (g sec^{-1} cm^{-2})

K = Clausing factor, a function of L/D

L = Length, along tube axis (cm)

$\dot{m}$ = Mass flow rate (g/sec)

M = Molecular weight of gas (g/mole)

p = Pressure (torr)

Q = Flow rate in terms of pressure x volume flow rate (proportional to mass flow rate at a given temperature) (torr liters/sec)

R = Gas constant (62.3 torr liter $mole^{-1}$ deg K^{-1}) or (8.31×10^7 dyne cm $mole^{-1}$ deg K^{-1})

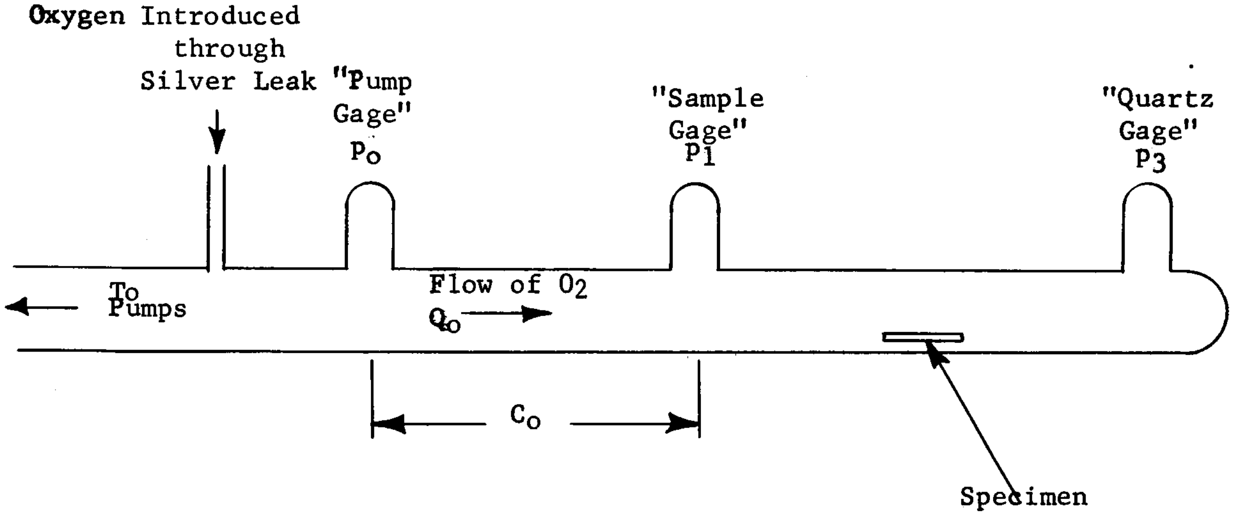
T = Absolute temperature (deg K)

$\dot{V}$ = Volume flow rate (liters/sec)

3. EXPERIMENTAL APPARATUS

Exposures were conducted in two different vacuum systems. One system, which was used during the period preceding the present program, and modified for the present program, is designated the "10^{-8} system" inasmuch as the system is capable of achieving pressures in the low 10^{-8} torr range after bake-out. The other system, constructed early in the present program, is designated the "10^{-10} system" because the system -- by virtue of its design and construction -- is able to reach the mid 10^{-10} torr range after bake-out.

The schematic below is common to both systems, and indicates the scheme used to determine gettering rates.



System Schematic

The gas flow rate, Q_o, is determined by measuring the pressures, p_o and p_1, and then using the equation

$$Q_o = C_o (p_o - p_1)$$

where C_o is the known flow conductance between pressure gages. In the systems, this conductance consists of a right circular cylinder of accurately measured dimensions, from which C_o is calculated.

Details on the construction of the flow conductances and calculation of C_o are given in the Appendix, Section 9-1..

The ion gage measuring pressure p_3 is used, with a thermal transpiration correction,[*] to determine the pressure directly over the specimen, p_2. This pressure is needed to establish the sticking factor.

3-1. Description of 10^{-8} System

The 10^{-8} system is shown schematically in more detail in Figure 1, with dimensions of various elements given. Not shown is the pumping system, which consists of a 115 liter/sec oil diffusion pump, a non-migrating optically-tight liquid nitrogen trap, and a forepump.

A photograph of the 10^{-8} system is given in Figure 2, showing the specimen furnace removed and the induction heater coil in place. For bake-out of the entire system, a large bake-out furnace was lowered over the table, and the strip heaters were used to produce a 350°C bake-out temperature.

Introduction of oxygen to the system was accomplished with a heated silver permeation membrane. It was found that with constant specimen gettering rate and with approximately one atmosphere of O_2 applied to the membrane, the system pressures held constant for many days. The silver membrane leak was found to be relatively insensitive to small O_2 pressure changes on the atmosphere side.

The vacuum system usually reached the low 10^{-8} torr range after bake-out, before O_2 was admitted. This was sufficiently low, inasmuch as the lowest exposure pressure was 10^{-7} torr.

The specimen was normally brought up to temperature by a resistance-heated furnace. This furnace also heated the quartz walls surrounding the specimen. In order to test the effect of room temperature walls, an induction heater was used for two exposures (No. 12 and No. 15).

(*) For example of thermal transpiration correction, see Appendix, Section 9-2.

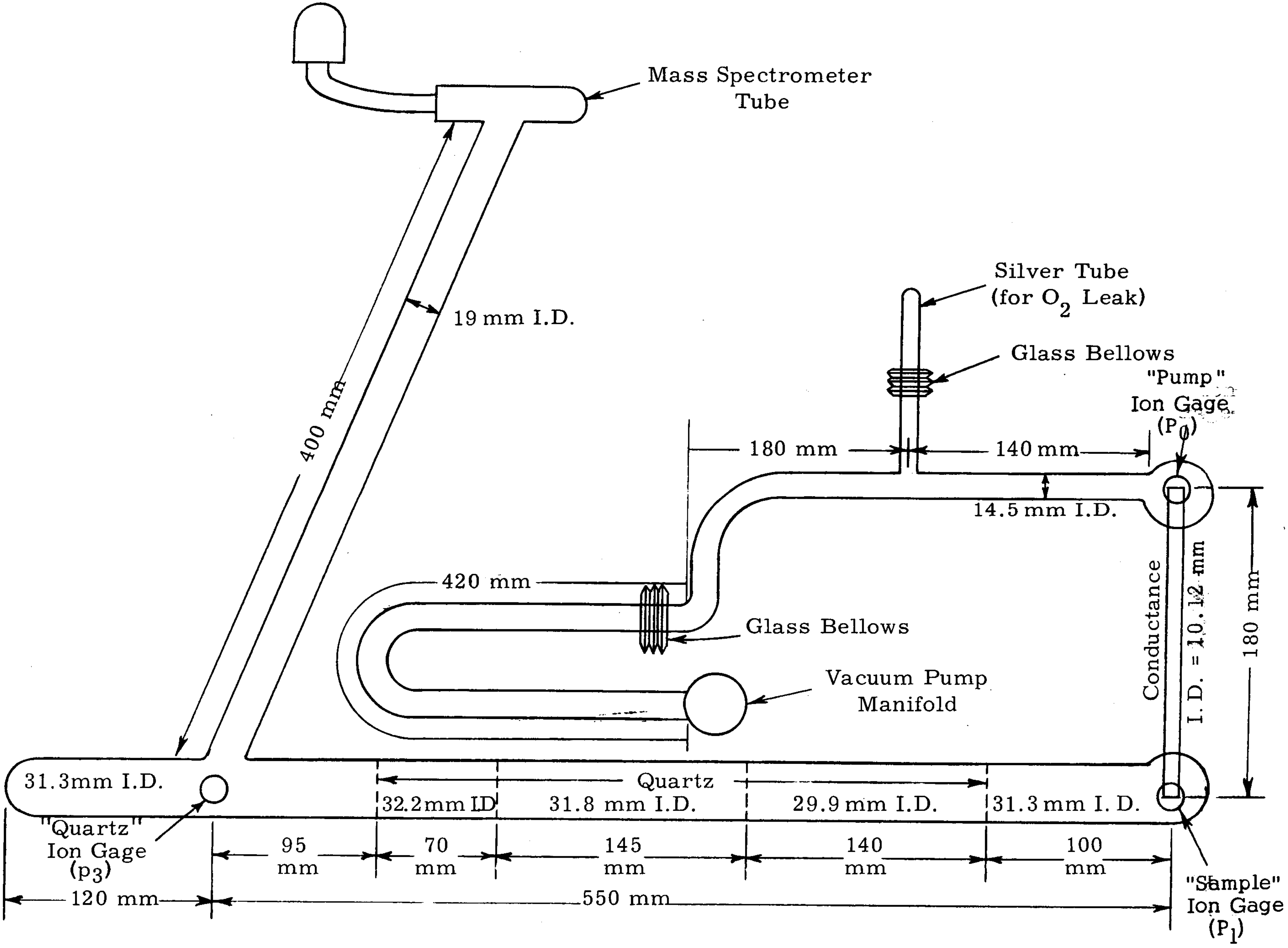


Figure 1. Schematic, 10^{-8} System.

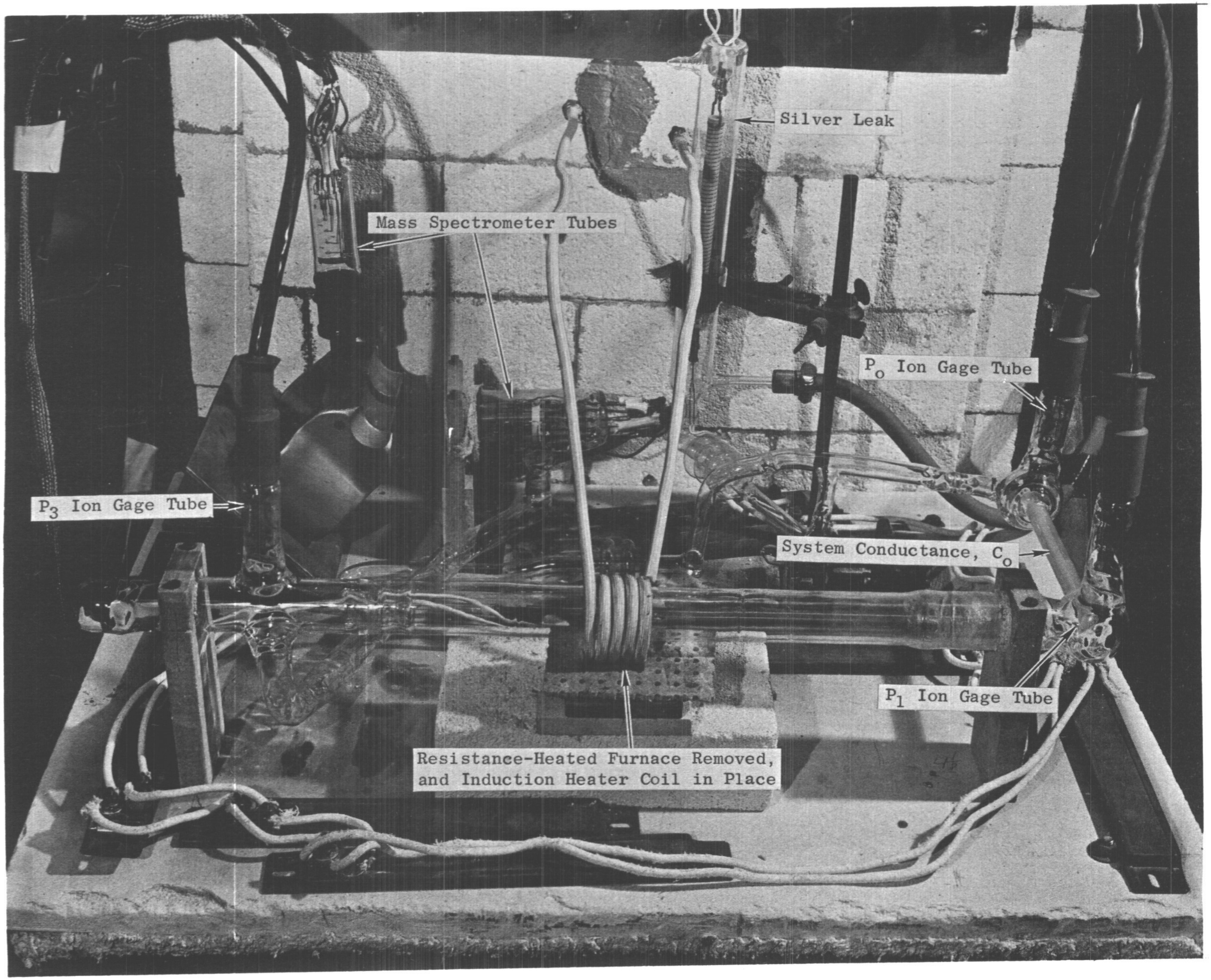


Figure 2. Photograph of 10^{-8} System

System is shown with resistance-heated furnace removed and induction heater in place. Conductance is at far right, silver leak is at rear center, mass spectrometer tube is at rear left.

3-2. Description of 10^{-10} System

The 10^{-10} system is shown schematically in detail in Figure 3. The significant differences between the 10^{-10} and 10^{-8} systems are; (1) the 10^{-10} system has considerably higher pumping speed and therefore can attain lower pressures because of larger diameter piping between traps and system; (2) on the 10^{-10} system there are two traps (instead of one) in series, with the trap nearest the system being a bakeable trap; and (3) the 10^{-10} system has a double-walled quartz tube surrounding the specimen, with provision for vacuum pumping between the walls, to prevent helium permeation.

Photographs of the 10^{-10} system are given in Figures 4 and 5.

The pumping system for the 10^{-10} system consisted of the two non-migrating optically-tight traps in series, each of which was cooled with liquid nitrogen, two 115 liter/sec diffusion pumps in series, and a forepump. The upper trap was bakeable with the system.

Bakeout and introduction of oxygen into the system were accomplished the same as in the 10^{-8} system.

After bakeout, but before admission of oxygen, pressures in the 10^{-10} system usually reach the mid to high 10^{-10} torr level. However, two overnight bakeouts were generally required to reach this pressure.

3-3. Instrumentation

Ion gages

The ion gage tubes were General Electric model 22GT-102, which contain one thoria-coated iridium filament and one tungsten filament. Only the thoria-coated filaments were used. These filaments were preferred to the tungsten because of longer life and a reduction in the rate of conversion of O_2 to CO at the filament.

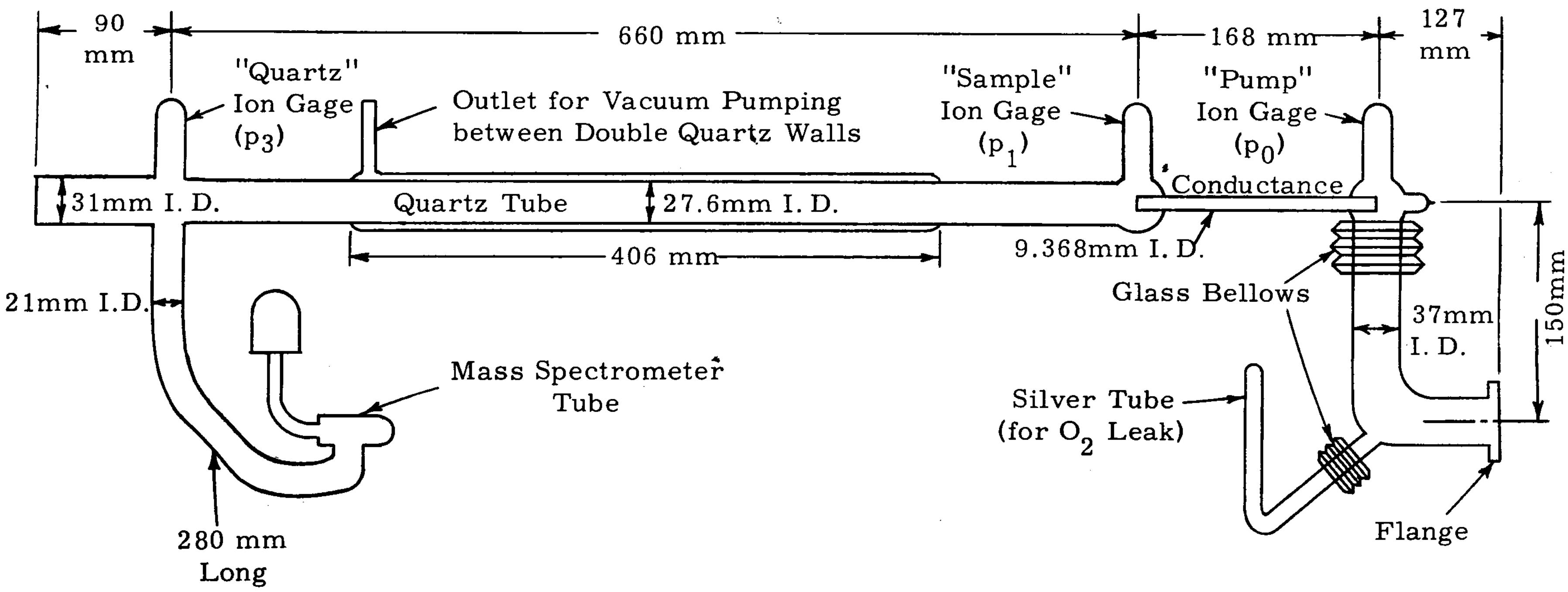


Figure 3. Schematic of 10^{-10} System.

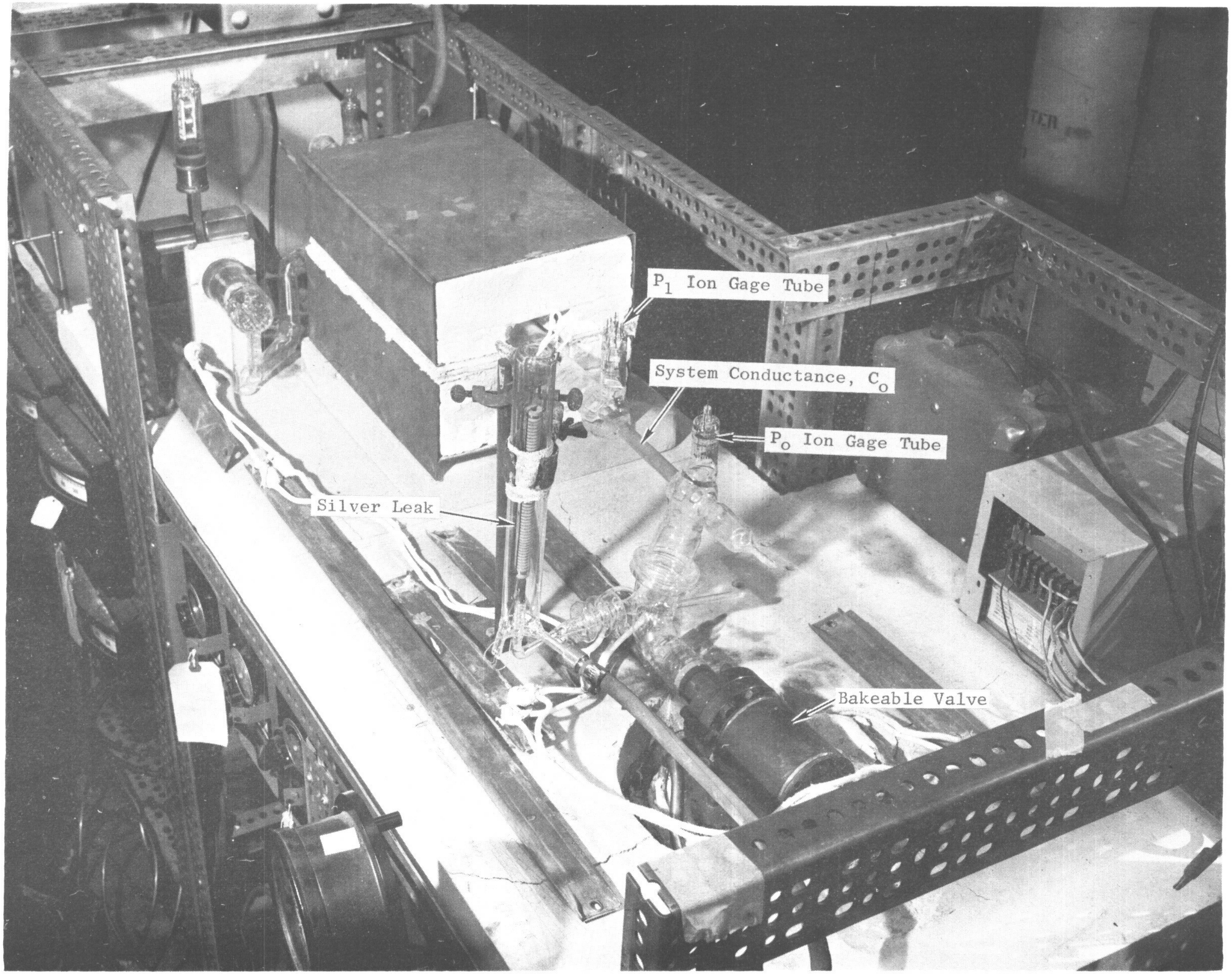


Figure 4. Photograph of 10^{-10} System

Bakeable valve is at front. Silver leak and conductance are at center. Resistance-heated furnace and mass spectrometer tube are in background.

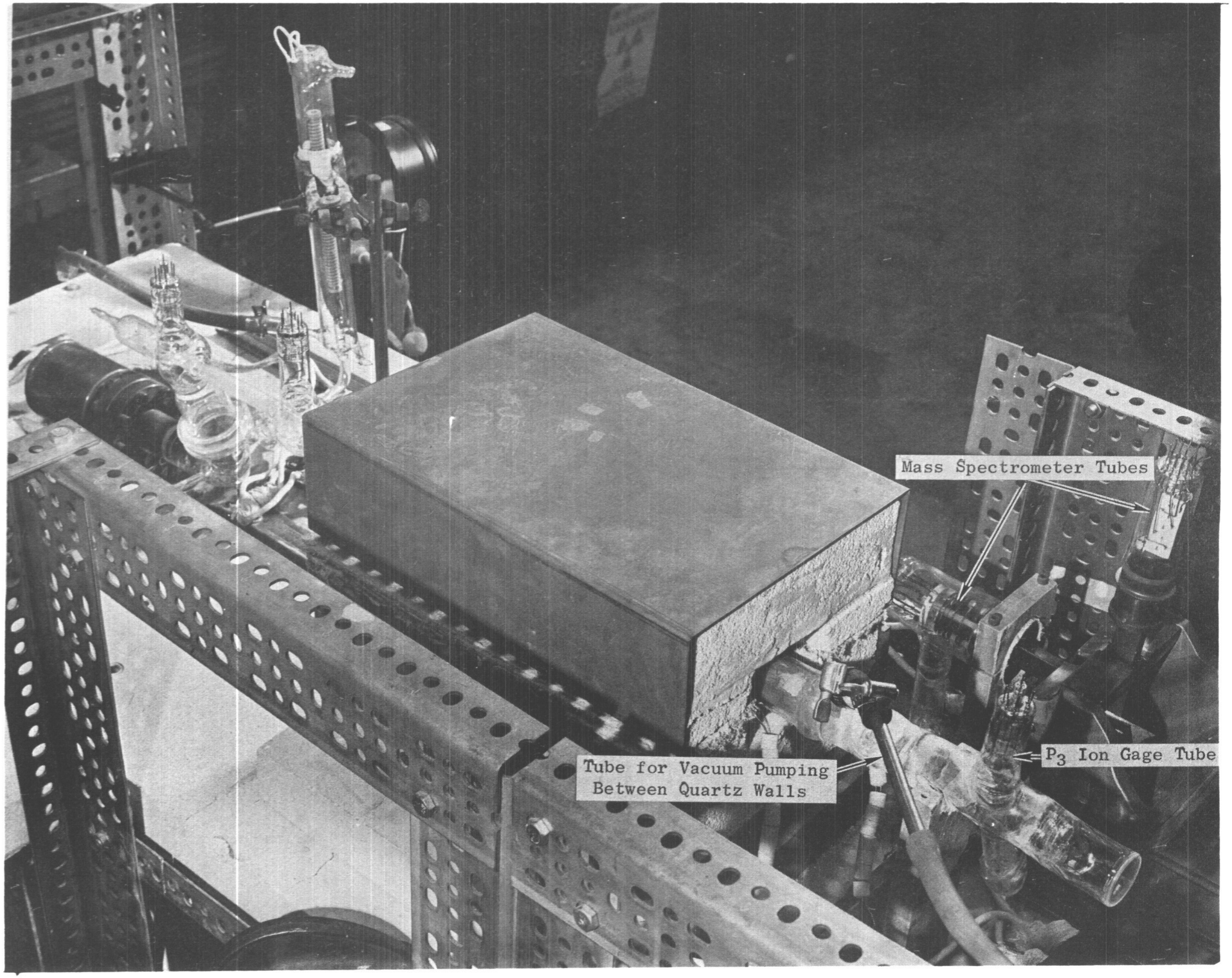


Figure 5. **Photograph of 10^{-10}** System

View shows p_3 ion gage and mass spectrometer tube at lower right. Rubber hose at lower right is for **pumping** between the two quartz walls.

The ion gage tubes were operated initially at 0.1 ma. However, after some instabilities were observed in their operation(1) it was decided to increase the emission current to 1 ma. This was done just prior to the third exposure, and the gages were re-calibrated at 1 ma.

Two types of ion gage control panels were used. On one system, three Consolidated Vacuum Corporation (C.V.C.) type GIC-100 ion gage controllers were used. On the other system, there were three General Electric model 22GC-101 panels. Just prior to the 11th exposure, the control panels and tubes were switched between systems, so that the General Electric control panels ended up on the 10^{-8} system and the C.V.C. control panels ended up on the 10^{-10} system. The reason for this switch was that the General Electric control panels did not appear to be affected by the high frequency field created by the induction heater which was installed on the 10^{-8} system toward the end of the program.

Although the gage tubes and G. E. panels were not affected by the field set up by the induction heater, there was initially (prior to the 12th exposure) a problem with a gage instability which seemed to be due to a charge building up on the glass walls after the induction heater was on for a period of time. This problem was eliminated by wrapping aluminum foil around the main glass tube near each ion gage, on the induction-coil side of the gage. The aluminum foil was then electrically grounded. The "quartz gage" (p_3) was also shielded with a grounded flat copper plate on the induction coil side of the gage.

Mass Spectrometer

The mass spectrometer tubes used to determine gas composition were

---

(1)These instabilities may have been similar to those described by G. Carter and J. H. Leck: "Bistable Behavior of the Bayard-Alpert Ionization Gauge", British Journal of Applied Physics, Vol. 10, August, 1959, p. 364. In addition, errors at low emission currents were described by J. W. Ackley, C. F. Lothrop, and W. R. Wheeler, in the 1962 Transactions of the American Vacuum Society. Both conditions (the instability and the low emission error) were predicted to be improved by increasing the emission current to 1 ma. No further difficulties were experienced at 1 ma emission.

General Electric model 22PT-103 Glass Analyser Tubes with thoria-coated iridium filaments operating at 1 ma electron emission. The control panel used for read-out was a General Electric Partial Pressure Analyser Model 514. The mass spectrometers were calibrated with known gases at the beginning of the program.

Temperature Measurement

Measurement of specimen temperature was by means of a Leeds & Northrup Optical Pyrometer, Catalog No. 8622-C. The pyrometer was calibrated prior to the start of the program. In this initial calibration, two different specimens of columbium were observed through a belljar at a true temperature of 1110 deg C (the true temperature being established by thermocouples). The average "effective emissivity" (after correction for the transmittance of the glass) was calculated from these observations to be 0.58. This effective emissivity was then used with the standard Planck's law formula to establish a table of observed and corrected temperatures at a wave length of 0.65 microns. Selected values from this correction table follow:

Observed Temperature		True Temperature		ΔT Correction
Deg K	Deg C	Deg K	Deg C	
1125	852	1157	884	32
1215	942	1252	979	37
1330	1057	1375	1102	45

These values can be found in "Corrected Optical Pyrometer Readings", N. B. S. Monograph 30, 1961, by D. E. Poland, J. W. Green, and J. L. Margrave.

The small variation in emissivity with temperature was not accounted for, as the effective emissivity of 0.58 was assumed constant throughout the above range.

At the end of the last exposure, the pyrometer was calibrated again, this time against a standard tungsten ribbon filament which had been calibrated by the N. B. S., and which had an uncertainty of ± 3 deg C in the 900 to 1100 deg C range. The pyrometer reading was found to be within ± 3 deg K of the "brightness temperature" of the standard filament throughout the range of interest.

Therefore, the brightness temperature indication of the pyrometer is quite accurate (to within perhaps ± 6 deg C); the only question is regarding the specimen emissivity. If the effective emissivities of all specimens were close to 0.58 (corresponding to an actual emissivity of about 0.63 before correcting for glass losses), then the specimen temperatures given in this report are estimated to be accurate to within about ± 6 deg C. However, the range of spectral emissivities for columbium and tantalum in the literature is quite large, possibly from as low as 0.37 to as high as 0.77 at a wave length of 0.65 microns.

The Thermophysical Properties Research Center at Purdue University has supplied the value (under TPRC Serial No. 28741) for spectral emissivity of columbium at 0.65 microns of 0.42 (a value which holds quite well over the range 1100 deg K to 1500 deg K). The value of 0.42 when corrected for glass losses gives an effective emissivity of 0.39; the resulting ΔT correction at 980 deg C true temperature would be about 64 deg C, compared with the correction of 37 deg C used in this experiment (see table above). Thus, the correction used could be 27 deg C in error, under the above assumptions, at a true temperature of 980 deg C.

4. EXPERIMENTAL PROCEDURES

A condensed list of steps in preparing the apparatus and conducting the exposure is given below.

4-1. Preparation of Apparatus

(a) The first step in preparation was to load the specimen on a "sleigh" and then to seal off the glass system. The specimen was first located on the sleigh near the p_3 ion gage so that the specimen furnace could subsequently be raised in temperature (see step (f) below) while the specimen still remained cool. The specimen was generally located with identifying numbers facing upwards and at the end facing toward the oncoming oxygen stream.

(b) The next step was to flush the system with dry nitrogen gas at a pressure of 300 to 500 microns Hg for 16 to 24 hours.

(c) The trap was then filled with liquid nitrogen (only the lower trap was filled on the 10^{-10} system at this time), the nitrogen gas bleed was shut off, the diffusion pump was turned on (both diffusion pumps on the 10^{-10} system).

(d) The system was then pumped to the 10^{-6} torr level and leak checked.

(e) Next, the bake-out furnace was dropped over the apparatus and the system was baked out at between 300 and 350°C for 16 to 24 hours; a 48-hour bake-out was sometimes used to lower background pressure.

(f) The furnace was removed after cooling to below 100°C, and the ion gages were hooked up. The specimen furnace was turned on and brought up to temperature (885 to 1100° C).

(g) The ion gages were then degassed. After this step the 10^{-10} system often needed a second bake-out in order to reach the 10^{-10} torr range.

(h) The mass spectrometer was turned on and adjusted.

(i) The top trap on the 10^{-10} system was filled with liquid nitrogen.

(j) The system was then run until the ion gages reached the low 10^{-8} torr range for the 10^{-8} system, or the mid 10^{-10} torr range for the 10^{-10} system.

(k) The specimen was then pushed into its final position at the center of the specimen furnace, by means of magnets acting against an iron piece located at the end of the sleigh. This step was not necessary for the induction heated exposures, as the specimen was located properly within the induction heater coils initially, and did not have to be moved. Pushing the specimen into the furnace produced a burst of gas, raising the pressure suddenly.

(l) Pumping continued generally until the p_o pressure went back to nearly the same level as before step (k). This sometimes required two to three days of pumping. In the later exposures (beginning with the 11th exposure) the specimen temperature was raised to 25 to 50° C above the actual exposure temperature for at least 30 minutes in order to outgas the specimen before introducing oxygen. The system was then ready for introduction of oxygen gas through the silver leak.

4-2. Conducting Exposure

Oxygen pressure was regulated by controlling the temperature of the silver leak, and to some extent on the 10^{-10} system by adjusting the opening of the valve located between the traps and the glass system. Prior to admitting oxygen, the silver leak was outgassed by pumping a vacuum on the upstream side and heating. Oxygen was then admitted to the upstream side of the leak (at time = 0 for the exposure) and maintained at 2 to 3 psi above atmospheric for the remainder of the exposure. After admitting oxygen (at time = 0) it generally took about 15 minutes to reach the correct pressure decade and another 15 to 30 minutes to level off at the final pressure. No measurement was taken of the silver leak temperature.

All ion gage pressures were recorded continuously on strip chart recorders. Mass spectrometer readings were taken daily and ion gage readings were manually recorded daily, near the end of each step in a "step" exposure. Optical pyrometer readings were checked several times each day, and temperature adjustments made when necessary.

At the end of an exposure, the glass system was allowed to cool down to ambient and then the pressure was brought up to atmospheric. The glass system was then opened up, the specimen removed, and the specimen sent to the NASA Lewis Research Center for analysis. The strip-chart recorder rolls were also sent to NASA for use in reducing the data.

5. DATA REDUCTION

As mentioned in the Introduction, the principal data reduction was accomplished by NASA at the Lewis Research Center. The only data reduction performed by General Electric was the determination of corrected pressures from the ion gage calibration curves, and from those pressures, the calculation of gettering rates, expressed in terms of both Q (torr liters/sec) and $\dot{m}$ (micrograms/hour).

The equations used are developed as follows. The equation for determining Q is the well-known relationship:

$$Q_o = C_o \ (p_o - p_1)$$

where the conductance C_o can be expressed as a function of the gas molecular weight, the temperature of the conductance, and a constant B which depends upon the geometry of the conductance.

$$C_o = B(T_o/M)^{\frac{1}{2}}$$

Values for B for the specific conductances used are determined in the Appendix, Section 9-1.

To determine the mass gettering rate, the ideal gas law is first written:

$$p\dot{V}_o = \frac{\dot{m}RT_o}{M}$$

But since Q_o is defined as $Q_o = p\dot{V}_o$, the above equation can be written:

$$\dot{m} = \frac{MQ_o}{RT_o}$$

To express $\dot{m}$ in the units micrograms/hour (instead of grams/sec) the above value for $\dot{m}$ is multiplied by the factor 3600 x 10^6.

6. EXPERIMENTAL RESULTS FROM EXPOSURES

6-1. General

Of the 15 exposures conducted during the program, there were two types: (1) a "steady state" exposure in which the pressure and temperature of the specimen were held constant for a 236-hour period (approximately 10 days), and (2) a "step" exposure in which the pressure and temperature were held constant for 16 to 24 hours or more, after which they were changed to new values for the next step or phase of the exposure. The "step" exposures generally consisted of nine steps or phases.

The reporting of data for the two types of exposures is different. In the steady state exposures (represented by Exposure Nos. 1, 2, and 9), gas compositions are given daily during the exposure, and uncorrected pressure readings -- taken from the recorder rolls -- are plotted each 6 hours during the exposure. A single average gettering rate, based upon a corrected average pressure during the exposure, is given.

For the step exposures, uncorrected pressure readings are reported in the form of a daily log, with readings taken from the control panel indication, at the end of each phase, at which time a steady-state should have been reached. Gas compositions are also given in a table for each phase, near the end of the phase. Another table reports corrected pressures and gettering rates based upon those pressures, for each phase.

6-2. Index to Exposure Data

As a summary of the specimens tested and as an aid in locating data, the following index is presented:

Exposure No.	Specimen No.	Composition	Vacuum System	Data found in Table No.	Notes
1	59	Cb-1Zr	10^{-8}	1-a,b; Fig. 6	(1)
2	60	Cb-1Zr	10^{-8}	2-a,b; Fig. 7	(1)
3	69	Cb-5Zr	10^{-10}	3-a, b, c, d	(2)
4	61	Cb-1Zr	10^{-8}	4-a, b, c, d	
5	55	Ta	10^{-10}	5-a, b, c, d	
6	57	Ta-10W	10^{-10}	6-a, b, c, d	
7	83	T-111	10^{-8}	7-a, b, c, d	
8	74	B-33	10^{-8}	8-a, b, c, d	
9	51	Cb	10^{-10}	9-a, b; Fig. 8	(1)
10	111	Zr	10^{-10}	10-a, b, c, d	(3)
11	90	Cb-1Zr	10^{-10}	11-a, b, c, d	(4)
12	88	Cb-.75Zr	10^{-8}	12-a, b, c, d	(5)
13	110	FS-85	10^{-10}	13-a, b, c, d	
14	99	SCb-291	10^{-10}	14-a, b, c, d	
15	94	Ta	10^{-8}	15-a, b, c, d	(5)

NOTE (1) "Steady state" exposure; conditions held constant during exposure. Other exposures were "step" exposures in which pressure or temperature levels were changed daily.

NOTE (2) Ion gage emission current increased from 0.1 to 1.0 prior to Exposure No. 3, and new calibration curves prepared.

NOTE (3) New gage calibration beginning with Exposure No. 10.

NOTE (4) Prior to Exposure No. 11, C.V.C. ion gage control panels and gage tubes switched from 10^{-8} to 10^{-10} system, and G.E. panels and tubes switched to 10^{-8} system.

NOTE (5) Specimen heated by induction heater rather than furnace.

6-3 Tabulated Results

Tabular results begin with the table below:

TABLE 1-a

GENERAL PARAMETERS, EXPOSURE NO. 1

Specimen Composition:	Cb-1Zr
Specimen Number:	59
Specimen Size:	0.0292 x 1.052 x 1.502 in.
Specimen Weight:	6.512 gram
Specimen Temperature:	1100 deg C
Pressure, "Sample Gage" (p_1):	1×10^{-6} torr (approx.)
Vacuum System:	10^{-8} system
Conductance Temperature (T_o):	324 deg K (51 deg C) (estimated)
System Conductance:	$C_o = 0.187\ (T_o/M)^{1/2}$ liters/sec (for O_2 at 324°K, $C_o = 0.595$)
Gage Calibrations:	Figures 20 - 23, Final Report, NAS 3-4169 (Jan. 1965)* (0.1 ma emission current)
Distance L:	30 cm. (estimated)

* Corrected pressures are given below.

Calculation of Gettering Rate

Estimated average pressures in torr are as follows:

	Uncorrected Pressure	Corrected Pressure
Pump Ion Gage (p_o)	6.6×10^{-6}	8.7×10^{-6}
Sample Ion Gage (p_1)	1.1×10^{-6}	1.4×10^{-6}
Quartz Ion Gage (p_3)	1.2×10^{-7}	1.1×10^{-7}

$Q_o = C_o\ (p_o - p_1) = 4.34 \times 10^{-6}$ torr-liter/sec

$\dot{m} = (MQ_o/RT_o) \times 3600 \times 10^6 = 24.8$ microgram/hr

TABLE I-b

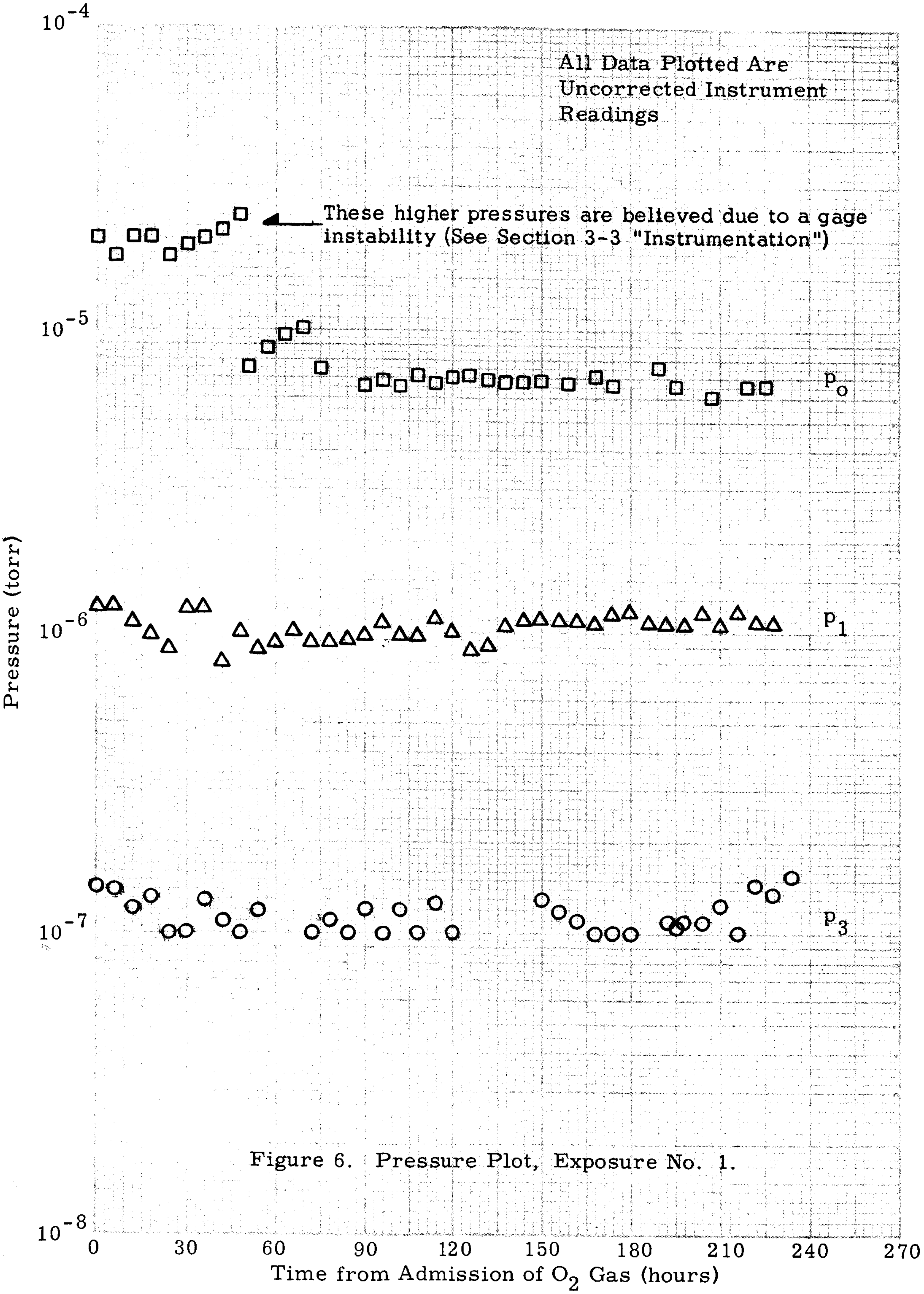
GAS COMPOSITION DURING THE EXPOSURE OF

Cb-1Zr SPECIMEN NO. 59

EXPOSURE NO. 1

	Gas Composition, Molar Percent					
Time,Hrs.*	CO_2	O_2	CO	H_2O	He	H_2
23	5.6	81.9	3.1	3.0	0.6	6.8
46	4.6	85.8	1.7	2.4	0.6	4.9
66	4.6	86.2	1.3	2.3	0.6	5.0
93	4.5	87.1	1.2	2.2	0.8	4.2
143	4.4	91.7	0.9	2.0	0.74	3.0
168	4.6	89.5	1.0	1.7	0.6	2.6
192	4.3	89.6	1.1	2.0	0.9	2.11
214	4.4	89.2	1.4	2.0	1.0	2.0
232	5.1	88.4	2.1	1.7	1.0	1.7

* From start of O_2 admission.



All Data Plotted Are Uncorrected Instrument Readings

These higher pressures are believed due to a gage instability (See Section 3-3 "Instrumentation")

Figure 6. Pressure Plot, Exposure No. 1.

TABLE 2-a

GENERAL PARAMETERS, EXPOSURE NO. 2

Specimen Composition:	Cb-1Zr
Specimen Number:	60
Specimen Size:	0.029 x 1.052 x 1.502 in.
Specimen Weight:	6.469 gram
Specimen Temperature:	980 deg C
Pressure, "Sample Gage" (p_1):	1×10^{-7} (approx.)
Vacuum System:	10^{-8} system
Conductance Temperature (T_o):	324 deg K (51 deg C) (estimated)
System Conductance:	$C_o = 0.203\ (T_o/M)^{1/2}$ liters/sec (for O_2 at 324°K, $C_o = 0.646$)
Gage Calibrations:	Figures 20 - 23, Final Report, NAS 3-4169 (Jan. 1965)* (0.1 ma emission current)
Distance L:	30 cm. (estimated)

* Corrected pressures are given below.

Calculation of Gettering Rate

Estimated average pressures in torr are as follows:

	Uncorrected Pressure	Corrected Pressure
Pump Ion Gage (p_o)	1.5×10^{-6}	1.69×10^{-6}
Sample Ion Gage (p_1)	1.4×10^{-7}	1.51×10^{-7}
Quartz Ion Gage (p_3)	5.8×10^{-8}	5.2×10^{-8}

$Q_o = C_o\ (p_o - p_1) = 9.94 \times 10^{-7}$ torr-liter/sec

$\dot{m} = (MQ_o/RT_o) \times 3600 \times 10^{6} = 5.66$ microgram/hr

TABLE 2-b

GAS COMPOSITION DURING THE EXPOSURE OF

Cb-1Zr SPECIMEN NO. 60

EXPOSURE NO. 2

	Gas Composition, Molar Percent					
Time,Hrs.*	CO_2	O_2	CO	H_2O	He	H_2
24	22.9	46.5	18.6	1.8	3.4	6.8
45	18.7	49.6	16.0	1.4	7.9	6.4
71	14.5	55.8	13.0	1.5	9.5	5.7
92	12.8	61.5	11.5	1.4	8.7	4.1
122	14.5	57.3	11.4	1.8	8.0	7.0
146	16.6	53.6	10.9	1.9	8.8	8.2
169	15.7	56.0	12.0	1.7	8.7	5.9
193	14.0	57.7	10.8	1.7	10.5	5.3
234	13.9	60.2	9.8	1.4	11.1	3.6

* From start of O_2 admission.

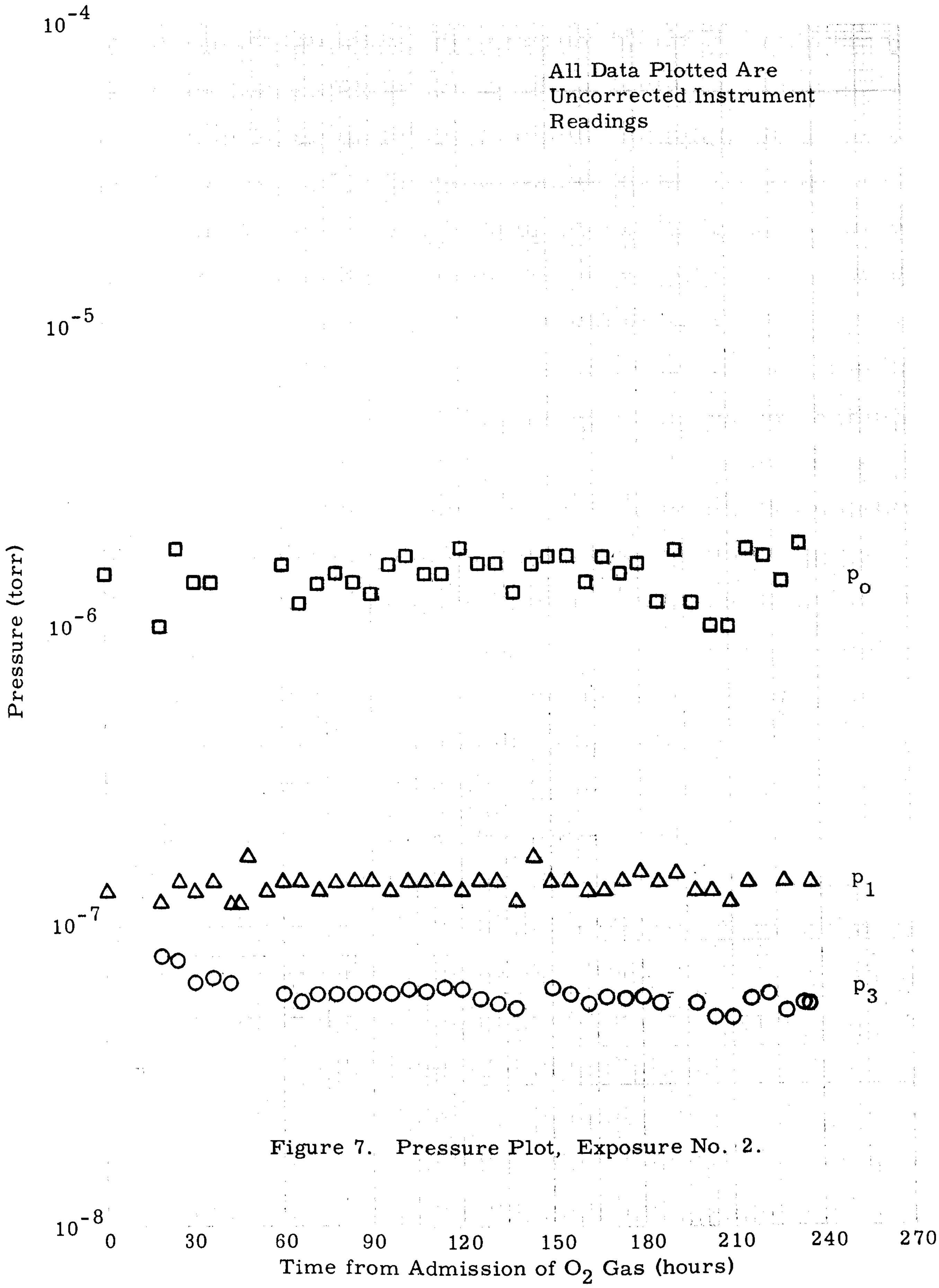


Figure 7. Pressure Plot, Exposure No. 2.

TABLE 3-a

GENERAL PARAMETERS, EXPOSURE NO. 3

Specimen Composition:	Cb-5Zr
Specimen Number:	69
Specimen Size:	0.028 x 1.052 x 1.5035 in.
Specimen Weight:	6.153 gram
Specimen Temperature:	885, 980, & 1100 deg C
Pressure, "Sample Gage" (p_1):	1×10^{-7} to 1×10^{-5} torr (approx.) (in steps)
Vacuum System:	10^{-10} system
Conductance Temperature (T_o):	313 deg K (40 deg C) (estimated)
System Conductance:	$C_o = 0.172 \, (T_o/M)^{1/2}$ liters/sec (for O_2 at $313^{o}K$, $C_o = 0.538$)
Gage Calibrations:	First calibration, G.E. panels, Figures 13, 14 and 15.
Distance L:	35 cm.(estimated)

TABLE 3-b

DAILY LOG OF PRESSURE MEASUREMENTS

EXPOSURE NO. 3

Cb-5Zr Specimen No. 69

Pressures given are uncorrected ion gage readings.

7-22-65	2:17 PM	Oxygen introduced to mid 10^{-9} torr system - Exposure 10^{-7} torr O_2 - 885°C	
7-23-65	2:00 PM	Pump ig	2.0×10^{-6} torr
		Sample ig	1.6×10^{-7} torr
		Quartz ig	3.2×10^{-8} torr
	3:55 PM	Specimen temp increased to 980°C - 10^{-7} torr O_2	
7-24-65 (Sat.)	1:30 PM	Pump ig	2.1×10^{-6} torr
		Sample ig	1.9×10^{-7} torr
		Quartz ig	5.5×10^{-8} torr
7-25-65 (Sunday)	9:00 AM	Pump ig	2.0×10^{-6} torr
		Sample ig	1.6×10^{-7} torr
		Quartz ig	5.0×10^{-8} torr
	9:10 AM	Specimen temp increased to 1100°C - 10^{-7} torr O_2	
7-26-65	9:25 AM	Pump ig	2.0×10^{-6} torr
		Sample ig	1.9×10^{-7} torr
		Quartz ig	6.8×10^{-8} torr
	10:15 AM	Oxygen increased to 10^{-6} torr O_2 - 1100°C	
7-27-65	10:50 AM	Pump ig	1.4×10^{-5} torr
		Sample ig	1.0×10^{-6} torr
		Quartz ig	1.55×10^{-7} torr
	10:55 AM	Specimen temp decreased to 980°C - 10^{-6} torr O_2	
7-28-65	11:00 AM	Pump ig	1.5×10^{-5} torr
		Sample ig	1.0×10^{-6} torr
		Quartz ig	1.5×10^{-7} torr
	11:30 AM	Specimen temp decreased to 885°C - 10^{-6} torr O_2	

TABLE 3-b(cont'd.)

7-29-65	9:00 AM	Pump ig	1.4×10^{-5} torr
		Sample ig	1.0×10^{-6} torr
		Quartz ig	1.7×10^{-7} torr
	10:00 AM	Specimen temp increased to 1100°C	
	11:00 AM	Oxygen increased to 10^{-5} torr O_2 - 1100°C	
7-30-65	5:00 PM	Pump ig	1.6×10^{-4} torr
		Sample ig	1.1×10^{-5} torr
		Quartz ig	1.3×10^{-6} torr
7-31-65 (Sat.)	8:51 AM	Pump ig	1.6×10^{-4} torr
		Sample ig	1.1×10^{-5} torr
		Quartz ig	1.1×10^{-6} torr
	8:55 AM	Specimen temp decreased to 980°C - 10^{-5} torr O_2	
8-1-65 (Sunday)	8:30 AM	Pump ig	1.55×10^{-4} torr
		Sample ig	1.05×10^{-5} torr
		Quartz ig	1.3×10^{-6} torr
	9:00 AM	Specimen temp decreased to 885°C - 10^{-5} torr O_2	
8-2-65	246 hrs.	Pump ig	1.5×10^{-4} torr
		Sample ig	1.15×10^{-5} torr
		Quartz ig	1.9×10^{-6} torr

At approximately 250 hrs., all ion gages started full scale oscillations. The silver permeation membrane was intermittently leaking and on the verge of opening up. This can be attributed to running at the extreme end of the leaks rated current, to obtain 10^{-5} torr 0_2. (The test ended 8:10 AM, August 2.)

TABLE 3-c

GAS COMPOSITION DURING THE EXPOSURE OF

Cb-5Zr SPECIMEN NO. 69

EXPOSURE NO. 3

		Gas Composition, Molar Percent					
Exposure	**Time,Hrs***	**CO_2**	**O_2**	**CO**	**H_2O**	**He**	**H_2**
1.6×10^{-7}Torr-O_2-885°C	24	6.1	61.0	26.5	0.20	3.0	3.2
1.6×10^{-7}Torr-O_2-980°C	64	8.1	50.2	17.0	3.0	6.2	15.5
1.9×10^{-7}Torr-O_2-1100°C	89	8.5	40.0	13.4	2.0	11.7	24.4
1.0×10^{-6}Torr-O_2-1100°C	114	3.5	84.6	8.0	0.60	0.40	2.9
1.0×10^{-6}Torr-O_2-980°C	139	3.7	89.5	6.0	0.10	0.30	0.40
1.0×10^{-6}Torr-O_2-885°C	161	2.8	93.1	3.6	0	0.20	0.30
1.1×10^{-5}Torr-O_2-1100°C	195	1.7	95.9	1.8	0.2	0	0.4
1.05×10^{-5}Torr-O_2-980°C	236	2.4	92.8	4.8	0	0	0

* From start of O_2 admission.

TABLE 3-d

GETTERING RATES

EXPOSURE No. 3

Cb-5Zr Specimen No. 69

Pressures given were taken at the end of each phase, and are corrected below by use of calibration data. Pressures are in torr.

					Gettering Rates		
Phase	Specimen Temp (°C)	Pump Gage, p_o	Sample Gage, p_1	Quartz Gage, p_3	Q_o torr L/sec	m, micro-grams/hr	Approx. Exposure Time, Hrs.
1	885	3.1(-6)	2.45(-7)	4.8(-8)	1.54(-6)	9.06	24
2	980	3.1(-6)	2.45(-7)	7.4(-8)	1.54(-6)	9.06	41
3	1100	3.1(-6)	2.9 (-7)	10.0(-8)	1.51(-6)	8.92	24
4	1100	1.95(-5)	1.5 (-6)	2.35(-7)	9.68(-6)	57.1	25
5	980	2.1 (-5)	1.5 (-6)	2.3(-7)	1.05(-5)	61.9	24
6	885	1.95(-5)	1.5 (-6)	2.6(-7)	9.68(-6)	57.1	22
7	1100	2.05(-4)	1.52(-5)	1.65(-6)	1.02(-4)	602	46
8	980	2.0 (-4)	1.45(-5)	1.98(-6)	9.98(-5)	589	24
9	885	1.92(-4)	1.6 (-5)	2.92(-6)	9.47(-5)	558	23

NOTE: Figures in parenthesis are powers of 10; 1(-4) = 1 x 10^{-4}

TABLE 4-a

GENERAL PARAMETERS, EXPOSURE NO. 4

Specimen Composition:	Cb-1Zr
Specimen Number:	61
Specimen Size:	0.0295 x 1.052 x 1.503 in.
Specimen Weight:	6.557 gram
Specimen Temperature:	885, 980 and 1100 deg C
Pressure, "Sample Gage" (p_1):	1 x 10^{-7} to 1 x 10^{-5} torr (approx.) (in steps)
Vacuum System:	10^{-8} system
Conductance Temperature (T_o)	324 deg K (51 deg C) (estimated)
System Conductance:	$C_o = 0.203 (T_o/M)^{1/2}$ liter/sec (for O_2 at 324°K, $C_o = 0.646$)
Gage Calibrations:	First calibration, C.V.C. panels, Figures 10, 11 and 12
Distance L:	30 cm. (estimated)

TABLE 4-b

DAILY LOG OF PRESSURE MEASUREMENTS

EXPOSURE NO. 4

Cb-1Zr Specimen No. 61

Pressures given are uncorrected ion gage readings.

7-28-65		(All ion gages in low 10^{-8} torr) sample introduced to 885°C furnace 7-26-65	
	2:29 PM	O_2 introduced to system exposure 10^{-6} torr O_2 -885°C	
7-29-65	8:52 AM	Pump ig	1.8×10^{-5} torr
		Sample ig	1.2×10^{-6} torr
		Quartz ig	2.8×10^{-7} torr
	10:00 AM	Specimen temp increased to 980°C - 10^{-6} torr O_2	
7-30-65	8:30 AM	Pump ig	2.05×10^{-5} torr
		Sample ig	1.3×10^{-6} torr
		Quartz ig	2.4×10^{-7} torr
	11:00 AM	Specimen temp increased to 1100°C - 10^{-6} torr O_2	
7-31-65 (Sat.)	9:01 AM	Pump ig	2.05×10^{-5} torr
		Sample ig	1.4×10^{-6} torr
		Quartz ig	2.6×10^{-7} torr
	9:05 AM	Specimen temp decreased to 885°C	
	9:50 AM	Oxygen cut back to 10^{-7} torr O_2	
8-1-65 (Sunday)	8:14 AM	Pump ig	1.5×10^{-6} torr
		Sample ig	1.2×10^{-7} torr
		Quartz ig	4.8×10^{-8} torr
8-2-65	9:50 AM	Pump ig	1.95×10^{-6} torr
		Sample ig	1.4×10^{-7} torr
		Quartz ig	4.2×10^{-8} torr
	9:50 AM	Specimen temp increased to 980°C 10^{-7} torr O_2	

TABLE 4-b(cont'd.)

8-3-65	8:25 AM	Pump ig	1.8×10^{-6} torr
		Sample ig	1.3×10^{-7} torr
		Quartz ig	4.6×10^{-8} torr
	10:15 AM	Specimen temp increased to 1100°C - 10^{-7} torr 0_2	
8-4-65	8:20 AM	Pump ig	1.8×10^{-6} torr
		Sample ig	1.9×10^{-7} torr
		Quartz ig	9.0×10^{-8} torr
	10:28 AM	Oxygen increased to 10^{-5} torr 0_2 1100°C	
8-5-65	10:00 AM	Pump ig	2.9×10^{-4} torr
		Sample ig	1.6×10^{-5} torr
		Quartz ig	2.35×10^{-6} torr
	10:13 AM	Specimen temp decreased to 980°C - 10^{-5} torr 0_2	
8-6-65	9:20 AM	Pump ig	2.65×10^{-4} torr
		Sample ig	1.55×10^{-5} torr
		Quartz ig	3.5×10^{-6} torr
	9:23 AM	Specimen temp decreased to 885°C - 10^{-5} torr 0_2	
8-7-65	8:34 AM	Pump ig	3.0×10^{-4} torr
		Sample ig	1.7×10^{-5} torr
		Quartz ig	4.0×10^{-6} torr
	9:19 AM	0_2 off - specimen heat off	

TABLE 4-c

GAS COMPOSITION DURING THE EXPOSURE OF

Cb-1Zr SPECIMEN NO. 61

EXPOSURE NO. 4

Exposure	Time, Hrs*	Gas Composition, Molar Percent					
		CO_2	O_2	CO	H_2O	He	H_2
1.2 x 10^{-6}Torr-O_2-885°C	18	3.0	92.1	4.2	1.0	0.3	0.4
1.4 x 10^{-6}Torr-O_2-1100°C	64	3.9	86.4	8.0	0.60	0.5	0.6
1.4 x 10^{-7}Torr-O_2-885°C	84	7.0	62.5	25.2	0.3	2.0	3.0

Trouble was experienced with the mass spectrometer filament and meaningful spectrums could not be obtained after 84 hours.

* From start of O_2 admission.

TABLE 4-d

GETTERING RATES

EXPOSURE NO.4

Cb-1Zr Specimen No. 61

Pressures given were taken at the end of each phase, and are corrected below by use of calibration data. Pressures are in torr.

					Gettering Rates		
Phase	Specimen Temp (°C)	Pump Gage, p_o	Sample Gage, p_1	Quartz Gage, p_3	Q_o torr L/sec	$\dot{m}$, micrograms/hr	Approx. Exposure Time, Hrs.
1	885	0.84(-5)	0.77(-6)	2.25(-7)	4.93(-6)	28.1	19
2	980	0.97(-5)	0.85(-6)	1.95(-7)	5.72(-6)	32.6	22
3	1100	0.97(-5)	0.92(-6)	2.1 (-7)	5.67(-6)	32.3	22
4	885	1.15(-6)	1.0 (-7)	3.7 (-8)	6.78(-7)	3.86	48
5	980	1.05(-6)	0.95(-7)	3.95(-8)	6.17(-7)	3.51	23
6	1100	1.05(-6)	1.3 (-7)	6.9 (-8)	5.94(-7)	3.39	24
7	1100	2.9 (-4)*	0.91(-5)	1.9 (-6)	--	--	24
8	980	2.65(-4)*	0.90(-5)	2.95(-6)	--	--	23
9	885	3.0 (-4)*	1.0 (-5)	3.45(-6)	--	--	24

* Uncorrected instrument reading (no calibration available in this range)

NOTE: Figures in parenthesis are powers of 10; 1(-4) = 1 x 10^{-4}

TABLE 5-a

GENERAL PARAMETERS, EXPOSURE No. 5

Specimen Composition:	Ta (unalloyed)
Specimen Number:	55
Specimen Size:	0.031 x 0.807 x 1.7525 in.
Specimen Weight:	12.128 gram
Specimen Temperature:	885, 980, and 1100 deg C
Pressure, "Sample Gage" (p_1):	1 x 10^{-7} to 1 x 10^{-5} torr (approx.) (in steps)
Vacuum System:	10^{-10} system
Conductance Temperature (T_o):	313 deg K (40 deg C) (estimated)
System Conductance:	$C_o = 0.172\ (T_o/M)^{1/2}$ liter/sec (for O_2 at 313°K, $C_o = 0.538$)
Gage Calibrations:	First calibration, G. E. panels, Figures 13, 14 and 15
Distance L:	35 cm. (estimated)

TABLE 5-b

DAILY LOG OF PRESSURE MEASUREMENTS

EXPOSURE NO. 5

Ta Specimen No. 55

Pressures given are uncorrected ion gage readings.

8-17-65	12:50 PM	Oxygen introduced to high 10^{-9} torr vacuum - Exposure 10^{-7} torr O_2 - 885°C	
8-19-65	8:30 AM	Pump ig	1.4×10^{-6} torr
		Sample ig	1.7×10^{-7} torr
		Quartz ig	6.0×10^{-8} torr
	10:15 AM	Specimen temp increased to 980°C - 10^{-7} torr O_2	
8-20-65	8:30 AM	Pump ig	1.3×10^{-6} torr
		Sample ig	1.8×10^{-7} torr
		Quartz ig	7.5×10^{-8} torr
	9:40 AM	Specimen temp increased to 1100°C - 10^{-7} torr O_2	
8-21-65 (Sat.)	9:15 AM	Pump ig	7.2×10^{-7} torr
		Sample ig	1.3×10^{-7} torr
		Quartz ig	5.6×10^{-8} torr
	10:10 AM	Specimen temp decreased to 885°C - Oxygen increased to 10^{-6} torr O_2	
8-22-65 (Sun.)	9:10 AM	Pump ig	1.1×10^{-5} torr
		Sample ig	1.3×10^{-6} torr
		Quartz ig	5.5×10^{-7} torr
	10:50 AM	Specimen temp increased to 980°C -10^{-6} torr O_2	
8-23-65	8:10 AM	Pump ig	1.1×10^{-5} torr
		Sample ig	1.4×10^{-6} torr
		Quartz ig	5.4×10^{-7} torr
	9:15 AM	Specimen temp increased to 1100°C - 10^{-6} torr O_2	
8-24-65	8:10 AM	Pump ig	1.3×10^{-5} torr
		Sample ig	1.6×10^{-6} torr
		Quartz ig	6.0×10^{-7} torr
	9:30 AM	Oxygen increased to 10^{-5} torr O_2 - 1100°C	

TABLE 5-b(cont'd.)

8-25-65	8:05 AM	Pump ig	1.0 x 10^{-4} torr
		Sample ig	1.2 x 10^{-5} torr
		Quartz ig	4.8 x 10^{-6} torr
	9:25 AM	Specimen temp decreased to 980°C - 10^{-5} torr O_2	
8-26-65	8:05 AM	Pump ig	1.0 x 10^{-4} torr
		Sample ig	1.2 x 10^{-5} torr
		Quartz ig	4.6 x 10^{-6} torr
	9:00 AM	Specimen temp decreased to 885°C - 10^{-5} torr O_2	
8-27-65	8:00 AM	Pump ig	1.0 x 10^{-4} torr
		Sample ig	1.4 x 10^{-5} torr
		Quartz ig	6.8 x 10^{-6} torr
	9:35 AM	= O_2 off = Sample furnace off.	

TABLE 5-c

GAS COMPOSITION DURING THE EXPOSURE OF

Ta SPECIMEN NO. 55

EXPOSURE NO. 5

		Gas Composition, Molar Percent					
Exposure	**Time, Hrs.***	CO_2	O_2	CO	H_2O	He	H_2
1.7×10^{-7} Torr-O_2-885°C	43	5.5	81.7	6.5	0.30	1.7	4.3
1.9×10^{-7} Torr-O_2-980°C	64	5.2	76.2	8.8	0.50	7.3	2.0
1.3×10^{-7} Torr-O_2-1100°C	90	6.3	52.7	13.6	1.0	2.9	23.5
1.3×10^{-6} Torr-O_2-885°C	113	4.8	91.7	3.0	0.1	0.10	0.3
1.4×10^{-6} Torr-O_2-980°C	134	7.2	88.3	4.2	0.1	0.2	0
1.6×10^{-6} Torr-O_2-1100°C	157	8.0	87.3	4.2	0.20	0.3	0
1.2×10^{-5} Torr-O_2-1100°C	179	12.0	84.8	3.0	0.1	0.1	0
1.3×10^{-5} Torr-O_2-980°C	211	7.9	90.1	2.0	0	0	0
1.3×10^{-5} Torr-O_2-885°C	235	2.5	96.5	1.0	0	0	0

* From start of O_2 admission.

TABLE 5-d

GETTERING RATES

EXPOSURE NO. 5

Ta Specimen No. 55

Pressures given were taken at the end of each phase, and are corrected below by use of calibration data. Pressures are in torr.

Phase	Specimen Temp (°C)	Pump Gage, p_o	Sample Gage, p_1	Quartz Gage, p_3	Gettering Rates Q_o torr L/sec	 m, micro-grams/hr	Approx. Exposure Time, Hrs.
1	885	2.13(-6)	2.6 (-7)	8.85(-8)	1.01 (-6)	5.93	44
2	980	1.98(-6)	2.7 (-7)	11.0 (-8)	9.17 (-7)	5.41	23.5
3	1100	10.0 (-7)	1.98(-7)	8.3 (-8)	4.31 (-7)	2.54	24
4	885	1.52(-5)	1.98(-6)	8.1 (-7)	7.11 (-6)	41.9	23
5	980	1.52(-5)	2.13(-6)	8.0 (-7)	7.03 (-6)	41.5	21
6	1100	1.8 (-5)	2.45(-6)	8.75(-7)	8.36 (-6)	49.3	23
7	1100	1.25(-4)	1.67(-5)	7.65(-6)	5.83 (-5)	344.	22.5
8	980	1.25(-4)	1.67(-5)	7.3 (-6)	5.83 (-5)	344.	23
9	885	1.25(-4)	1.95(-5)	11.0 (-6)	5.68 (-5)	335.	23

NOTE: Figures in parenthesis are powers of 10; $1(-4) = 1 \times 10^{-4}$

TABLE 6-a

GENERAL PARAMETERS, EXPOSURE NO. 6

Specimen Composition:	Ta-10 W
Specimen Number:	57
Specimen Size:	0.0315 x 0.811 x 1.750 in.
Specimen Weight:	12.270 gram
Specimen Temperature:	885, 980, and 1100 deg C
Pressure, "Sample Gage" (p_1):	5×10^{-8} to 1×10^{-5} torr (approx.) (in steps)
Vacuum System:	10^{-10} system
Conductance Temperature (T_o):	313 deg K (40 deg C) (estimated)
System Conductance:	$C_o = 0.172 \, (T_o/M)^{1/2}$ liter/sec (for O_2 at 313°K, $C_o = 0.538$)
Gage Calibrations:	First calibration, G. E. panels, Figures 13, 14 and 15
Distance L:	35 cm. (estimated)

TABLE 6-b

DAILY LOG OF PRESSURE MEASUREMENTS

EXPOSURE NO. 6

Ta-10W Specimen No. 57

Pressures given are uncorrected ion gage readings.

9-23-65	3:22 PM	Oxygen introduced to mid 10^{-9} torr vacuum - Exposure mid 10^{-8} torr O_2 - 980° C	
9-24-65	8:30 AM	Pump ig	2.3×10^{-7} torr
		Sample ig	4.6×10^{-8} torr
		Quartz ig	2.3×10^{-8} torr
	9:37 AM	Specimen temp decreased to 885° C - mid 10^{-8} torr O_2	
9-25-65 Sat.		Trouble was experienced with a cycling pressure, the sample ig going as high as 10^{-7}. Adjusted specimen temp to 980°C at 11:18 AM	
9-26-65 Sun.	8:20 AM	Pump ig	1.0×10^{-7} torr
		Sample ig	2.9×10^{-8} torr
		Quartz ig	1.7×10^{-8} torr
	8:59 AM	Specimen temp decreased to 885°C - mid 10^{-8} torr O_2	
9-27-65	9:30 AM	Pump ig	9.7×10^{-8} torr
		Sample ig	2.6×10^{-8} torr
		Quartz ig	1.4×10^{-8} torr
	10:15 AM	Oxygen increased to 10^{-7} torr O_2 - 885° C	
9-28-65	9:12 AM	Pump ig	6.6×10^{-7} torr
		Sample ig	1.4×10^{-7} torr
		Quartz ig	7.5×10^{-8} torr
	9:14 AM	Specimen temp increased to 980° C - 10^{-7} torr O_2	
9-29-65	9:43 AM	Pump ig	7.7×10^{-7} torr
		Sample ig	1.6×10^{-7} torr
		Quartz ig	9.2×10^{-8} torr
	10:10 AM	Specimen temp increased to 1100° C - 10^{-7} torr O_2	
9-30-65	9:26 AM	Pump ig	7.6×10^{-7} torr
		Sample ig	1.5×10^{-7} torr
		Quartz ig	$.9 \times 10^{-7}$ torr
	9:50 AM	Oxygen increased to 10^{-6} torr O_2 - 1100° C	

TABLE 6-b(cont'd.)

10-1-65	8:22 AM	Pump ig	1.1 x 10^{-5} torr
		Sample ig	1.7 x 10^{-6} torr
		Quartz ig	8.2 x 10^{-7} torr
	9:50 AM	Specimen temp decreased to 980° C - 10^{-6} torr O_2	
10-2-65	12:00 PM	Pump ig	1 x 10^{-5} torr
		Sample ig	1.5 x 10^{-6} torr
		Quartz ig	7.1 x 10^{-7} torr
	12:35 PM	Specimen temp decreased to 885° C - 10^{-6} torr O_2	
10-4-65	10:00 AM	Pump ig	1.1 x 10^{-5} torr
		Sample ig	1.7 x 10^{-6} torr
		Quartz ig	8 x 10^{-7} torr
	10:15 AM	Oxygen increased to 10^{-5} torr O_2 - 885° C	
10-5-65	11:00 AM	Pump ig	1.1 x 10^{-4} torr
		Sample ig	2.4 x 10^{-5} torr
		Quartz ig	1.5 x 10^{-5} torr
	1:25 PM	Oxygen off - Specimen heat off	

TABLE 6-c

GAS COMPOSITION DURING THE EXPOSURE OF

Ta-10W SPECIMEN NO. 57

EXPOSURE NO. 6

		Gas Composition, Molar Percent					
Exposure	**Time, Hrs.***	**CO_2**	**O_2**	**CO**	**H_2O**	**He**	**H_2**
4.6×10^{-8} Torr-O_2-980°C	18	4.5	71.2	20.2	0	0	4.1
2.7×10^{-8} Torr-O_2-885°C	90	0	76.5	16.5	0	0	7.0
1.4×10^{-7} Torr-O_2-885°C	113	1.6	95.0	2.8	0	0	0.6
1.6×10^{-7} Torr-O_2-980°C	138	2.8	90.4	3.2	0.3	0	3.3
1.5×10^{-7} Torr-O_2-1100°C	162	5.2	75.1	6.2	1.0	0	12.5
1.7×10^{-6} Torr-O_2-1100°C	186	5.1	92.8	1.1	0.2	0	0.8
1.5×10^{-6} Torr-O_2-980°C	213	4.1	94.2	1.0	0.2	0	0.5
1.7×10^{-6} Torr-O_2-885°C	236	2.1	96.9	0.8	0.1	0	0.1
2.4×10^{-5} Torr-O_2-885°C	260	1.1	98.7	0.2	0	0	0

* From start of O_2 admission.

TABLE 6-d

GETTERING RATES

EXPOSURE NO. 6

Ta-10W Specimen No. 57

Pressures given were taken at the end of each phase, and are corrected below by use of calibration data. Pressures are in torr.

Phase	Specimen Temp (°C)	Pump Gage, p_o	Sample Gage, p_1	Quartz Gage, p_3	Gettering Rates		Approx. Exposure Time, Hrs.
					Q_0 torr L/sec	$\dot{m}$, micrograms/hr	
1	980	3.38(-7)	7.8 (-8)	3.5 (-8)	1.40(-7)	0.825	18
2	980	1.4 (-7)	5.2 (-8)	2.65(-8)	4.73(-8)	0.279	46
3	885	9.3 (-7)	2.14(-7)	1.1 (-7)	3.85(-7)	2.27	39
4	980	1.08(-6)	2.45(-7)	1.35(-7)	4.49(-7)	2.65	25
5	1100	1.06(-6)	2.3 (-7)	1.32(-7)	4.46(-7)	2.63	24
6	1100	1.52(-5)	2.61(-6)	1.17(-6)	6.77(-6)	39.9	24
7	980	1.4 (-5)	2.3(-6)	1.02(-6)	6.29(-6)	37.1	26
8	885	1.52(-5)	2.62(-6)	1.15(-6)	6.77(-6)	39.9	46
9	885	1.4 (-4)	3.4 (-5)	2.1 (-5)	5.70(-5)	336.	22

NOTE: Figures in parenthesis are powers of 10; 1(-4) = 1 x 10^{-4}

TABLE 7-a

GENERAL PARAMETERS, EXPOSURE NO. 7

Specimen Composition:	T-111
Specimen Number:	83
Specimen Size:	0.031 x 1.053 x 1.501 in.
Specimen Weight:	13.596 gram
Specimen Temperature:	885, 980 and 1100 deg C
Pressure, "Sample Gage" (p_1):	3×10^{-8} to 1×10^{-5} torr (approx.) (in steps)
Vacuum System:	10^{-8} system
Conductance Temperature (T_o):	324 deg K (51 deg C) (estimated)
System Conductance:	$C_o = 0.203 \, (T_o/M)^{1/2}$ liters/sec (for O_2 at 324°K, $C_o = 0.646$)
Gage Calibrations:	First calibration, C.V.C. panels, Figures 10, 11 and 12.
Distance L:	30 cm. (estimated)

TABLE 7-b

DAILY LOG OF PRESSURE MEASUREMENTS

EXPOSURE NO. 7

T-111 Specimen No. 83

Pressures given are uncorrected ion gage readings.

9-16-65	2:51 PM	Oxygen introduced to low 10^{-8} torr vacuum Exposure 10^{-7} torr O_2 - 885°C	
9-17-65	9:00 AM	Pump ig	1.25×10^{-6} torr
		Sample ig	1.35×10^{-7} torr
		Quartz ig	5.0×10^{-8} torr
	10:00 AM	Specimen temp increased to 980°C - 10^{-7} torr O_2	
9-18-65	9:00 AM	Pump ig	1.4×10^{-6} torr
		Sample ig	1.2×10^{-7} torr
		Quartz ig	4.1×10^{-8} torr
	9:50 AM	Specimen temp increased to 1100°C 10^{-7} torr O_2	
	11:00 AM	Experienced trouble with foreline of vacuum system - pumped out O_2 from silver leak (specimen temp decreased to 980°C)	
9-19-65 Sunday	7:00 AM	Pump ig	2.4×10^{-8} torr
		Sample ig	2.6×10^{-8} torr
		Quartz ig	1.6×10^{-8} torr
	7:27 AM	Oxygen introduced (Repeat) Exposure 10^{-7} torr O_2 - 980°C	
9-20-65	8:09 AM	Pump ig	1.8×10^{-6} torr
		Sample ig	1.5×10^{-7} torr
		Quartz ig	5.1×10^{-8} torr
	10:20 AM	Specimen temp increased to 1100°C - 10^{-7} torr O_2	
9-22-65	10:00 AM	Pump ig	1.8×10^{-6} torr
		Sample ig	1.8×10^{-7} torr
		Quartz ig	6.5×10^{-8} torr
	11:59 AM	Oxygen increased to 10^{-6} torr O_2 - 1100°C	
9-23-65	9:30 AM	Pump ig	2.0×10^{-5} torr
		Sample ig	1.5×10^{-6} torr
		Quartz ig	2.9×10^{-7} torr
	10:15 AM	Specimen temp decreased to 980°C - 10^{-6} torr O_2	

TABLE 7-b(cont'd.)

9-24-65	10:00 AM	Pump ig	2.15 x 10^{-5} torr
		Sample ig	1.9 x 10^{-6} torr
		Quartz ig	5.4 x 10^{-7} torr
	10:40 AM	Specimen temp decreased to 885°C - 10^{-6} torr O_2	
9-26-65 Sunday	7:30 AM	Pump ig	2.0 x 10^{-5} torr
		Sample ig	2.2 x 10^{-6} torr
		Quartz ig	1.0 x 10^{-6} torr
	8:00 AM	Specimen temp increased to 1100°C	
	8:22 AM	Oxygen increased to 10^{-5} torr O_2 - 1100°C	
9-28-65	9:30 AM	Pump ig	1.8 x 10^{-4} torr
		Sample ig	1.2 x 10^{-5} torr
		Quartz ig	3.8 x 10^{-6} torr
	10:10 AM	Specimen temp decreased to 980°C - 10^{-5} torr O_2	
9-29-65	8:10 AM	Pump ig	1.9 x 10^{-4} torr
		Sample ig	1.7 x 10^{-5} torr
		Quartz ig	6.9 x 10^{-6} torr
	8:41 AM	Specimen temp decreased to 885°C - 10^{-5} torr O_2	
9-30-65	8:45 AM	Pump ig	1.8 x 10^{-4} torr
		Sample ig	1.6 x 10^{-5} torr
		Quartz ig	7.3 x 10^{-6} torr
	10:00 AM	O_2 off - Specimen furnace off	

TABLE 7-c

GAS COMPOSITION DURING THE EXPOSURE OF

T-111 SPECIMEN NO. 83

EXPOSURE NO. 7

			Gas Composition, Molar Percent				
Exposure	**Time,Hrs.***	**CO_2**	**O_2**	**CO**	**H_2O**	**He**	**H_2**
1.3×10^{-7} torr-O_2-885°C	19	15.9	48.7	23.7	0.3	3.5	7.9
1.5×10^{-7} torr-O_2-980°C	92	25.0	48.7	6.8	2.0	2.7	14.8
1.5×10^{-6} torr-O_2-1100°C	163	6.1	88.3	2.8	1.8	0.4	0.6
1.9×10^{-6} torr-O_2-980°C	187	1.9	96.8	1.0	0.3	0	0
2×10^{-6} torr-O_2-885°C	233	3.9	95.0	0.8	0	0	0.3
1.2×10^{-5} torr-O_2-1100°C	283	1.3	97.9	0.4	0.2	0.2	0
1.7×10^{-5} torr-O_2-980°C	305	1.5	98.0	0.5	0	0	0

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* From start of O_2 admission.

TABLE 7-d

GETTERING RATES

EXPOSURE NO. 7

T-111 Specimen No. 83

Pressures given were taken at the end of each phase, and are corrected below by use of calibration data. Pressures are in torr.

Phase	Specimen Temp (°C)	Pump Gage, p_o	Sample Gage, p_1	Quartz Gage, p_3	Gettering Rates Q_o torr L/sec	Gettering Rates $\dot{m}$, micro-grams/hr	Approx. Exposure Time, Hrs.
1	885	0.71(-6)	0.96(-7)	4.22(-8)	3.97(-7)	2.26	18
2	980	1.06(-6)	1.06(-7)	4.3(-8)	6.16(-7)	3.51	73
3	1100	1.06(-6)	1.25(-7)	5.25(-8)	6.04(-7)	3.44	49
4	1100	0.94(-5)	0.98(-6)	2.33(-7)	5.44(-6)	31.0	23
5	980	1.02(-5)	1.26(-6)	4.2 (-7)	5.77(-6)	32.9	24
6	885	0.94(-5)	1.47(-6)	0.72(-6)	5.12(-6)	29.2	45
7	1100	1.8(-4)*	1.2(-5)	3.25(-6)	--	--	51
8	980	1.9(-4)*	1.7(-5)	6.35(-6)	--	--	22
9	885	1.8(-4)*	1.6(-5)	6.8(-6)	--	--	25

* Uncorrected instrument reading (no calibration available in this range)

NOTE: Figures in parenthesis are powers of 10; 1(-4) = 1 x 10^{-4}

TABLE 8-a

GENERAL PARAMETERS, EXPOSURE NO. 8

Specimen Composition:	B-33
Specimen Number:	74
Specimen Size:	0.032 x 1.051 x 1.503 in.
Specimen Weight:	6.908 gram
Specimen Temperature:	885, 980 and 1100 deg C
Pressure, "Sample Gage" (p_1):	1 x 10^{-7} to 1 x 10^{-5} torr (approx.) (in steps)
Vacuum System:	10^{-8} system
Conductance Temperature (T_o):	324 deg K (51 deg C)
System Conductance:	$C_o = 0.203 (T_o/M)^{1/2}$ liters/sec (for O_2 at 324° K, $C_o = 0.646$)
Gage Calibrations:	First calibration, C.V.C. panels, Figures 10, 11 and 12.
Distance L:	30 cm. (estimated)

TABLE 8-b

DAILY LOG OF PRESSURE MEASUREMENTS

EXPOSURE NO. 8

B-33 Specimen No. 74

Pressures given are uncorrected ion gage readings.

10-19-65	1:30 PM	Oxygen introduced to low 10^{-8} torr vacuum Exposure 10^{-7} torr - O_2 - 1100°C	
10-20-65	8:30 AM	Pump ig	1.8×10^{-6} torr
		Sample ig	1.7×10^{-7} torr
		Quartz ig	6.0×10^{-8} torr
	10:18 AM	Specimen temp decreased to 980°C - 10^{-7} torr - O_2	
10-21-65	9:00 AM	Pump ig	1.8×10^{-6} torr
		Sample ig	1.5×10^{-7} torr
		Quartz ig	4.8×10^{-8} torr
	10:31 AM	Specimen temp decreased to 885°C - 10^{-7} torr - O_2	
10-22-65	8:45 AM	Pump ig	1.7×10^{-6} torr
		Sample ig	1.3×10^{-7} torr
		Quartz ig	4.6×10^{-8} torr
	11:05 AM	Oxygen increased to 10^{-6} torr - O_2 - 885°C	
10-23-65 Sat.	7:58 AM	Pump ig	1.4×10^{-5} torr
		Sample ig	1.2×10^{-6} torr
		Quartz ig	3.3×10^{-7} torr
	11:21	Specimen temp increased to 980°C - 10^{-6} torr - O_2	
10-24-65 Sun.	8:36 AM	Pump ig	1.6×10^{-5} torr
		Sample ig	1.2×10^{-6} torr
		Quartz ig	2.95×10^{-7} torr
	8:47 AM	Specimen temp increased to 1100°C - 10^{-6} torr - O_2	
10-25-65	8:30 AM	Pump ig	1.3×10^{-5} torr
		Sample ig	1.2×10^{-6} torr
		Quartz ig	2.3×10^{-7} torr
	2:43 PM	Oxygen increased to 10^{-5} torr - O_2 - 1100°C	

TABLE 8-b(cont'd.)

10-26-65	8:20 AM	Pump ig	2.0×10^{-4} torr
		Sample ig	1.3×10^{-5} torr
		Quartz ig	3.0×10^{-6} torr
	2:44 PM	Specimen temp reduced to 980°C - 10^{-5} torr - O_2	
10-27-65	10:00 AM	Pump ig	2.0×10^{-4} torr
		Sample ig	1.3×10^{-5} torr
		Quartz ig	3.6×10^{-6} torr
	1:42 PM	Specimen temp reduced to 885°C	
10-28-65	9:30 AM	Pump ig	1.9×10^{-4} torr
		Sample ig	1.45×10^{-5} torr
		Quartz ig	4.8×10^{-6} torr
	11:45 AM	Oxygen off - Specimen heat off	

TABLE 8-c

GAS COMPOSITION DURING THE EXPOSURE OF

B-33 SPECIMEN NO. 74

EXPOSURE NO. 8

		Gas Composition, Molar Percent					
Exposure	**Time,Hrs.***	**CO_2**	**O_2**	**CO**	**H_2O**	**He**	**H_2**
1.7×10^{-7} torr-O_2-1100°C	20	7.0	45.5	15.8	0.5	2.8	28.4
1.5×10^{-7} torr-O_2-980°C	45	9.6	63.2	15.0	0.3	2.9	9.0
1.3×10^{-7} torr-O_2-885°C	69	7.1	72.3	11.7	0	3.0	5.9
1.2×10^{-6} torr-O_2-885°C	90	1.8	96.9	0.8	0	0.1	0.4
1.2×10^{-6} torr-O_2-980°C	115	1.7	97.3	0.6	0	0.2	0.2
1.2×10^{-6} torr-O_2-1100°C	142	2.5	95.2	1.0	0	0.3	1.0
1.3×10^{-5} torr-O_2-1100°C	164	0.8	98.9	0.3	0	0	0
1.3×10^{-5} torr-O_2-980°C	192	0.6	99.2	0.2	0	0	0
1.45×10^{-5} torr-O_2-885°C	213	0.5	99.3	0.2	0	0	0

* From start of O_2 admission.

TABLE 8-d

GETTERING RATES

EXPOSURE NO. 8

B-33 Specimen No. 74

Pressures given were taken at the end of each phase, and are corrected below by use of calibration data. Pressures are in torr.

					Gettering Rates		Approx.
Phase	Specimen Temp (°C)	Pump Gage, p_o	Sample Gage, p_1	Quartz Gage, p_3	Q_o torr L/ sec	$\dot{m}$, micro-grams/hr	Exposure Time, Hrs.
1	1100	1.07 (-6)	1.3 (-7)	4.9 (-8)	6.07 (-7)	3.46	21
2	980	1.07 (-6)	1.06 (-7)	4.1 (-8)	6.23 (-7)	3.55	24
3	885	1.0 (-6)	9.3 (-8)	3.95 (-8)	5.86 (-7)	3.34	24.5
4	885	6.4 (-6)	7.8 (-7)	2.65 (-7)	3.63 (-6)	20.7	24
5	980	7.4 (-6)	7.8 (-7)	2.37 (-7)	4.28 (-6)	24.4	21
6	1100	5.9 (-6)	7.8 (-7)	1.87 (-7)	3.31 (-6)	18.8	30
7	1100	2.0 (-4)*	1.3 (-5)	2.5 (-6)	--	--	24
8	980	2.0 (-4)*	1.3 (-5)	3.05 (-6)	--	--	23
9	885	1.9 (-4)*	1.45 (-5)	4.25 (-6)	--	--	22

* Uncorrected instrument reading (no calibration available in this range)

NOTE: Figures in parenthesis are powers of 10; 1(-4) = 1 x 10^{-4}

TABLE 9-a

GENERAL PARAMETERS, EXPOSURE NO. 9

Specimen Composition:	Cb (unalloyed)
Specimen Number:	51
Specimen Size:	0.030 x 0.807 x 1.755 in.
Specimen Weight:	5.585 gram
Specimen Temperature:	980 deg C.
Pressure, "Sample Gage", (p_1):	5×10^{-8} torr (approx.)
Vacuum System:	10^{-10} system
Conductance Temperature (T_o):	311 deg K (38 deg C)
System Conductance:	$C_o = 0.172\ (T_o/M)^{1/2}$ liters/sec (for O_2 at 311°K, $C_o = 0.536$ liters/sec)
Gage Calibrations:	First calibration, G. E. panels, Figures 13, 14 and 15*
Distance L:	31 cm.

* Corrected pressures are given below.

Calculation of Gettering Rate

Estimated average pressures in torr are as follows:

	Uncorrected Pressure	Corrected Pressure
Pump Ion Gage (p_o)	4.0×10^{-7}	5.8×10^{-7}
Sample Ion Gage (p_1)	5.3×10^{-8}	8.8×10^{-8}
Quartz Ion Gage (p_3)	1.94×10^{-8}	3.0×10^{-8}

$Q_o = C_o\ (p_o - p_1) = 2.64 \times 10^{-7}$ torr-liter/sec

$\dot{m} = (MQ_o/RT) \times 3600 \times 10^6 = 1.57$ microgram/hr.

TABLE 9-b

GAS COMPOSITION DURING THE EXPOSURE OF

Cb SPECIMEN NO. 51

EXPOSURE NO.9

	Gas Composition, Molar Percent					
Time, Hrs.*	CO_2	O_2	CO	H_2O	He	H_2
29	1.8	89.2	6.0	0.2	0	2.8
41	0.7	91.6	4.4	0.2	0	3.1
67	1.0	92.0	3.7	0.2	0	2.2
91	1.0	91.9	4.4	0.2	0	2.5
115	1.0	92.1	4.5	0.2	0	2.2
139	1.2	92.6	4.0	0.2	0	2.0
167	0.9	92.6	3.7	0.2	0	2.5
186	1.2	91.8	4.3	0.2	0	2.5
235	1.3	92.4	3.7	0.2	0	2.4

* From start of O_2 admission.

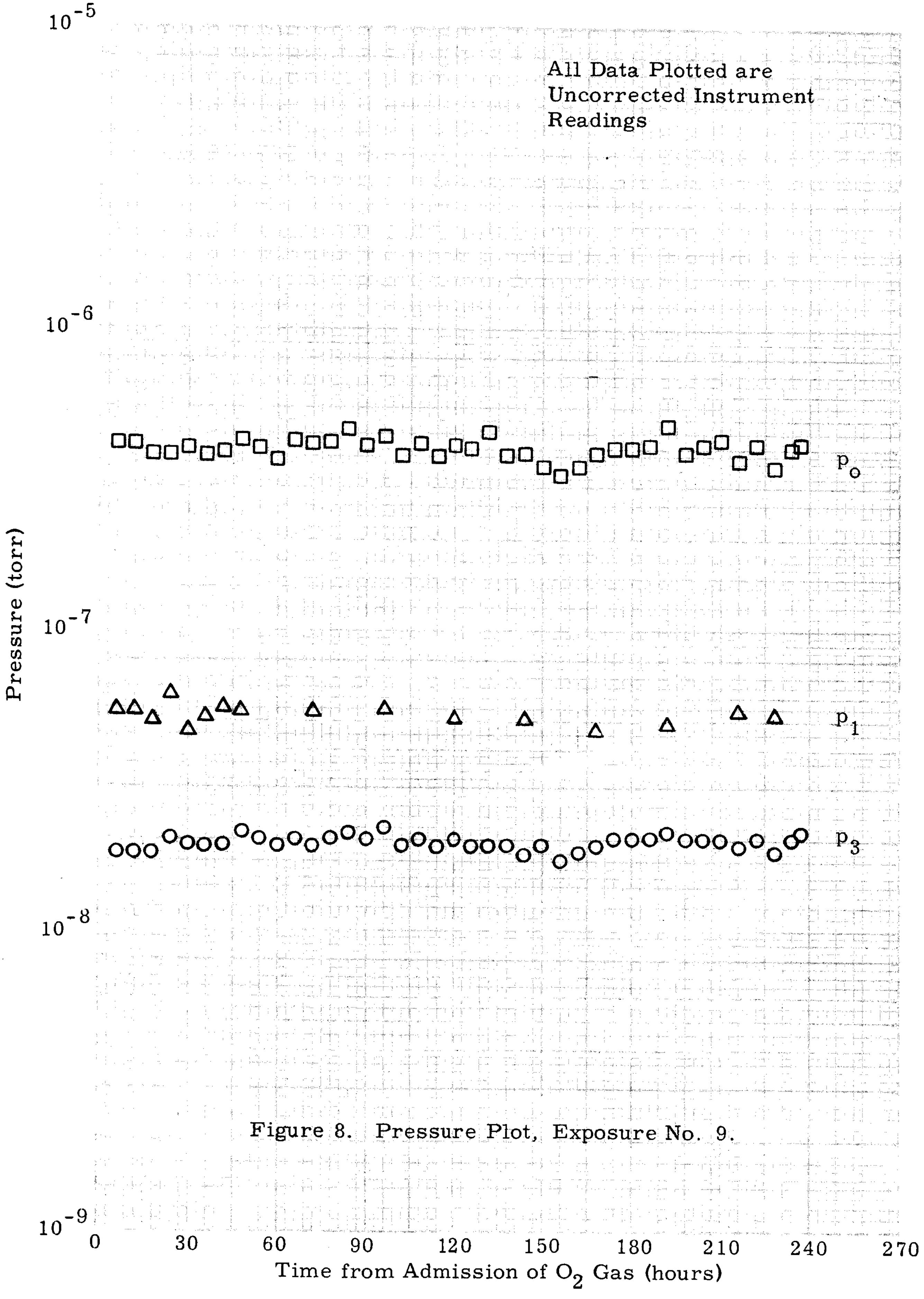

Figure 8. Pressure Plot, Exposure No. 9.

TABLE 10-a

GENERAL PARAMETERS, EXPOSURE NO. 10

Specimen Composition:	Zr (unalloyed)
Specimen Number:	111
Specimen Size:	0.030 x 0.957 x 1.500 in.
Specimen Weight:	4.622 gram
Specimen Temperature:	885, 980, and 1100°C
Pressure, "Sample Gage" (p_1):	1×10^{-7} to 1×10^{-5} torr (in steps)
Vacuum System:	10^{-10} system
Conductance Temperature (T_o):	313 deg K (40 deg C)
System Conductance:	$C_o = 0.172\ (T_o/M)^{1/2}$ liters/sec (for O_2 at 313°K, $C_o = 0.538$)
Gage Calibrations:	Second calibration, G. E. panels, Figures 19, 20 and 21
Distance L:	33 cm.

TABLE 10-b

DAILY LOG OF PRESSURE MEASUREMENTS

EXPOSURE NO. 10

Zr Specimen No. 111

Pressures given are uncorrected ion gage readings.

12-22-65	11:05 AM	Oxygen introduced to mid 10^{-9} torr system	
Wed.		Exposure 10^{-7} torr O_2 - 1100°C	
12-23-65	8:00 AM	Pump ig	1.9×10^{-6} torr
Thurs.		Sample ig	1.8×10^{-7} torr
		Quartz ig	2.2×10^{-8} torr
	11:00 AM	Specimen temp. decreased 980°C - 10^{-7} torr O_2	
12-24-65	10:21 AM	Pump ig	3.0×10^{-6} torr
Fri.		Sample ig	2.5×10^{-7} torr
		Quartz ig	2.3×10^{-8} torr
	11:00 AM	Specimen temp. decreased to 885°C - 10^{-7} torr O_2	
12-25-65	5:35 PM	Pump ig	3.2×10^{-6} torr
Sat.		Sample ig	2.8×10^{-7} torr
		Quartz ig	2.9×10^{-8} torr
		Specimen temp. 885°C	
12-26-65	7:30 PM	Pump ig	3.1×10^{-6} torr
Sun.		Sample ig	2.7×10^{-7} torr
		Quartz ig	2.9×10^{-8} torr
		Specimen temp. 885°C	
12-27-65	8:25 AM	Pump ig	2.7×10^{-6} torr
Mon.		Sample ig	2.3×10^{-7} torr
		Quartz ig	2.6×10^{-8} torr
		Specimen temp.885°C	
	9:30 AM	Increased O_2 to 10^{-6} torr	
	10:10 AM	Pump ig	1.4×10^{-5} torr
		Sample ig	1.1×10^{-6} torr
		Quartz ig	2.1×10^{-7} torr
12-28-65	8:10 AM	Pump ig	1.9×10^{-5} torr
Tues.		Sample ig	1.6×10^{-6} torr
		Quartz ig	1.4×10^{-7} torr
		Specimen temp. 885°C	
	9:30 AM	Specimen temp. increased to 980°C	

TABLE 10-b (cont'd.)

12-29-65	8:00 AM	Pump ig	2.0×10^{-5} torr
Wed.		Sample ig	1.5×10^{-6} torr
		Quartz ig	1.3×10^{-7} torr
	9:30 AM	Specimen temp. increased to 1100°C	
12-30-65	8:15 AM	Pump ig	1.8×10^{-5} torr
Thurs.		Sample ig	1.4×10^{-6} torr
		Quartz ig	1.8×10^{-7} torr
	9:30 AM	Increased O_2 to 10^{-5} torr Specimen temp. 1100°C	
12-31-65	9:50 AM	Pump ig	1.4×10^{-4} torr
Fri.		Sample ig	1.0×10^{-5} torr
		Quartz ig	7.2×10^{-7} torr
	10:15 AM	Specimen temp. decreased to 980°C	
1-1-66	6:00 PM	Pump ig	1.2×10^{-4} torr
Sat.		Sample ig	9.0×10^{-6} torr
		Quartz ig	6.6×10^{-7} torr
		Specimen temp. 980°C	
1-2-66	3:55 PM	Pump ig	1.3×10^{-4} torr
Sun.		Sample ig	1.0×10^{-5} torr
		Quartz ig	7.4×10^{-7} torr
		Specimen temp. 980°C	
1-3-66	9:20 AM	Pump ig	1.4×10^{-4} torr
Mon.		Sample ig	1.1×10^{-5} torr
		Quartz ig	8.1×10^{-7} torr
		Specimen temp. 980°C	
	9:30 AM	Reduced specimen temp. to 885°C	
1-4-66	8:15 AM	Pump ig	1.5×10^{-4} torr
Tues.		Sample ig	1.2×10^{-5} torr
		Quartz ig	9.3×10^{-7} torr
	10:00 AM	End of exposure	

TABLE 10-c

GAS COMPOSITION DURING THE EXPOSURE OF

Zr SPECIMEN NO. 111

EXPOSURE NO. 10

		Gas Composition, Molar Percent					
Exposure	**Time,Hrs.***	**CO_2**	**O_2**	**CO**	**H_2O**	**He**	**H_2**
1.6×10^{-7} torr-O_2-1100°C	24	3.1	62.6	1.0	1.0	0	32.3
2.5×10^{-7} torr-O_2- 980°C	48	1.3	95.3	0.3	0.2	0	2.9
2.3×10^{-7} torr-O_2- 885°C	118	0.5	98.6	0.3	0.1	0	0.5
1.6×10^{-6} torr-O_2- 885°C	142	0.1	99.6	0.2	0	0	0.1
1.5×10^{-6} torr-O_2- 980°C	166	0.3	99.4	0.2	0	0	0.1
1.4×10^{-6} torr-O_2-1100°C	190	0.3	99.0	0.1	0.1	0	0.5
1.0×10^{-5} torr-O_2-1100°C	215	0.2	99.5	0.1	0.1	0	0.1
1.1×10^{-5} torr-O_2- 980°C	286	0.1	99.8	0.1	0	0	0
1.2×10^{-5} torr-O_2- 885°C	310	0.1	99.8	0.1	0	0	0

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* From start of O_2 admission.

TABLE 10-d

GETTERING RATES

EXPOSURE NO. 10

Zr Specimen No. 111

Pressures given were taken at the end of each phase, and are corrected below by use of calibration data. Pressures are in torr.

					Gettering Rates		Approx.
Phase	Specimen Temp (°C)	Pump Gage, p_o	Sample Gage, p_1	Quartz Gage, p_3	Q_o torr L/sec	$\dot{m}$, micro-grams/hr	Exposure Time, Hrs.
1	1100	3.32(-6)	2.62(-7)	3.40(-8)	1.64(-6)	9.70	24
2	980	5.10(-6)	3.65(-7)	3.50(-8)	2.55(-6)	15.0	24
3	885	4.60(-6)	3.35(-7)	3.95(-8)	2.29(-6)	13.5	70
4	885	3.30(-5)	2.55(-6)	2.40(-7)	1.64(-5)	96.6	24
5	980	3.45(-5)	2.40(-6)	2.22(-7)	1.73(-5)	102.	24
6	1100	3.10(-5)	2.25(-6)	3.10(-7)	1.55(-5)	91.2	24
7	1100	1.4(-4)*	1.60(-5)	1.25(-6)	--	--	25
8	980	1.4(-4)*	1.70(-5)	1.41(-6)	--	--	71
9	885	1.5(-4)*	1.85(-5)	1.63(-6)	--	--	24

* Uncorrected instrument reading (no calibration available in this range).

NOTE: Figures in parentheses are powers of 10; 1(-4) = 1 x 10^{-4}

TABLE 11-a

GENERAL PARAMETERS, EXPOSURE NO. 11

Specimen Composition:	Cb-1Zr
Specimen Number:	90
Specimen Size:	0.045 x 0.965 x 1.500 in.
Specimen Weight:	9.003 gram
Specimen Temperature:	980 deg C
Pressure, "Sample Gage" (p_1):	5 x 10^{-8} to 3 x 10^{-5} torr (approx.) (in steps)
Vacuum System:	10^{-10} system
Conductance Temperature (T_o):	313 deg K (40 deg C)
System Conductance:	$C_o = 0.172\ (T_o/M)^{1/2}$ liters/sec (for O_2 at 313°K, $C_o = 0.538$)
Gage Calibrations:	Second calibration, C.V.C. panels, Figures 16, 17, and 18 (Figure 10 for 10^{-8} range)
Distance L:	36 cm.

TABLE 11-b

DAILY LOG OF PRESSURE MEASUREMENTS

EXPOSURE NO. 11

Cb-1Zr Specimen No. 90

Pressures given are uncorrected ion gage readings.

Specimen temperature = 980°C throughout exposure.

2-17-66	11:45 AM	Oxygen introduced to low 10^{-9} torr pressure Exposure 10^{-5} torr-O_2-980°C	
	4:20 PM	Pump ig Sample ig Quartz ig	1.1×10^{-4} torr 1.15×10^{-5} torr 1.2×10^{-6} torr
2-18-66	8:05 AM	Pump ig Sample ig Quartz ig	1.3×10^{-4} torr 1.2×10^{-5} torr 1.1×10^{-6} torr
	11:50 AM	O_2 reduced to 7×10^{-6} torr O_2	
2-19-66	10:30 AM	Pump ig Sample ig Quartz ig	8.5×10^{-5} torr 7.2×10^{-6} torr 7.4×10^{-7} torr
	11:45 AM	O_2 reduced to 1×10^{-6} torr O_2	
2-20-66	9:30 AM	Pump ig Sample ig Quartz ig	1.6×10^{-5} torr 1.6×10^{-6} torr 1.55×10^{-7} torr
	10:45 AM	O_2 reduced to 7×10^{-7} torr O_2	
2-21-66	8:15 AM	Pump ig Sample ig Quartz ig	8×10^{-6} torr 7.0×10^{-7} torr 1.0×10^{-7} torr
	11:30 AM	Reduced O_2 to 3×10^{-7} torr O_2	
2-22-66	8:30 AM	Pump ig Sample ig Quartz ig	3.1×10^{-6} torr 3.2×10^{-7} torr 5.1×10^{-8} torr
	11:45 AM	Increased O_2 to 3×10^{-5} torr O_2	
2-23-66	9:05 AM	Pump ig Sample ig Quartz ig	3.2×10^{-4} torr 3.0×10^{-5} torr 3.0×10^{-6} torr
	11:30 AM	Reduced O_2 to 1×10^{-7} torr O_2	

TABLE 11-b (cont'd.)

2-24-66	8:45 AM	Pump ig	1.1×10^{-6} torr
		Sample ig	1.25×10^{-7} torr
		Quartz ig	2.9×10^{-8} torr
	11:45 AM	Reduced O_2 to	7×10^{-8} torr O_2
2-25-66	9:00 AM	Pump ig	4.7×10^{-7} torr
		Sample ig	6.8×10^{-8} torr
		Quartz ig	1.7×10^{-8} torr
	11:45 AM	Reduced O_2 to	5×10^{-8} torr O_2
2-26-66	10:45 AM	Pump ig	2.2×10^{-7} torr
		Sample ig	5.0×10^{-8} torr
		Quartz ig	1.4×10^{-8} torr
	11:45 AM	Oxygen off -	Specimen heat off

TABLE 11-c

GAS COMPOSITION DURING THE EXPOSURE OF

Cb-1Zr SPECIMEN NO. 90

EXPOSURE NO. 11

Exposure	Time, Hrs.*	Gas Composition, Molar Percent					
		CO_2	O_2	CO	H_2O	He	H_2
1.2×10^{-5} torr-O_2- 980°C	22	2.1	97.1	0.2	0	0.6	0
7.2×10^{-6} torr-O_2- 980°C	48	1.4	97.7	0.2	0	0.7	0
1.4×10^{-6} torr-O_2- 980°C	70	3.5	90.8	0.7	0.1	4.5	0.4
7.0×10^{-7} torr-O_2- 980°C	95	1.3	92.2	0.5	0.1	5.7	0.2
3.3×10^{-7} torr-O_2- 980°C	119	1.0	93.8	0.5	0.3	1.5	2.9
2.8×10^{-5} torr-O_2- 980°C	143	0.6	99.3	0.1	0	0	0
1.25×10^{-7} torr-O_2- 980°C	167	15.5	75.6	6.6	0.4	0	1.9
7.0×10^{-8} torr-O_2- 980°C	191	19.0	65.5	12.0	0.5	0	3.0
4.9×10^{-8} torr-O_2- 980°C	216	18.0	59.1	19.0	0.4	0	3.5

* From start of O_2 admission.

TABLE 11-d

GETTERING RATES

EXPOSURE NO. 11

Cb-1Zr Specimen No. 90

Pressures given were taken at the end of each phase, and are corrected below by use of calibration data. Pressures are in torr.

					Gettering Rates		
Phase	Specimen Temp (°C)	Pump Gage, p_o	Sample Gage, p_1	Quartz Gage, p_3	Q_o torr L/ Sec	$\dot{m}$, micro-grams/hr	Approx. Exposure Time, Hrs.
1	980	1.3(-4)*	7.4(-6)	7.8(-7)	--	--	24
2	980	5.9(-5)	5.0 (-6)	5.8 (-7)	2.90(-5)	171	24
3	980	1.0(-5)	1.08(-6)	1.3 (-7)	4.80(-6)	28.3	23
4	980	5.1(-6)	4.8 (-7)	8.4 (-8)	2.49 (-6)	14.7	25
5	980	1.94(-6)	2.2 (-7)	4.3 (-8)	9.25 (-7)	5.46	24
6	980	3.2(-4)*	1.95(-5)	2.26(-6)	--	--	24
7	980	6.7(-7)	8.4 (-8)	2.7 (-8)	3.15 (-7)	1.86	24
8	980	3.2 (-7)	5.0 (-8)	1.75 (-8)	1.45 (-7)	0.857	24
9	980	1.5 (-7)	3.9 (-8)	1.48 (-8)	5.97 (-8)	0.352	24

* Uncorrected instrument reading (no calibration available in this range)

Note: Figure in parenthesis are powers of 10; 1(-4) = 1 x 10^{-4}

TABLE 12-a

GENERAL PARAMETERS, EXPOSURE NO. 12

Specimen Composition:	Cb-.75Zr
Specimen Number:	88
Specimen Size:	0.050 x 0.959 x 1.497 in.
Specimen Weight:	10.125 gram
Specimen Temperature:	885 and 980 deg C
Pressure, "Sample Gage" (p_1):	1×10^{-7} to 1×10^{-5} torr (approx.) (in steps)
Vacuum System:	10^{-8} system (with special induction heater)
Conductance Temperature (T_o)	298 deg K (25 deg C)
System Conductance:	$C_o = 0.203 \, (T_o/M)^{1/2}$ liters/sec (for O_2 at 298°K, $C_o = 0.619$)
Gage Calibrations:	Second calibration, G. E. panels, Figures 19, 20, and 21 (Figure 13 for 10^{-8} range).
Distance L:	35 cm

TABLE 12-b

DAILY LOG OF PRESSURE MEASUREMENTS

EXPOSURE NO. 12

Cb-.75Zr Specimen No. 88

Pressures given are uncorrected ion gage readings.

3-22-66	11:30 AM	Applied heat to specimen at 1005°C
	4:30 PM	Specimen temperature lowered to 980°C O_2 introduced to low 10^{-8} pressure Exposure 10^{-5} torr-O_2- 980°C
3-23-66	8:00 AM	Pump ig 1.4×10^{-4} torr Sample ig 1.0×10^{-5} torr Quartz ig 1.3×10^{-6} torr
	2:30 PM	O_2 off. Heat off.
	4:30 PM	Heat on - 980°C
	4:30 PM	O_2 on - 5×10^{-6} torr
3-24-66	7:45 AM	Pump ig 5.9×10^{-5} torr Sample ig 4.8×10^{-6} torr Quartz ig 5.2×10^{-7} torr
	11:50 AM	O_2 off. Heat off.
	4:10 PM	Heat on - 1005°C
	4:30 PM	O_2 on - 10^{-6} torr. Specimen temperature 980°C
3-25-66	8:00 AM	Pump ig 1.4×10^{-5} torr Sample ig 1.1×10^{-6} torr Quartz ig 1.5×10^{-7} torr
	1:30 PM	O_2 off. Heat off.
	4:20 PM	Heat on - 1005°C
	4:45 PM	O_2 on - 5×10^{-7} torr. Specimen temperature 980°C
3-26-66	10:00 AM	Pump ig 5.8×10^{-6} torr Sample ig 5.1×10^{-7} torr Quartz ig 7.5×10^{-8} torr
	10:05 AM	Reduced O_2 to 1×10^{-7} torr. Specimen temp. 980°C
3-27-66	10:00 AM	Pump ig 1.6×10^{-6} Sample ig 1.5×10^{-7} Quartz ig 2.5×10^{-8}
	10:20 AM	Increased O_2 to 10^{-5} torr Lowered temp. to 885°C
3-28-66	8:00 AM	Pump ig 1.3×10^{-4} torr Sample ig 1.0×10^{-5} torr Quartz ig 2.3×10^{-6} torr

TABLE 12-b(cont'd.)

3-28-66 (cont'd)			
	9:10 AM	O_2 off. Heat off.	
	2:30 PM	Heat on - 910°C	
	4:30 PM	O_2 on - 5 x 10^{-6} torr. Specimen temp. 885°C	
3-29-66	8:05 AM	Pump ig	5.2 x 10^{-5} torr
		Sample ig	4.7 x 10^{-6} torr
		Quartz ig	9.0 x 10^{-7} torr
	9:45 AM	O_2 off. Heat off.	
	11:30 AM	Heat on 910°C.	
	1:30 PM	O_2 on - 1 x 10^{-6} torr. Specimen temp. 885°C	
3-30-66	8:00 AM	Pump ig	1.2 x 10^{-5} torr
		Sample ig	1.5 x 10^{-6} torr
		Quartz ig	2.5 x 10^{-7} torr
	1:30 PM	O_2 reduced to 1 x 10^{-7}. Specimen temp. 885°C	
3-31-66	8:05 AM	Pump ig	8.7 x 10^{-7} torr
		Sample ig	1.0 x 10^{-7} torr
		Quartz ig	3.7 x 10^{-8} torr
	1:30 PM	O_2 off. Specimen heat off.	

TABLE 12-c

GAS COMPOSITION DURING THE EXPOSURE OF

Cb-.75Zr SPECIMEN NO. 88

EXPOSURE NO. 12

Exposure	Time,Hrs*	CO_2	O_2	CO	H_2O	He	H_2
		Gas Composition, Molar Percent					
1.0×10^{-5} torr-O_2- 980°C	0.3	3.8	95.6	0.6	0	0	0
1.0×10^{-5} torr-O_2- 980°C	21	5.1	94.5	0.4	0	0	0
4.9×10^{-6} torr-O_2- 980°C	40	1.8	97.7	0.4	0	0	0.1
1.1×10^{-6} torr-O_2- 980°C	57	6.5	92.7	0.7	0	0	0.1
5.1×10^{-7} torr-O_2- 980°C	74	10.0	88.4	1.4	0	0	0.2
1.5×10^{-7} torr-O_2- 980°C	98	14.4	81.7	3.3	0	0	0.6
1.1×10^{-5} torr-O_2- 885°C	120.5	6.8	92.7	0.5	0	0	0
4.6×10^{-6} torr-O_2- 885°C	137.5	6.8	92.7	0.5	0	0	0
1.2×10^{-6} torr-O_2- 885°C	161	8.2	90.6	1.1	0	0	0.1
1.0×10^{-7} torr-O_2- 885°C	185	23.0	68.2	8.3	0	0	0.5

* From start of O_2 admission.

TABLE 12-d

GETTERING RATES

EXPOSURE NO. 12

Cb-.75Zr Specimen No. 88

Pressures given were taken at the end of each phase, and are corrected below by use of calibration data. Pressures are in torr.

Phase	Specimen Temp (°C)	Pump Gage, p_0	Sample Gage, p_1	Quartz Gage, p_3	Gettering Rates Q_0 torr L/Sec	 m, micro-grams/hr	Approx. Exposure Time, Hrs.
1	980	1.4(-4)*	1.55(-5)	2.35(-6)	--	--	16
2	980	1.0(-4)	7.1 (-6)	9.0 (-7)	5.75 (-5)	357	16
3	980	2.4(-5)	1.8 (-6)	2.6 (-7)	1.38 (-5)	85.2	16
4	980	9.4(-6)	7.5 (-7)	1.1 (-7)	5.36 (-6)	33.2	18
5	980	2.85(-6)	2.2 (-7)	3.8 (-8)	1.63 (-6)	10.1	24
6	885	1.3 (-4)*	1.55(-5)	4.0 (-6)	--	--	22
7	885	8.9 (-5)	7.0 (-6)	1.57(-6)	5.08 (-5)	315	16
8	885	2.07(-5)	2.4 (-6)	4.3 (-7)	1.13 (-5)	70.2	18
9	885	1.46(-6)	1.45(-7)	5.6 (-8)	8.15 (-7)	5.05	18

* Uncorrected instrument reading (no calibration available in this range)

Note: Figures in parenthesis are powers of 10; 1(-4) = 1 x 10^{-4}

TABLE 13-a

GENERAL PARAMETERS, EXPOSURE NO. 13

Specimen Composition:	FS-85
Specimen Number:	110
Specimen Size:	0.029 x 0.953 x 1.496 in.
Specimen Weight:	6.807 gram
Specimen Temperature:	885 and 980 deg C
Pressure, "Sample Gage" (p_1):	1×10^{-7} to 1×10^{-5} torr (approx.) (in steps)
Vacuum System:	10^{-10} system
Conductance Temperature (T_o):	311 deg K (38 deg C)
System Conductance:	$C_o = 0.172 \ (T_o/M)^{1/2}$ liters/sec (for O_2 at 311^oK, $C_o = 0.537$)
Gage Calibrations:	Second calibration, C.V.C. panels, Figures 16, 17, and 18 (Figure 10 for 10^{-8} range).
Distance L:	37 cm

TABLE 13-b

DAILY LOG OF PRESSURE MEASUREMENTS

EXPOSURE NO. 13

FS-85 Specimen No. 110

Pressures given are uncorrected ion gage readings.

3-10-66	10:30 AM	System pressure at low 10^{-9}	
		Applied heat to specimen at 1025°C	
	11:30 AM	Oxygen introduced to low 10^{-8} pressure	
		Exposure 10^{-5} torr-O_2- 980°C	
3-11-66	8:00 AM	Pump ig	1.5×10^{-4}torr
		Sample ig	1.2×10^{-5}torr
		Quartz ig	1.3×10^{-6}torr
	11:30 AM	Oxygen reduced to 5×10^{-6}torr-O_2- 980°C	
3-12-66	10:00 AM	Pump ig	4.8×10^{-5}torr
		Sample ig	4.4×10^{-6}torr
		Quartz ig	4.5×10^{-7}torr
	11:15 AM	Oxygen reduced to 1×10^{-6}torr-O_2- 980°C	
3-13-66	9:40 AM	Pump ig	1.4×10^{-5}torr
		Sample ig	1.2×10^{-6}torr
		Quartz ig	1.5×10^{-7}torr
	10:30 AM	Oxygen reduced to 5×10^{-7} torr-O_2- 980°C	
3-14-66	9:40 AM	Pump ig	6.3×10^{-6}torr
		Sample ig	5.6×10^{-7}torr
		Quartz ig	8.1×10^{-8}torr
	11:30 AM	Oxygen reduced to 1×10^{-7} torr-O_2- 980°C	
3-15-66	8:05 AM	Pump ig	1.3×10^{-6}torr
		Sample ig	1.4×10^{-7}torr
		Quartz ig	3.0×10^{-8}torr
	11:30 AM	Oxygen increased to 1×10^{-5}rorr	
		Specimen temperature reduced to 885°C	
3-16-66	8:00 AM	Pump ig	1.3×10^{-4}torr
		Sample ig	1.2×10^{-5}torr
		Quartz ig	2.2×10^{-6}torr
	11:30 AM	Oxygen reduced to 5×10^{-6} torr-O_2 - 885°C	

TABLE 13-b (cont'd.)

3-17-66	8:10 AM	Pump ig	4.9×10^{-5}torr
		Sample ig	5.1×10^{-6}torr
		Quartz ig	8.7×10^{-7}torr
	11:30 AM	Oxygen reduced to 1.0×10^{-6}torr-O_2- 885°C	
3-18-66	8:10 AM	Pump ig	1.3×10^{-5} torr
		Sample ig	1.5×10^{-6} torr
		Quartz ig	3.5×10^{-7} torr
	11:30 AM	Oxygen reduced to 1×10^{-7} torr-O_2- 885°C	
3-19-66	10:15 AM	Pump ig	1.3×10^{-6} torr
		Sample ig	1.4×10^{-7} torr
		Quartz ig	3.5×10^{-8} torr
	11:30 AM	Oxygen off. Specimen heat off.	

TABLE 13-c

GAS COMPOSITION DURING THE EXPOSURE OF

FS-85 SPECIMEN NO. 110

EXPOSURE NO. 13

Exposure	Time, Hrs.*	CO_2	O_2	CO	H_2O	He	H_2
		Gas Composition, Molar Percent					
1.2×10^{-5} torr-O_2- 980°C	1	4.8	92.1	2.1	0.4	0	0.6
1.2×10^{-5} torr-O_2- 980°C	23	0.8	98.2	0.7	0.1	0.1	0.1
4.6×10^{-6} torr-O_2- 980°C	47	0.4	98.4	0.8	0.1	0.1	0.2
1.7×10^{-6} torr-O_2- 980°C	71	1.0	95.7	1.5	0.5	0.7	0.6
5.4×10^{-7} torr-O_2- 980°C	95	0.7	86.1	5.4	1.6	2.4	3.8
1.4×10^{-7} torr-O_2- 980°C	119	3.8	83.4	7.9	2.4	0.8	1.7
1.4×10^{-5} torr-O_2- 885°C	143	2.0	97.2	0.8	0	0	0
4.8×10^{-6} torr-O_2- 885°C	168	2.2	96.6	1.2	0	0	0
1.5×10^{-6} torr-O_2- 885°C	192	0.6	97.9	1.0	0	0	0.5
1.45×10^{-7} torr-O_2- 885°C	216	3.0	89.1	3.6	0.5	0	3.8

* From start of O_2 admission.

TABLE 13-d

GETTERING RATES

EXPOSURE NO. 13

FS-85 SPECIMEN NO. 110

Pressures given were taken at the end of each phase, and are corrected below by use of calibration data. Pressures are in torr.

Phase	Specimen Temp (^{o}C)	Pump Gage, p_o	Sample Gage, p_1	Quartz Gage, p_3	Gettering Rates		Approx. Exposure Time, Hrs.
					Q_o torr L/ Sec	m, micro-grams/hr	
1	980	1.5 (-4)*	7.4 (-6)	9.4 (-7)	--	--	21
2	980	3.2 (-5)	3.0 (-6)	3.45 (-6)	1.56 (-5)	92.3	23
3	980	8.7 (-6)	8.6 (-7)	1.25 (-7)	4.20 (-6)	25.0	22
4	980	4.0 (-6)	3.8 (-7)	6.3 (-8)	1.94 (-6)	11.5	23
5	980	8.0 (-7)	9.4 (-8)	2.8 (-8)	3.79 (-7)	2.25	20
6	885	1.3 (-4)*	7.4 (-6)	1.63 (-6)	--	--	20
7	885	3.3 (-5)	3.5 (-6)	6.8 (-7)	1.58 (-5)	93.9	20
8	885	8.1 (-6)	1.0 (-6)	2.8 (-7)	3.81 (-6)	22.6	20
9	885	8.0 (-7)	9.4 (-8)	3.2 (-8)	3.79 (-7)	2.25	24

* Uncorrected instrument reading (no calibration available in this range)

Note: Figures in parenthesis are powers of 10; 1(-4) = 1 x 10^{-4}

TABLE 14-a

GENERAL PARAMETERS, EXPOSURE NO. 14

Specimen Composition:	SCb-291
Specimen Number:	99
Specimen Size:	0.028 x 0.964 x 1.497 in.
Specimen Weight:	6.218 gram
Specimen Temperature:	885 and 980 deg C
Pressure, "Sample Gage" (p_1):	1×10^{-7} to 1×10^{-5} torr (approx.) (in steps)
Vacuum System:	10^{-10} system
Conductance Temperature (T_o):	311 deg K (38 deg C)
System Conductance:	$C_o = 0.172 \, (T_o/M)^{1/2}$ liters/sec (for O_2 at 311°K, $C_o = 0.536$)
Gage Calibrations:	Second calibration, C.V.C. panels, Figures 16, 17 and 18 (Figure 10 for 10^{-8} range).
Distance L:	35 cm.

TABLE 14-b

DAILY LOG OF PRESSURE MEASUREMENTS

EXPOSURE NO. 14

SCb-291 Specimen No. 99

Pressures given are uncorrected ion gage readings.

4-18-66	9:00 AM	Heat applied to specimen at 1005°C System pressure low 10^{-9}
4-20-66	11:20 AM	Oxygen introduced to high 10^{-10} torr pressure. Exposure 10^{-5} torr - O_2 - 980°C

Following this a series of steps was run with sample gage readings of 1×10^{-5}, 5×10^{-6}, ---etc.; however, the bakeable system valve (between glass system and pumps) had become positioned nearly closed, with a consequence of reduced pumping speed for residual gases, and a high partial pressure of these gases. Therefore, the opening of the valve was increased and a repeated exposure was started at this time, as tabulated below.

4-29-66	11:15 AM	Exposure 10^{-7} torr - O_2 - 885°C
4-30-66	10:00 AM	Pump ig 8.3×10^{-7} torr Sample ig 1.1×10^{-7} torr Quartz ig 2.9×10^{-8} torr
	10:05 AM	Oxygen increased to 1×10^{-6} torr - O_2 - 885°C
5-1-66	9:30 AM	Pump ig 1.25×10^{-5} torr Sample ig 1.35×10^{-6} torr Quartz ig 2.8×10^{-7} torr
	10:00 AM	Oxygen increased to 5×10^{-6} torr - O_2 - 885°C
5-2-66	9:30 AM	Pump ig 5.2×10^{-5} torr Sample ig 5.2×10^{-6} torr Quartz ig 1.3×10^{-6} torr
	10:15 AM	Oxygen increased to 1×10^{-5} torr - O_2 - 885°C
5-3-66	10:00 AM	Pump ig 1.2×10^{-4} torr Sample ig 1.3×10^{-5} torr Quartz ig 2.8×10^{-6} torr
	10:15 AM	Specimen temperature increased to 980°C Oxygen reduced to 1×10^{-7} torr O_2

TABLE 14-b(cont'd.)

5-4-66	10:50 AM	Pump ig	1.2×10^{-6} torr
		Sample ig	1.3×10^{-7} torr
		Quartz ig	3.2×10^{-8} torr
	11:00 AM	Oxygen increased to 5×10^{-7} torr - O_2 - 980°C	
5-5-66	10:00 AM	Pump ig	5.5×10^{-6} torr
		Sample ig	5.1×10^{-7} torr
		Quartz ig	1.2×10^{-7} torr
	10:15 AM	Oxygen increased to 10^{-6} torr - O_2 - 980°C	
5-6-66	9:30 AM	Pump ig	1.25×10^{-5} torr
		Sample ig	1.15×10^{-6} torr
		Quartz ig	2.1×10^{-7} torr
	10:00 AM	Oxygen increased to 5×10^{-6} torr - O_2 - 980°C	
5-7-66	9:20 AM	Pump ig	6.4×10^{-5} torr
		Sample ig	6.0×10^{-6} torr
		Quartz ig	1.2×10^{-6} torr
	9:30 AM	Oxygen increased to 10^{-5} torr - O_2 - 980°C	
5-8-66	9:45 AM	Pump ig	1.3×10^{-4}
		Sample ig	1.2×10^{-5} torr
		Quartz ig	2.2×10^{-6} torr
	10:30 AM	Oxygen off. Heat off.	

TABLE 14-c

GAS COMPOSITION DURING THE EXPOSURE OF

SCb-291 SPECIMEN NO. 99

EXPOSURE NO. 14

		Gas Composition, Molar Percent					
Exposure	**Time, Hrs.***	**CO_2**	**O_2**	**CO**	**H_2O**	**He**	**H_2**
1.1×10^{-7} torr-O_2-885°C	23	8.6	77.2	13.2	0	0	1.0
1.35×10^{-6} torr-O_2-885°C	46.5	3.7	94.0	2.2	0	0	0.1
5.2×10^{-6} torr-O_2-885°C	70.5	9.1	87.2	3.7	0	0	0
1.3×10^{-5} torr-O_2-885°C	95	2.6	96.3	1.1	0	0	0
1.3×10^{-7} torr-O_2-980°C	120	5.8	71.0	22.6	0	0	0.6
5.1×10^{-7} torr-O_2-980°C	143	2.6	85.7	11.3	0	0	0.4
1.15×10^{-6} torr-O_2-980°C	166.5	1.4	93.7	4.8	0	0	0.1
6.0×10^{-6} torr-O_2-980°C	190.5	2.1	95.3	2.6	0	0	0
1.2×10^{-5} torr-O_2-980°C	215	2.1	96.9	1.0	0	0	0

* From start of O_2 admission.

TABLE 14-d

GETTERING RATES

EXPOSURE NO.14

SCb-291 Specimen No. 99

Pressures given were taken at the end of each phase, and are corrected below by use of calibration data. Pressures are in torr.

					Gettering Rates		
Phase	**Specimen Temp (°C)**	**Pump Gage, p_o**	**Sample Gage, p_1**	**Quartz Gage, p_3**	Q_o torr L/ sec	$\dot{m}$, micro-grams/hr	**Approx. Exposure Time, Hrs.**
1	885	5.6(-7)	7.4(-8)	2.7(-8)	2.61(-7)	1.55	23
2	885	7.7(-6)	9.1(-7)	2.3(-7)	3.64(-6)	21.6	24
3	885	3.5(-5)	3.6(-6)	9.4(-7)	1.68(-5)	99.9	24
4	885	1.2(-4)*	8.1(-6)	2.1(-6)	--	--	24
5	980	7.3(-7)	8.7(-8)	2.9(-8)	3.45(-7)	2.05	24
6	980	3.5(-6)	3.5(-7)	1.0(-7)	1.69(-6)	10.0	23
7	980	7.7(-6)	8.2(-7)	1.73(-7)	3.69(-6)	21.9	24
8	980	4.4(-5)	4.1(-6)	8.6(-6)	2.14(-5)	127	23
9	980	1.3(-4)*	7.4(-6)	1.63(-6)	--	--	24

* Uncorrected instrument reading (no calibration available in this range)

Note: Figures in parenthesis are powers of 10; 1(-4) = 1 x 10^{-4}

TABLE 15-a

GENERAL PARAMETERS, EXPOSURE NO. 15

Specimen Composition:	Ta (unalloyed)
Specimen Number:	94
Specimen Size:	0.031 x 0.963 x 1.496 in.
Specimen Weight:	12.069 gram
Specimen Temperature:	885 and 950 deg C
Pressure, "Sample Gage" (p_1):	1×10^{-7} to 1×10^{-5} torr (approx.) (in steps)
Vacuum System:	10^{-8} system (with special induction heater)
Conductance Temperature (T_o):	300 deg K (27 deg C)
System Conductance:	$C_o = 0.203\ (T_o/M)^{1/2}$ liters/sec (for O_2 at 300°K, $C_o = 0.622$)
Gage Calibrations:	Second calibration, G. E. panels, Figures 19, 20 and 21 (Figure 13 for 10^{-8} range).
Distance L:	35 cm.

TABLE 15-b

DAILY LOG OF PRESSURE MEASUREMENTS

EXPOSURE NO. 15

Ta Specimen No. 94

Pressures given are uncorrected ion gage readings.

4-22-66	3:40 PM	Heat applied to specimen at 980°C
	4:15 PM	Oxygen introduced to 1.0×10^{-8} pressure Exposure 10^{-5} torr-O_2- 950°C
	5:15 PM	Pump ig 1.1×10^{-4} torr Sample ig 1.0×10^{-5} torr Quartz ig 2.9×10^{-6} torr
4-23-66	9:30 AM	Pump ig 1.1×10^{-4} torr Sample ig 1.4×10^{-5} torr Quartz ig 5.5×10^{-6} torr
	10:50 AM	O_2 reduced to 5×10^{-6} torr-O_2-950°C
4-24-66	10:20 AM	Pump ig 4.2×10^{-5} torr Sample ig 5.4×10^{-6} torr Quartz ig 2.2×10^{-6} torr
	10:30 AM	O_2 reduced to 10^{-6} torr-O_2-950°C
4-25-66	10:30 AM	Pump ig 1.1×10^{-5} torr Sample ig 1.4×10^{-6} torr Quartz ig $6. \times 10^{-7}$ torr
	10:40 AM	O_2 reduced to 5×10^{-7} torr-O_2-950°C
4-26-66	10:00 AM	Pump ig 2.9×10^{-6} torr Sample ig 4.6×10^{-7} torr Quartz 2.2×10^{-7} torr
	10:30 AM	O_2 reduced to 1×10^{-7} torr-O_2-950°C
4-27-66	8:00 AM	Pump ig 7.0×10^{-7} torr Sample ig 1.2×10^{-7} torr Quartz ig 7.5×10^{-8} torr
	1:45 PM	Specimen temperature reduced to 885°C O_2 increased to 1×10^{-5} torr-O^2

TABLE 15-b (cont'd.)

4-28-66	9:00 AM	Pump ig 1.1 x 10^{-4} torr Sample ig 1.6 x 10^{-5} torr Quartz ig 8.3 x 10^{-6} torr
	9:30 AM	0_2 off Specimen heat off
	4:10 PM	Heat applied to specimen at 910°C
	4:40 PM	0_2 introduced - 5 x 10^{-6} torr-0_2-885°C
4-29-66	11:45 AM	Pump ig 2.2 x 10^{-5} torr Sample ig 4.6 x 10^{-6} torr Quartz ig 2.6 x 10^{-6} torr
	12:00 Noon	0_2 off
	12:30 PM	Specimen heat off
5-2-66	3:10 PM	Heat applied to specimen at 910°C
	4:30 PM	0_2 introduced - 1 x 10^{-6} torr-885°C
5-3-66	8:30 AM	Pump ig 6.4 x 10^{-6} torr Sample ig 1.15 x 10^{-6} torr Quartz ig 7.2 x 10^{-7} torr
	8:50 AM	0_2 Off
	9:00 AM	Specimen heat off
	3:00 PM	Heat applied to specimen at 910°C
	4:30 PM	0_2 introduced - 1 x 10^{-7} torr - 885°C
5-4-66	10:00 AM	Pump ig 5.9 x 10^{-7} torr Sample ig 1.4 x 10^{-7} torr Quartz ig 9.5 x 10^{-8}
	10:10 AM	0_2 off. Specimen heat off.

TABLE 15-c

GAS COMPOSITION DURING THE EXPOSURE OF

Ta SPECIMEN NO. 94

EXPOSURE NO. 15

Exposure	Time, Hrs.*	Gas Composition, Molar Percent					
		CO_2	O_2	CO	H_2O	He	H_2
1×10^{-5} torr-O_2- 950°C	1	15.9	82.7	1.4	0	0	0
1.4×10^{-5} torr-O_2- 950°C	19	4.7	94.8	0.5	0	0	0
5.4×10^{-6} torr-O_2- 950°C	42	6.3	93.1	0.6	0	0	0
1.4×10^{-6} torr-O_2- 950°C	66	6.5	92.7	0.8	0	0	0
4.6×10^{-7} torr-O_2- 950°C	89.5	11.9	86.0	2.1	0	0	0
1.2×10^{-7} torr-O_2- 950°C	111	19.5	74.0	6.4	0	0	0.1
1.6×10^{-5} torr-O_2- 885°C	130	1.1	98.8	0.1	0	0	0
4.8×10^{-6} torr-O_2- 885°C	149	3.4	96.2	0.4	0	0	0
1.15×10^{-6} torr-O_2- 885°C	165	4.4	95.1	0.5	0	0	0
1.4×10^{-7} torr-O_2- 885°C	182.5	10.2	85.4	4.4	0	0	0

* From start of O_2 admission.

TABLE 15-d

GETTERING RATES

EXPOSURE NO.15

Ta Specimen No. 94

Pressures given were taken at the end of each phase, and are corrected below by use of calibration data. Pressures are in torr.

					Gettering Rates		Approx.
Phase	**Specimen Temp (°C)**	**Pump Gage, p_o**	**Sample Gage, p_1**	**Quartz Gage, p_3**	Q_o torr L/sec	$\dot{m}$, micrograms/hr	**Exposure Time, Hrs.**
1	950	1.1(-4)*	2.15(-5)	8.9(-6)	--	--	17
2	950	7.2(-5)	7.9(-6)	3.8(-6)	3.98(-5)	245	23
3	950	1.9(-5)	2.25(-6)	1.05(-6)	1.04(-5)	64.1	24
4	950	4.9(-6)	6.7(-7)	3.8(-7)	2.63(-6)	16.2	23
5	950	1.18(-6)	1.74(-7)	1.1(-7)	6.25(-7)	3.85	21
6	885	1.1(-4)*	2.5(-5)	1.2(-5)	--	--	19
7	885	3.8(-5)	6.8(-6)	4.5(-6)	1.94(-5)	119	19
8	885	1.03(-5)	1.88(-6)	1.25(-6)	5.23(-6)	32.2	16
9	885	8.7(-7)	2.1(-7)	8.7(-8)	4.10(-7)	2.52	17

*Uncorrected instrument reading (no calibration available in this range)

Note: Figures in parenthesis are powers of 10; 1(-4) = 1 x 10^{-4}

7 EXPERIMENTAL RESULTS FROM ION GAGE TESTS

7-1. Effect of Emission Current

Early in this program there was a strong indication that a 1 ma emission current would eliminate the instabilities which had been observed when the gages were operated at 0.1 ma emission. This brought about a change to a 1 ma emission current for all system gages. Some earlier tests had indicated that gage readings in the 10^{-9} torr range were quite different for the two emission currents.

To test this effect at higher pressures, tests were made on 13 January 1966, comparing 1 ma to 0.1 ma in the 10^{-9} through the 10^{-6} torr decades. The tests were made while the gages were in the calibration position (see Section 8). Results are shown in Table 16.

It can be seen from these results that there was very little, if any, effect of these emission currents, even in the 10^{-9} torr pressure range. The reason these results did not check with the previous tests is not known.

Later, during the 11th exposure, tests were made on the effect of 0.1, 1.0, and 5 ma emission currents. Results are given in Table 17. In these tests the 1.0 ma emissions gave generally higher readings than either the 0.1 or the 5 ma, by factors usually in the range 1.2 to 1.4.

7-2. Effect of Ion Gages on Gas Composition

Tests were made to determine whether the ion gages (with thoria-coated iridium filaments) were sources of CO and CO_2. The results, presented in Tables 18 and 19, indicate that the gages do seem to play a part in the production of these gases. For example, Table 19 shows that at 10^{-7} torr pressure, with all three gages on, the percentage of the residual gases went down as the emission current was lowered from 5 ma to 0.1 ma. Then, at 1.0 ma emission current, the residual gas percentage went down further as the gages were turned off one by one. Table 18 gives results for the 10^{-8} system (at room temperature), and Table 19 is for the 10^{-10} system with the specimen furnace on (but with no specimen in place).

TABLE 16

TESTS MADE TO COMPARE

1 MA WITH 0.1 MA EMISSION

Date of Tests:	13 January 1966
Gages used:	Those from 10^{-10} system with G.E. Controllers
Gas:	Oxygen

QUARTZ GAGE (P_3)		PUMP GAGE (P_1)		SAMPLE GAGE (P_o)	
1 ma	0.1 ma	1 ma	0.1 ma	1 ma	0.1 ma
7.4 (-8)	7.8 (-9)	7.2 (-8)	7.5 (-9)	8.2 (-8)	8.3 (-9)
4.3 "	4.3 "	4.0 "	4.0 "	4.6 "	4.4 "
3.1 "	2.9 "	2.7 "	2.6 "	3.0 "	2.7 "
1.6 "	1.6 "	1.5 "	1.5 "	1.7 "	1.5 "
8.0 "	8.1 "	7.8 "	7.8 "	8.7 "	8.6 "
1.95 (-7)	1.95 (-8)	1.95 (-7)	1.9 (-8)	2.25 (-7)	2.25 (-8)
4.6 "	4.7 "	4.65 "	4.6 "	5.3 "	5.2 "
6.8 "	7.0 "	6.9 "	6.85 "	7.8 "	7.6 "
1.6 (-6)	1.6 (-7)	1.6 (-6)	1.55 (-7)	1.75 (-6)	1.7 (-7)
5.45 "	5.4 "	5.5 "	5.5 "	6.3 "	6.1 "
7.2 "	7.0 "	7.25 "	7.15 "	8.1 "	7.8 "

All above readings were obtained by leaving all gages at 1 ma, then turning 1 one gage at a time down to 0.1 ma emission, and taking a reading after steady conditions were reached (in about 1 min.). To check long-term effects, the Quartz and Pump gages were then left at 0.1 ma overnight, and the following readings obtained the next morning (this time, by turning the emission <u>up</u> to 1 ma from 0.1 ma):

5.4 (-8)	5.6 (-9)	5.5 (-8)	5.6 (-9)	5.7 (-8)	5.5 (-9)

The system was then pumped down to the next lower decade (where the fraction O_2 was probably quite low) and the following results obtained:

9.4 (-9)	8.8 (-10)	8.3 (-9)	8.1 (-10)	8.8 (-9)	8.3 (-10)
7.0 (-9)	7.2 (-10)	6.7 (-9)	6.5 (-10)	7.0 (-9)	6.8 (-10)

Notes:

(1) Numbers in parentheses are powers of ten: (-6) = x 10^{-6}

(2) Table values are actual instrument readings (uncorrected). The values under "1 ma" are pressures in torr. The values under "0.1 ma" would have to be multiplied by 10 to obtain pressure in torr.

TABLE 17

TESTS TO DETERMINE THE EFFECT OF EMISSION CURRENT ON ION GAGE READINGS

Exposure No. 11

Time			Emission Current		
		Gage	1 mA	0.1 ma	5.0 ma
2-17-66	3:00 PM	P	1.25 (-4)	8.2 (-5)	8.2 (-5)
		S	1.15 (-5)	6.3 (-6)	7.4 (-6)
		Q	1.35 (-6)	1.0 (-6)	1.0 (-6)
2-18-66	11:00 AM	P	1.2 (-4)	8.5 (-5)	8.0 (-5)
		S	1.0 (-5)	6.3 (-6)	7.4 (-6)
		Q	1.2 (-6)	8.5 (-7)	8.4 (-7)
	2:35 PM	P	7.4 (-5)	5.5 (-5)	5.4 (-5)
		S	6.6 (-6)	4.2 (-6)	5.0 (-6)
		Q	6.8 (-7)	5.0 (-7)	5.0 (-7)
2-19-66	10:30 AM	P	8.5 (-5)	6.2 (-5)	5.8 (-5)
		S	7.2 (-6)	4.6 (-6)	5.2 (-6)
		Q	7.4 (-7)	5.5 (-7)	6.6 (-7)
	12:30 PM	P	1.5 (-5)	1.2 (-5)	1.2 (-5)
		S	1.4 (-6)	1.0 (-6)	1.1 (-6)
		Q	1.8 (-7)	1.4 (-7)	1.4 (-7)
2-20-66	9:30 AM	P	1.6 (-5)	1.15 (-5)	1.2 (-5)
		S	1.6 (-6)	9.4 (-7)	1.0 (-6)
		Q	1.55 (-7)	1.25 (-7)	1.2 (-7)
	11:00 AM	P	8.0 (-6)	5.4 (-6)	5.4 (-6)
		S	6.7 (-7)	5.0 (-7)	5.3 (-7)
		Q	1.0 (-7)	7.5 (-8)	7.8 (-8)
2-21-66	8:15 AM	P	8.0 (-6)	5.2 (-6)	5.2 (-6)
		S	7.0 (-7)	4.9 (-7)	5.0 (-7)
		Q	1.0 (-7)	7.5 (-8)	8.4 (-8)
	3:05 PM	P	3.0 (-6)	1.9 (-6)	2.0 (-6)
		S	3.2 (-7)	2.3 (-7)	2.8 (-7)
		Q	5.2 (-8)	4.1 (-8)	4.6 (-8)
2-22-66	8:00 AM	P	3.1 (-6)	2.0 (-6)	2.2 (-6)
		S	3.2 (-7)	2.4 (-7)	2.6 (-7)
		Q	5.1 (-8)	4.3 (-8)	4.7 (-8)
	1:15 PM	P	3.3 (-4)	2.1 (-4)	2.2 (-4)
		S	2.8 (-5)	1.7 (-5)	2.0 (-5)
		Q	2.5 (-6)	1.7 (-6)	1.8 (-6)

TABLE 17 (cont'd.)

Time			Emission Current		
		Gage	1 ma	0.1 ma	5.0 ma
2-23-66	9:05 AM	P	3.2 (-4)	2.2 (-4)	2.2 (-4)
		S	3.0 (-5)	1.9 (-5)	2.0 (-5)
		Q	3.0 (-6)	2.1 (-6)	2.0 (-6)
	4:15 PM	P	1.3 (-6)	1.0 (-6)	1.2 (-6)
		S	1.4 (-7)	1.0 (-7)	1.2 (-7)
		Q	3.6 (-8)	3.4 (-8)	3.4 (-8)
2-24-66	8:45 AM	P	1.1 (-6)	6.7 (-7)	7.4 (-7)
		S	1.25 (-7)	1.0 (-7)	1.1 (-7)
		Q	2.9 (-8)	2.7 (-8)	2.7 (-8)
	2:12 PM	P	4.8 (-7)	3.2 (-7)	3.6 (-7)
		S	7.2 (-8)	6.8 (-8)	7.0 (-8)
		Q	2.0 (-8)	1.6 (-8)	1.8 (-8)
2-25-66	9:00 AM	P	4.7 (-7)	3.1 (-7)	3.6 (-7)
		S	6.8 (-8)	6.4 (-8)	6.4 (-8)
		Q	1.7 (-8)	1.4 (-8)	1.6 (-8)
	4:00 PM	P	2.3 (-7)	1.5 (-7)	1.8 (-7)
		S	4.8 (-8)	4.8 (-8)	4.8 (-8)
		Q	1.6 (-8)	1.3 (-8)	1.8 (-8)
2-26-66	10:45 AM	P	2.2 (-7)	1.7 (-7)	1.8 (-7)
		S	5.0 (-8)	5.3 (-8)	4.8 (-8)
		Q	1.4 (-8)	1.3 (-8)	1.6 (-8)

Note: Numbers in parentheses are powers of 10.
All pressures are uncorrected ion gage readings.

TABLE 18

EFFECT OF ION GAGES ON GAS COMPOSITION

TESTS MADE ON MAY 12 and 13, 1966

on 10^{-10} System

Uncorrected Ion Gage Readings Torr				Gas Composition Molar Percent					
Pump Gage P_o	**Sample Gage** P_1	**Pressure Range (Multiplier)**	**Emission**	O_2	CO_2	CO	H_2O	He	H_2
0.74	0.7	10^{-5}	5 ma	97.0	1.2	1.6	0.2	0	0
1.2	1.2	10^{-5}	1 ma	97.6	1.0	1.3	0.1	0	0
0.76	0.66	10^{-5}	0.1 ma	97.5	1.0	1.4	0.1	0	0
0.62	0.6	10^{-6}	5 ma	93.8	2.1	3.0	0.7	0	0.4
1.1	1.1	10^{-6}	1 ma	94.4	2.0	2.8	0.5	0	0.3
0.65	0.71	10^{-6}	0.1 ma	95.0	2.0	2.3	0.5	0	0.2
0.62	1.2	10^{-7}	5 ma	76.5	6.0	8.2	3.6	0	5.7
1.0	1.7	10^{-7}	1 ma	80.5	4.9	8.2	2.6	0	3.8
0.8	1.7	10^{-7}	0.1 ma	81.7	6.5	6.8	2.1	0	2.9

Notes: Oxygen admitted through silver leak.
Quartz furnace maintained at 980°C.
System empty (no specimen in place).
Vacuum maintained between the two walls of quartz furnace.

Quartz gage (p_3) had burned out; therefore, no p_3 readings are available.

TABLE 19

EFFECT OF ION GAGES ON GAS COMPOSITION

Tests made on May 18, 1966

on 10^{-8} System

Uncorrected Ion Gage Readings Torr			Pressure Range (Multiplier)	Emission	Gas Composition Molar Percent		
Pump Ga. P_o	Sample Ga. P_1	Quartz Ga. P_3			O_2	CO_2	CO
1.3	1.4	1.2	10^{-5}	1 ma	97.3	2.4	0.3
1.4	1.5	1.2	10^{-6}	1 ma	96.3	3.2	0.5
1.6	1.6	1.4	10^{-7}	5 ma	75.5	19.0	5.5
1.5	1.4	1.2	10^{-7}	1 ma	78.0	18.1	3.9
1.6	1.6	1.4	10^{-7}	0.1 ma	85.9	12.0	2.1
1.7	1.7	Off	10^{-7}	1 ma	89.2	8.7	2.1
1.7	Off	Off	10^{-7}	1 ma	93.3	5.7	1.0
Off	Off	Off	*	--	96.0	3.5	0.5

*All gages were off, but conditions were held constant, so it is presumed that pressures remained constant.

Notes: H_2O, He and H_2 were negligible

O_2 Admitted through silver leak

System empty (no specimen in place) and entire system at room temperature

8. ION GAGE CALIBRATIONS FOR OXYGEN

The three ion gages used in each system (referred to as the "system" ion gages) were calibrated in the following manner. First, two special ion gage tubes and their respective control panels (referred to as the "secondary standard" ion gages) were sent to an independent laboratory for calibration. Each "secondary standard" ion gage tube was accompanied by its own individual control panel so that the characteristics of the control panel -- as well as the gage tube -- would be included in the calibration. The calibration of two secondary standards permitted periodic checks of one against the other, increasing the confidence level of each subsequent gage calibration; in addition, the risk of losing the calibration because of the malfunction of a single tube or panel was reduced.

After calibration of the secondary standards, both secondary standards and the three system gages were mounted in close proximity on a manifold as shown in Figure 9. The reason for close-mounting the gages is to reduce error from either gage pumping or system outgassing. Readings from all five gages were then taken at several steady-state pressure levels in each decade of pressure. The "true" pressure at each level was then taken as the arithmetic average of the corrected pressures of the two secondary standards.

There were three different calibrations used during the program. Each is described in the sections which follow.

8-1. Previous Calibration (Oxygen Gas)

At the beginning of the program there was a calibration in existence from the previous program; this calibration is given in the Final Report covering the period 22 January 1964 to 22 January 1965, NASA Contract NAS 3-4169. That calibration was made with emission currents of both the system gages and the secondary standards of 0.1 milliamp. The reason for this low emission current was to reduce gage pumping errors.

Figure 9. Photograph of Gages Mounted for Calibration

The calibration was used for the first two exposures, after which a new calibration at increased emission was generated.

8-2. First Calibration (Oxygen Gas)

The first calibration made during the present program was performed to increase the emission current of the system gages to 1.0 milliamp (the reasons for this were given in Section 3-3 "Instrumentation"). No new calibration of the secondary standards was made at this time. Results of the calibration are given in Tables 20 and 21, and plots are given in Figures 10 through 15. For the 10^{-5} torr range, an extrapolation of the curves had to be made.

8-3. Second Calibration (Oxygen Gas)

In preparation for the second calibration, both secondary standard gages were sent to an independent laboratory (AVCO Corporation, Wilmington, Massachusetts) for recalibration. The AVCO calibration was made in the decades 10^{-7}, 10^{-6}, and 10^{-5} torr, and had an estimated error limit of ± 10% of reading. Plotted results of the AVCO calibration are given in the Appendix, Section 9-3.

Results of the second calibration are given in Tables 22 and 23, and plots are given in Figures 16 through 21.

TABLE 20

FIRST CALIBRATION, ION GAGES WITH C.V.C. CONTROL PANELS

OXYGEN GAS

True Pressure (torr)	"Q" ion gage Panel 3016 (p_3)	"P" ion gage Panel 7087 (p_o)	"S" ion gage Panel 9097 (p_1)
	2.3×10^{-9} torr	2.3×10^{-9} torr	1.8×10^{-9} torr (O_2 off)
1.04×10^{-8} torr	9.5	9.6	8.7
1.64	1.6×10^{-8} torr	1.65×10^{-8} torr	1.55×10^{-8} torr
2.07	2.4	2.35	2.2
4.4	6.0	6.25	5.5
6.22	8.55	8.85	7.75
1.08×10^{-7} torr	1.55×10^{-7} torr	1.55×10^{-7} torr	1.35×10^{-7} torr
1.75	2.6	2.6	2.1
3.1	4.7	4.55	3.8
4.8	8.1	8.0	6.45
8.95	1.6×10^{-6} torr	1.4×10^{-6} torr	1.2×10^{-6} torr
1.7×10^{-6} torr	2.7	2.45	2.15
2.53	3.95	3.85	3.0
5.05	7.5	7.4	5.9
7.3	1.0×10^{-5} torr	1.0×10^{-5} torr	8.0
9.75	1.4	1.3	1.0
1.2×10^{-5} torr	1.6	1.7	1.4
1.71	2.3	2.35	2.0
2.23	2.95	3.0	2.55

TABLE 21

FIRST CALIBRATION, ION GAGES WITH G. E. CONTROL PANELS

OXYGEN GAS

True Pressure (torr)	"Q" ion gage Panel 40011 (p_3)	"P" ion gage Panel 50004 (p_o)	"S" ion gage Panel 40012 (p_1)	
	1.3×10^{-9} torr	1.3×10^{-9} torr	1.1×10^{-9} torr	(O_2 off)
2.20	1.5	1.7	1.25	
2.44	1.7	2.0	1.4	
4.93	3.3	4.9	2.4	
7.45	5.0	7.4	3.5	
9.3	6.0	9.0	4.2	
1.2×10^{-8} torr	8.4	1.2×10^{-8} torr	5.9	
1.63	1.0×10^{-8} torr	1.5	7.8	
1.91	1.2	1.7	9.4	
2.09	1.4	1.85	1.0×10^{-8} torr	
2.47	1.6	2.1	1.2	
4.9	3.2	3.7	2.7	
5.13	3.4	3.95	2.9	
5.73	3.8	4.3	3.3	
8.0	5.4	6.0	4.8	
9.4	6.4	7.0	5.7	
1.04×10^{-7} torr	6.9	7.5	6.3	
1.16	7.35	8.0	6.7	
1.4	1.0×10^{-7} torr	1.0×10^{-7} torr	9.4	
1.69	1.1	1.2	1.1×10^{-7} torr	
2.23	1.45	1.5	1.4	
3.98	2.55	2.7	2.6	
5.8	3.9	4.1	3.9	
6.1	4.2	4.3	4.2	
8.85	6.05	6.2	6.05	
9.2	6.3	6.5	6.3	
1.06×10^{-6} torr	7.25	7.5	7.3	
1.8	1.2×10^{-6} torr	1.2×10^{-6} torr	1.2×10^{-6} torr	
2.03	1.3	1.35	1.3	
2.93	1.9	1.95	1.9	
3.95	2.55	2.6	2.55	
5.4	3.5	3.5	3.45	
6.4	4.0	4.05	4.0	
9.5	5.9	5.9	5.8	
1.015×10^{-5} torr	6.2	6.25	6.15	
1.25	7.6	7.7	7.55	
1.3	8.0	8.05	7.9	
1.32	8.2	8.2	8.1	
	9.1	9.2	8.95	
1.5	9.1	9.2	9.0	
	9.2	9.3	9.05	
2.08	1.25×10^{-5} torr	1.25×10^{-5} torr	1.2×10^{-5} torr	

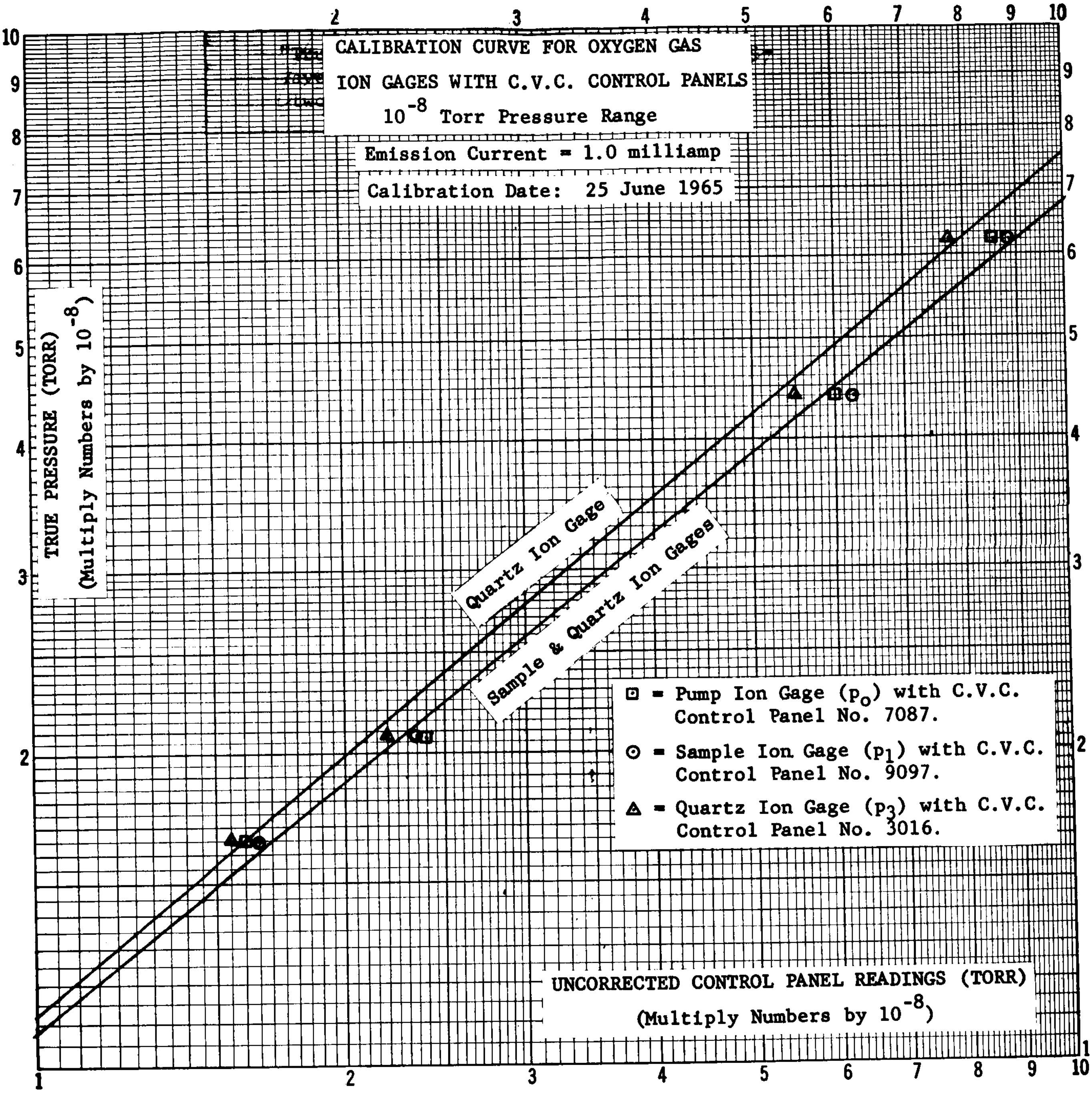


Figure 10. First Gage Calibration, C.V.C. Panels, 10^{-8} Range.

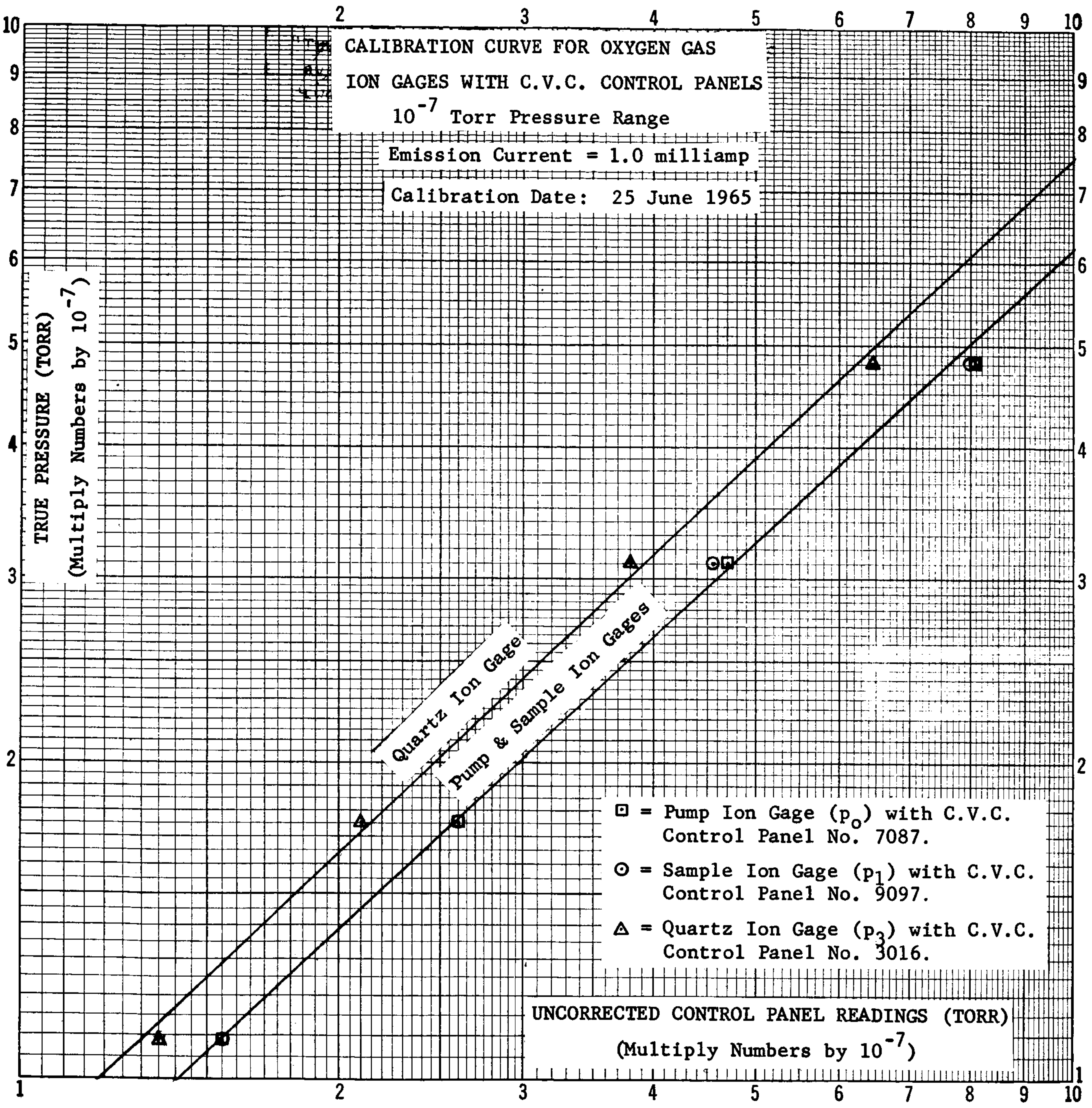


Figure 11. First Gage Calibration, C.V.C. Panels, 10^{-7} Range.

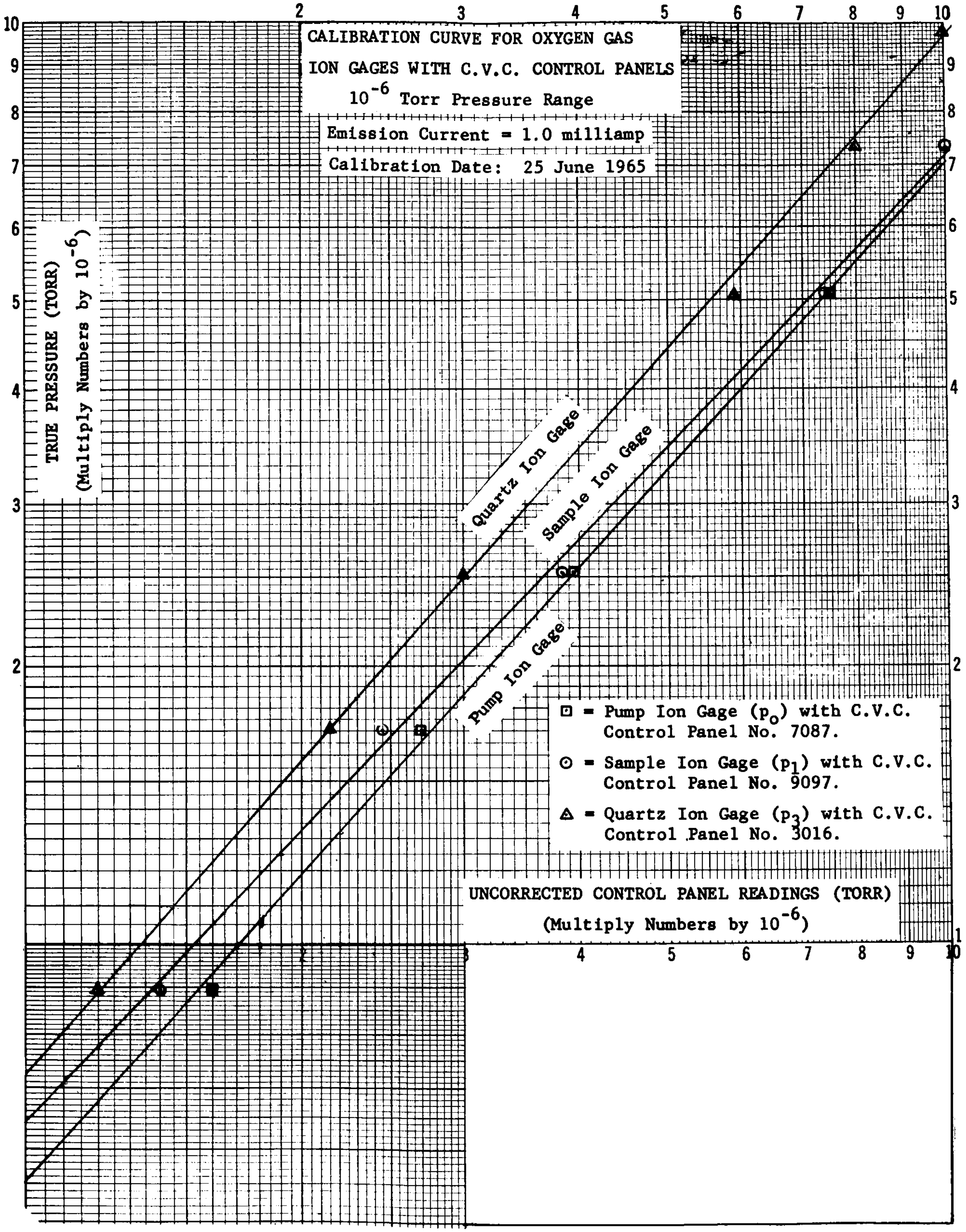


Figure 12. First Gage Calibration, C.V.C. Panels, 10^{-6} Range.

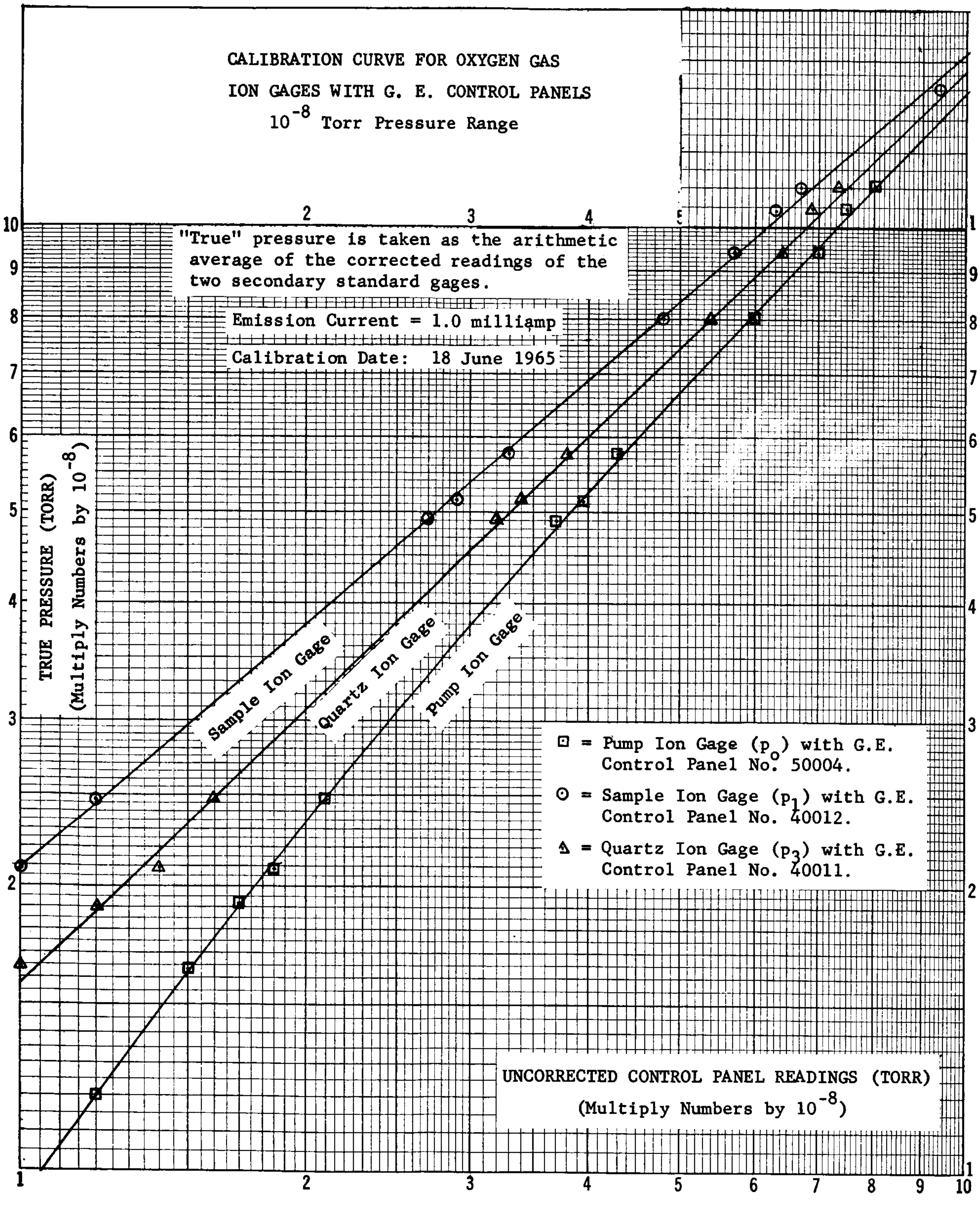


Figure 13. First Gage Calibration, G.E. Panels, 10^{-8} Range.

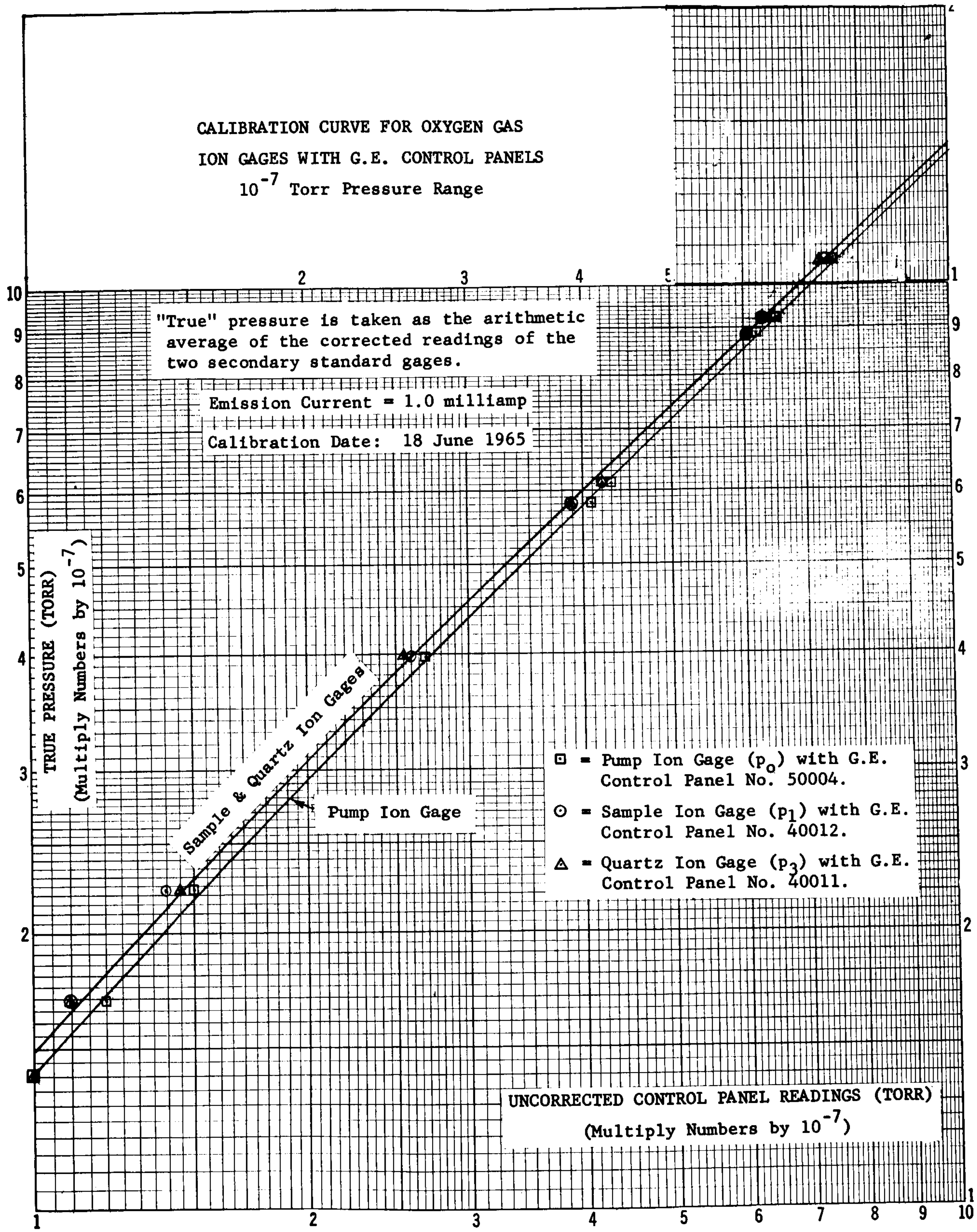


Figure 14. First Gage Calibration, G.E. Panels, 10^{-7} Range.

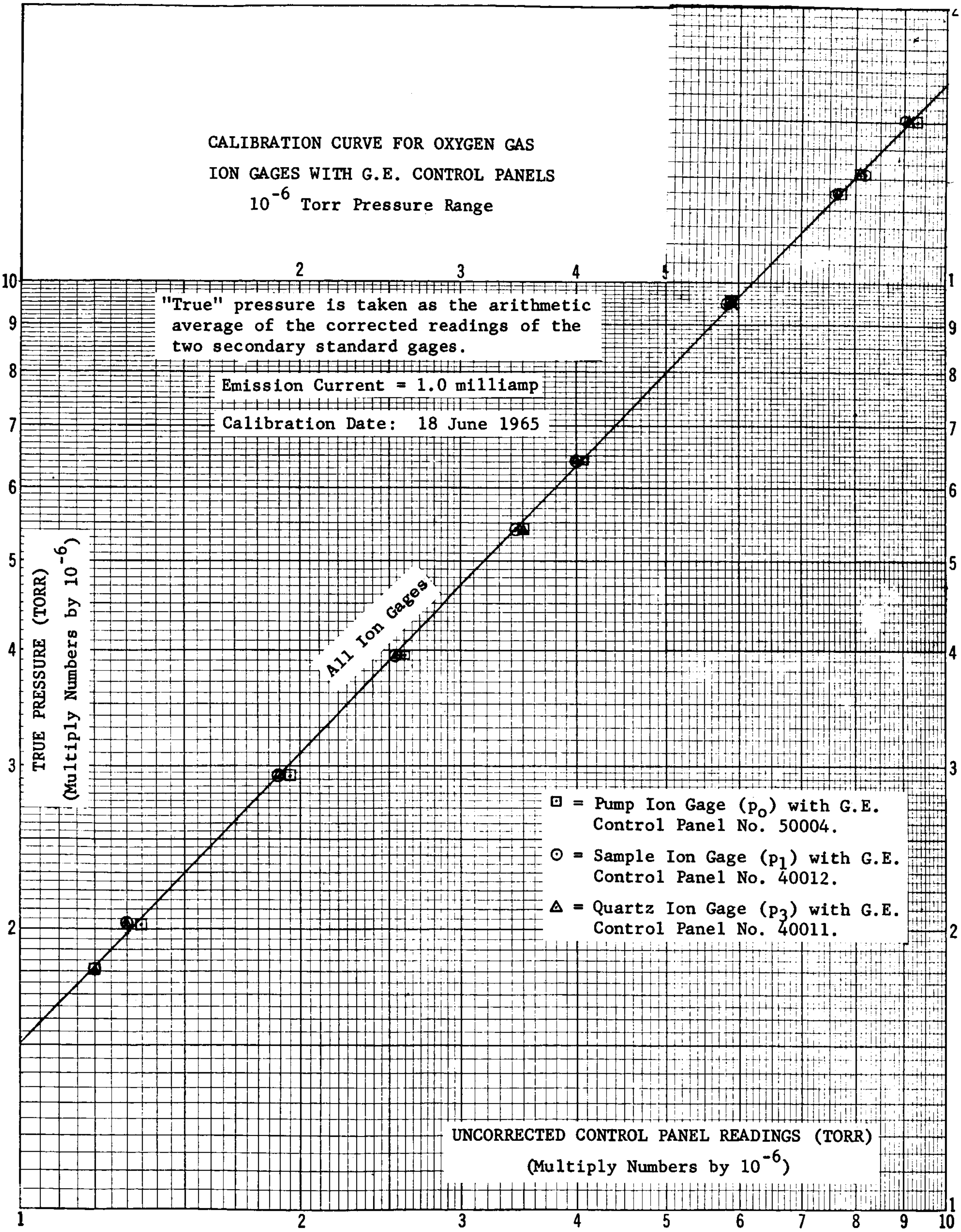


Figure 15. First Gage Calibration, G.E. Panels, 10^{-6} Range.

TABLE 22

SECOND CALIBRATION

C.V.C. Panels and G. E. Tubes which were on 10^{-8} system from
Exposures No. 1 through No. 8, and
which were on 10^{-10} system
starting with **Exposure** No. 11.

Calibration of 31 January 66
(oxygen gas)

#10 INDICATED	#10 TRUE (AVCO CALIB.)	#11 INDICATED	#11 TRUE (AVCO CALIB.)	AVG TRUE	#3016 INDICATED QUARTZ Q	#7087 INDICATED PUMP P	#3010 INDICATED SAMPLE S
7.6 (-5)	1.1 (-4)	6.1 (-5)	1.1 (-4)	1.1 (-4)	1.53 (-4)	1.8 (-4)	1.8 (-4)
4.2 "	6.5 (-5)	3.5 "	6.4 (-5)	6.45 (-5)	7.9 (-5)	9.5 (-5)	9.5 (-5)
3.1 "	4.9 "	2.6 "	4.8 "	4.85 "	5.9 "	7.0 "	7.0 "
2.2 "	3.6 "	1.9 "	3.5 "	3.55 "	4.35 "	5.15 "	5.1 "
1.4 "	2.35 "	1.2 "	2.2 "	2.27 "	2.75 "	3.4 "	3.4 "
7.6 (-6)	1.1 "	6.2 (-6)	1.05 "	1.07 "	1.35 "	1.7 "	1.7 "
9.2 "	6.4 (-6)	3.5 "	6.1 (-6)	6.2 (-6)	8.3 (-6)	1.0 "	9.2 (-6)
3.4 "	5.3 "	2.9 "	5.1 "	5.2 "	6.5 "	8.05 (-6)	7.4 "
2.4 "	3.7 "	2.0 "	3.5 "	3.6 "	4.6 "	5.65 "	5.2 "
1.7 "	2.8 "	1.4 "	2.5 "	2.65 "	3.3 "	4.05 "	3.85 "
9.3 (-7)	1.3 "	7.2 (-7)	1.35 "	1.32 "	1.85 "	2.25 "	2.15 "
6.4 "	9.3 (-7)	5.3 "	9.4 (-7)	9.35 (-7)	1.30 "	1.45 "	1.35 "
3.9 "	5.9 "	3.3 "	6.0 "	5.95 "	6.2 (-7)	9.0 (-7)	8.4 (-7)
2.8 "	4.4 "	2.4 "	4.4 "	4.4 "	5.1 "	6.6 "	6.2 "
1.9 "	3.05 "	1.7 "	3.2 "	3.12 "	3.9 "	4.75 "	4.55"
1.4 "	2.3 "	1.1 "	2.1 "	2.2 "	2.6 "	3.2 "	3.05"
9.3 (-8)	*	7.3 (-8)	*		1.8 "	2.2 "	2.05"
7.5 "	*	6.0 "	*		1.4 "	1.7 "	1.6 "
6.6 "	*	5.3 "	*		1.3 "	1.5 "	1.5 "
2.4 "	*	2.0 "	*		6.0 (-8)	6.3 (-8)	7.4 (-8)
1.6 "	*	1.4 "	*		4.2 "	4.35 "	5.35"
1.2 "	*	9.6 (-9)	*		3.15 "	3.15 "	4.75"
5.2 (-7)	7.7 (-7)	4.5 (-7)	8.1 (-7)	7.9 (-7)	10 (-7)	1.4 (-6)	1.3 (-6)
4.25 "	6.4 "	3.70 "	6.7 "	6.55 "	8.3 "	1.1 "	9.6 (-7)
3.6 "	5.5 "	3.1 "	5.65 "	5.57 "	7 "	8.6 (-7)	8.0 "
1.0 "	1.7 "	8.4 (-8)		1.7 (-7)	2.05 "	2.6 "	2.6 "
1.2 "	1.95 "	1.1 (-7)	2.1 "	2.02 "	2.45 "	3.0 "	3.0 "
1.5 "	2.45 "	1.3 "	2.45 "	2.45 "	3.0 "	3.8 "	3.8 "

Note: Pressures are in torr. Numbers in parentheses are powers of 10.

Note: Sample Ion gage has a different panel from previous calibration.

* Calibration not made.

TABLE 23

SECOND CALIBRATION

G.E. Panels & Tubes
which were on 10^{-10} system

Calibration of 12 Jan 66 (oxygen gas)

#10 INDICATED	#10 TRUE (AVCO CALIB.)	#11 INDICATED	#11 TRUE (AVCO CALIB.)	AVG TRUE	INDICATED QUARTZ Q	INDICATED PUMP P	INDICATED SAMPLE S
8.5 (-5)	1.26 (-4)	7.8 (-5)	1.42 (-4)	1.34 (-4)	8.4 (-5)	8.3 (-5)	9.3 (-5)
6.4 "	9.7 (-5)	5.7 "	1.05 "	1.01 "	6.0 "	5.9 "	6.6 "
4.4 "	6.8 "	3.7 "	6.8 (-5)	6.8 (-5)	4.0 "	3.95 "	4.45 "
1.9 "	3.1 "	1.7 "	3.1 "	3.1 "	1.85 "	1.80 "	2.0 "
1.2 "	2.1 "	9.0 (-6)	1.6 "	1.85 "	9.65 (-6)	9.6 (-6)	1.05 "
8.1 (-6)	1.1 "	6.7 "	1.15 "	1.12 "	7.3 "	7.25 "	8.1 (-6)
5.9 "	8.8 (-6)	5.1 "	8.8 (-6)	8.8 (-6)	5.35 "	5.3 "	5.95 "
3.55 "	5.5 "	3.15 "	5.5 "	5.5 "	3.25 "	3.2 "	3.6 "
1.8 "	2.9 "	1.6 "	2.8 "	2.85 "	1.6 "	1.6 "	1.8 "
9.3 (-7)	1.33 "	7.95 (-7)	1.38 "	1.35 "	8.15 (-7)	8.45 (-7)	9.3 (-7)
7.8 "	1.12 "	6.8 "	1.2 "	1.16 "	6.9 "	7.1 "	8.0 "
5.95 "	8.8 (-7)	5.3 "	9.5 (-7)	9.15 (-7)	5.25 "	5.4 "	6.1 "
3.8 "	5.8 "	3.4 "	6.2 "	6.0 "	3.4 "	3.5 "	3.95 "
1.9 "	3.05 "	1.7 "	2.8 "	2.92 "	1.7 "	1.75 "	2.0 "
1.05 "	1.75 "	8.5 (-8)	*		9.65 (-8)	9.4 (-8)	1.1 "
8.3 (-8)	*	6.7 "	*		7.4 "	7.2 "	8.2 (-8)
4.6 "	*	3.75 "	*		4.4 "	4.15 "	4.7 "
2.7 "	*	2.2 "	*		3.1 "	2.55 "	3.1 "
1.5 "	*	1.3 "	*		1.7 "	1.55 "	1.7 "

Note: Pressures are in torr. Numbers in parentheses are powers of 10.

* Calibration not made.

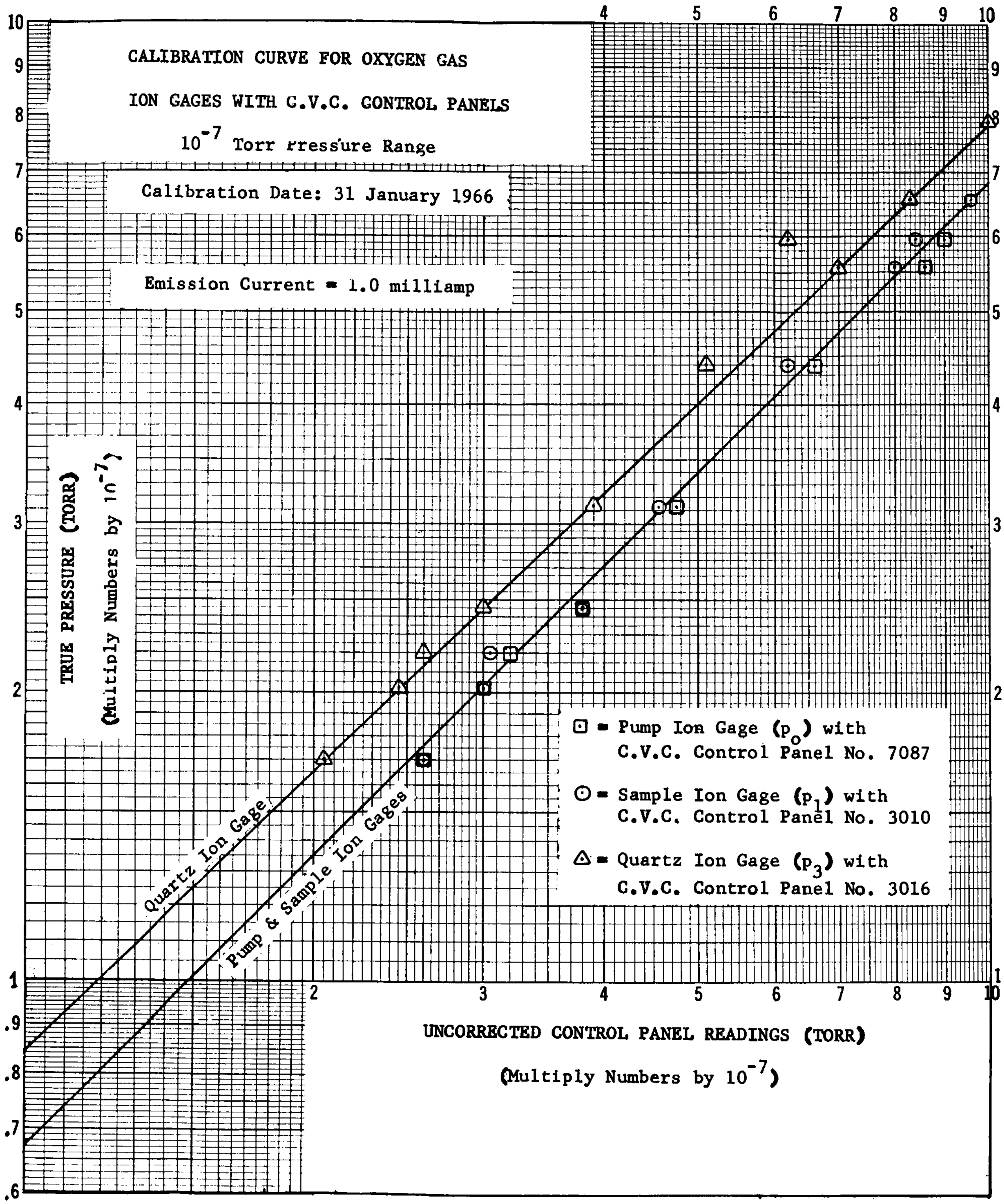


Figure 16. Second Gage Calibration, C.V.C. Panels, 10^{-7} Range.

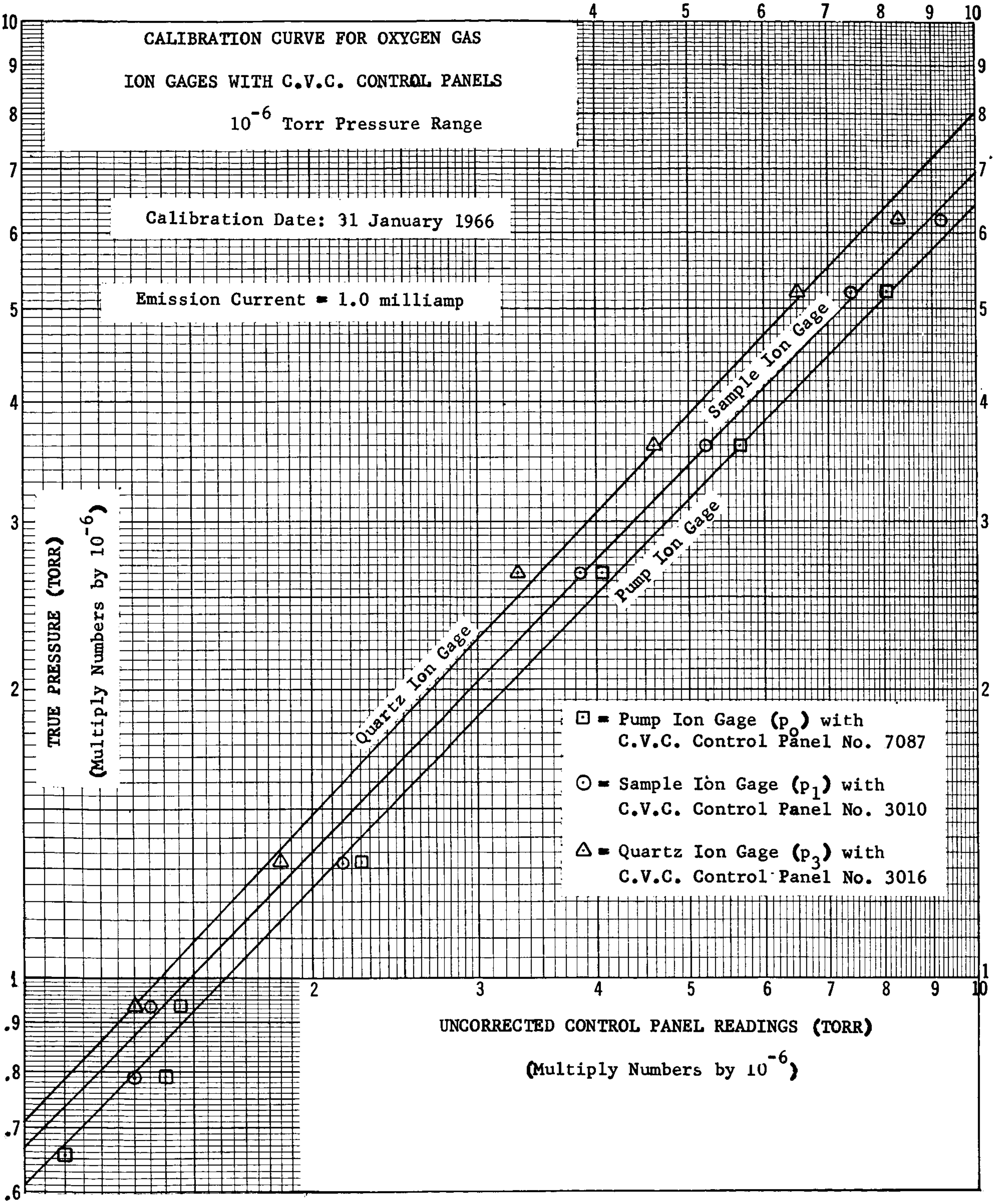


Figure 17. Second Gage Calibration, C.V.C. Panels, 10^{-6} Range.

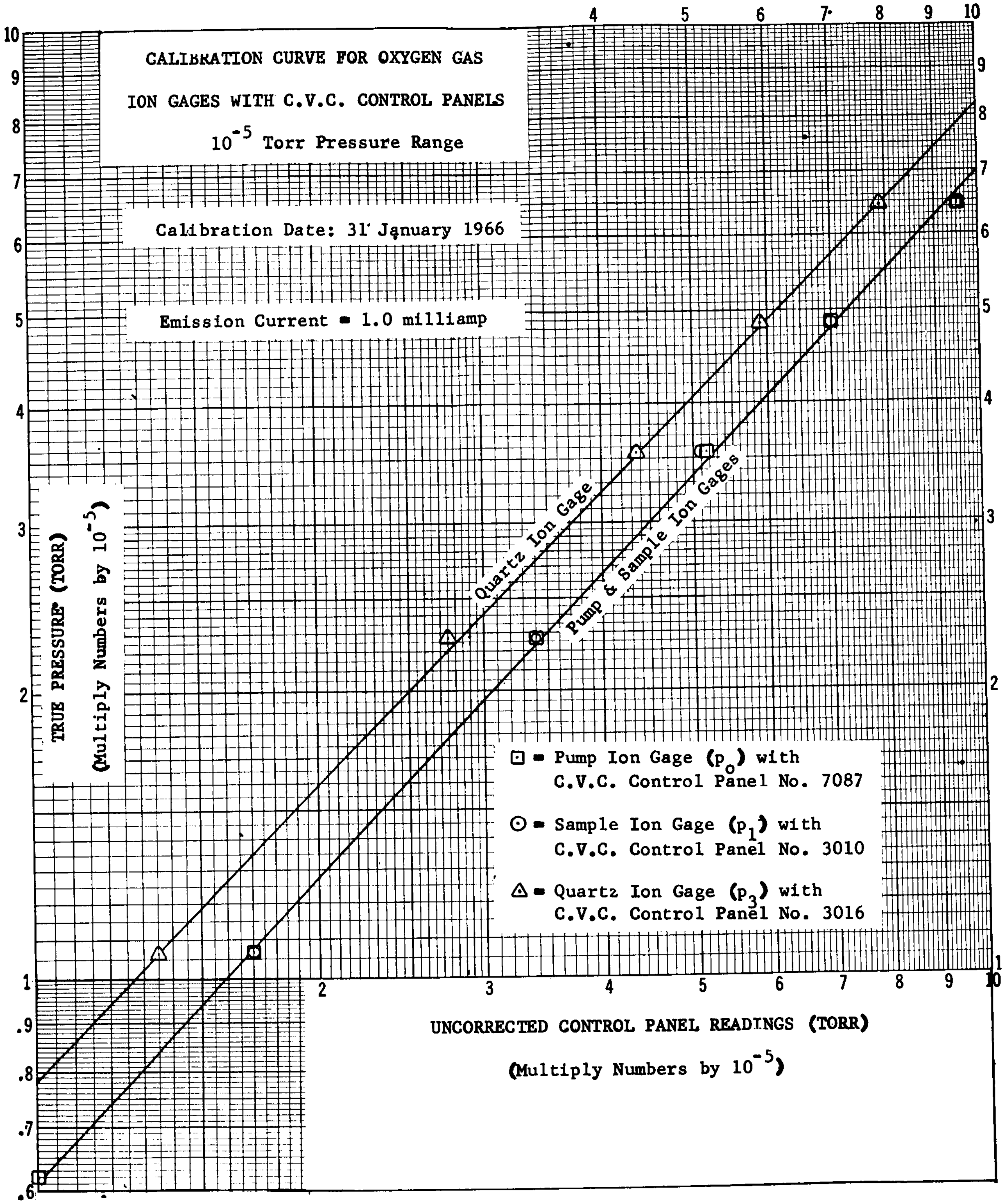


Figure 18. Second Gage Calibration, C.V.C. Panels, 10^{-5} Range.

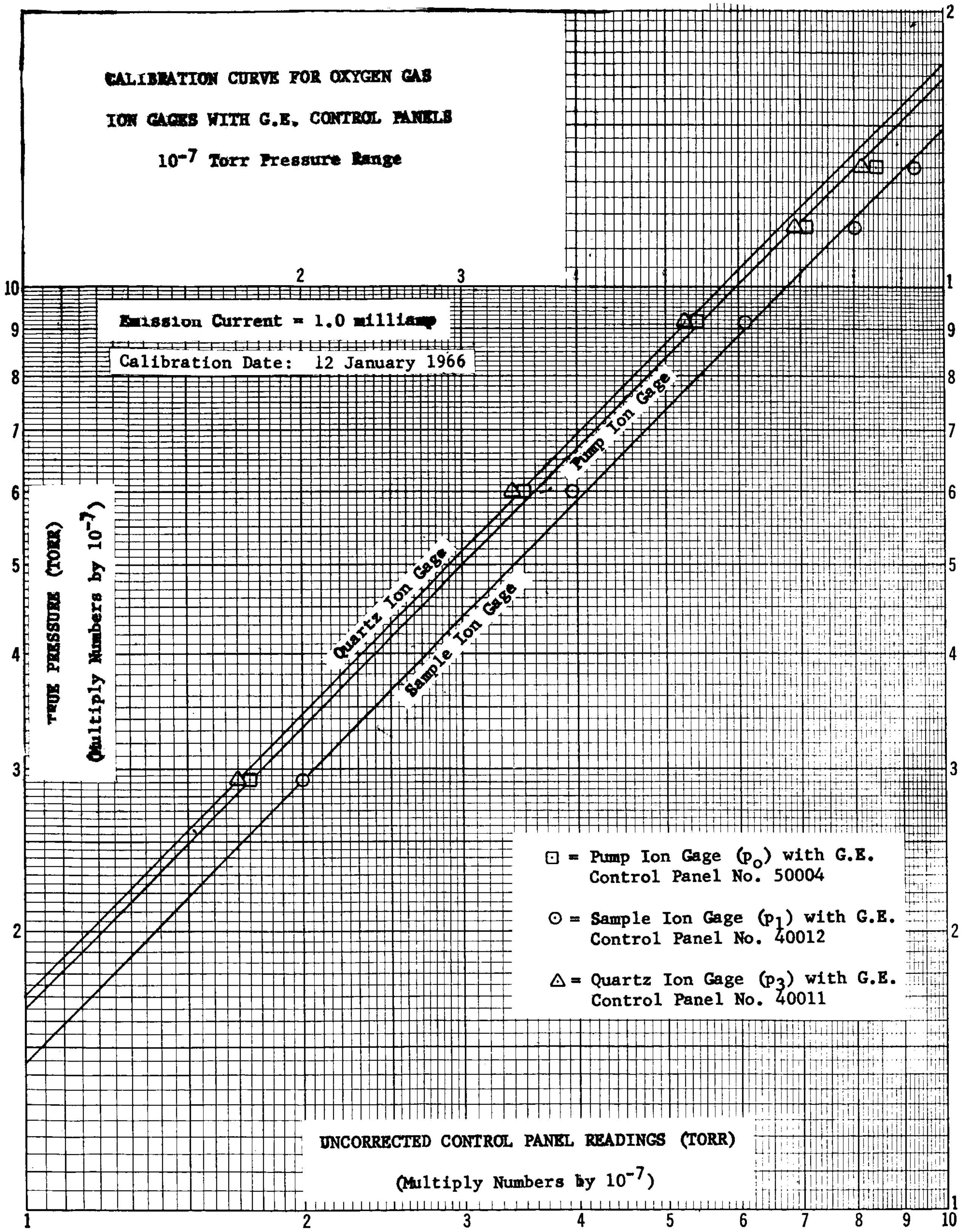


Figure 19. Second Gage Calibration, G.E. Panels, 10^{-7} Range.

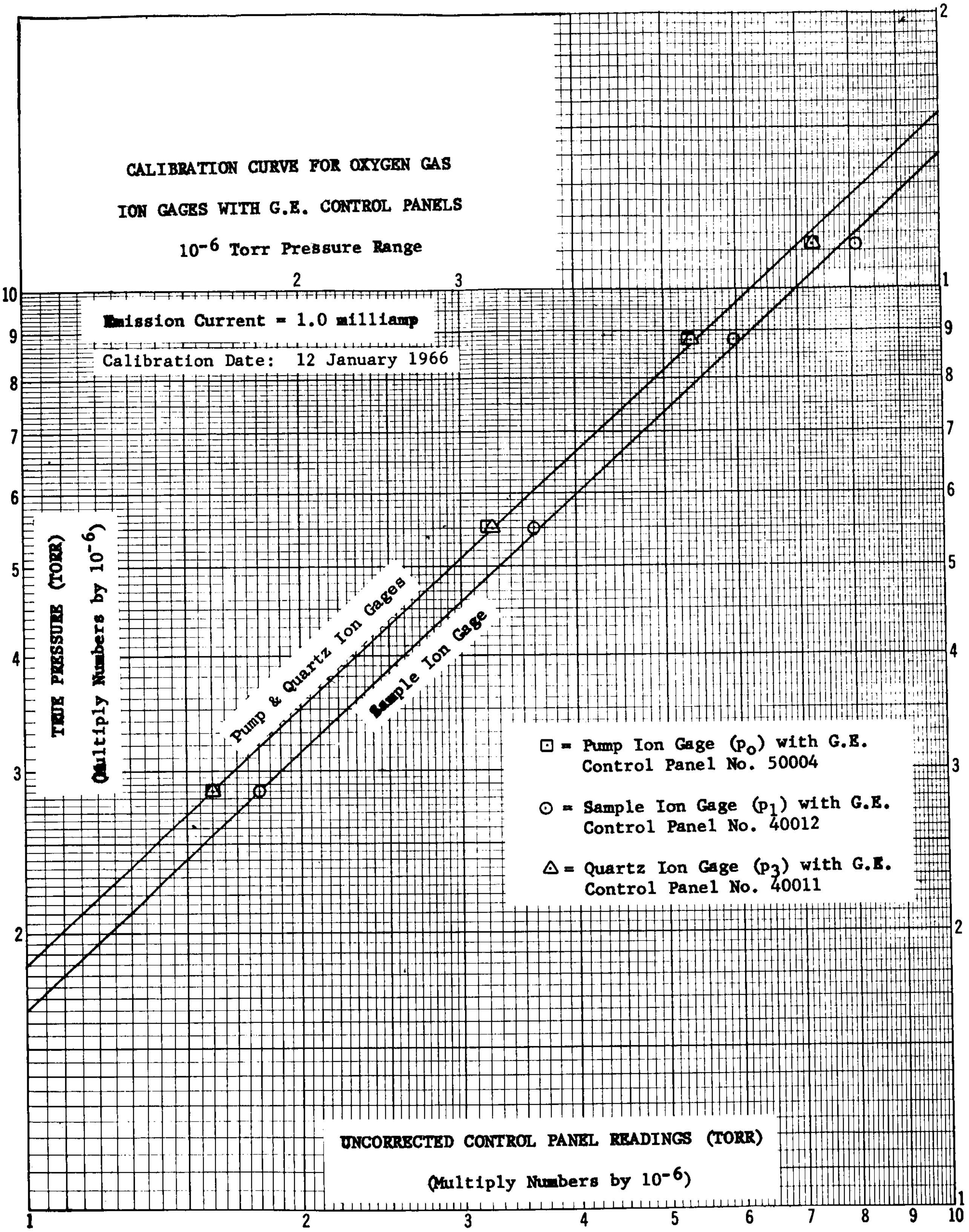


Figure 20. Second Gage Calibration, G.E. Panels, 10^{-6} Range.

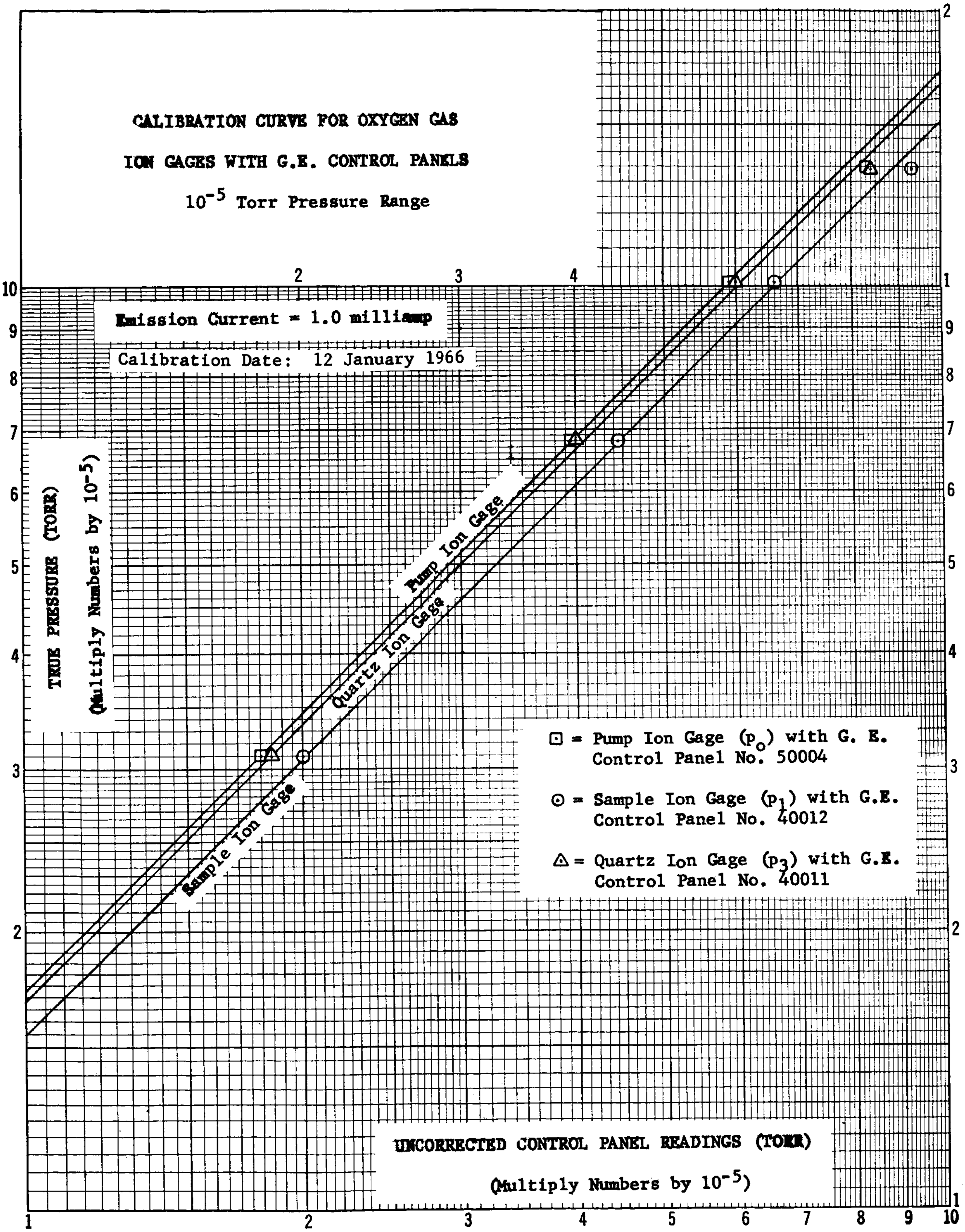
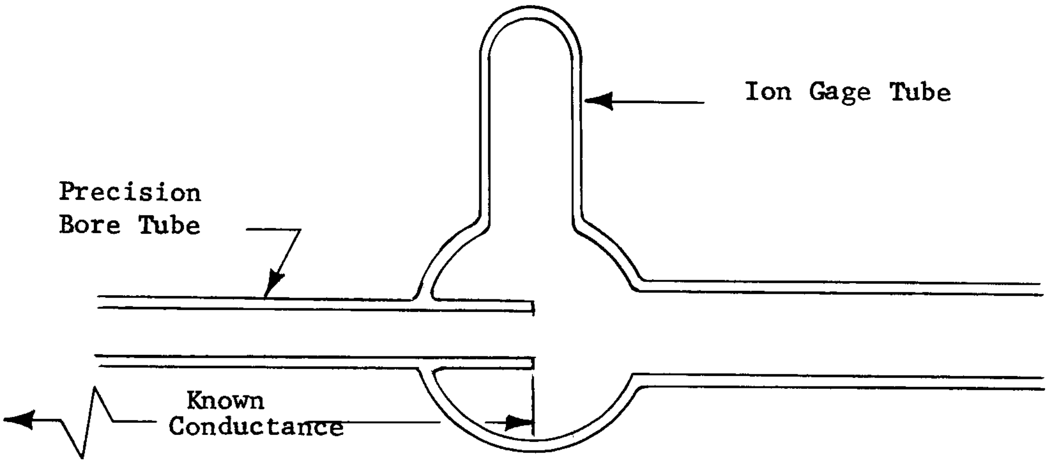


Figure 21. Second Gage Calibration, G.E. Panels, 10^{-5} Range.

9. APPENDIX

9-1. Calculation of System Conductance

The conductance between the two flow-measuring gages consisted of a specially-prepared length of precision bore tubing. In order to avoid possible uncertainties in conductance at the ends, the ends were prepared as shown below.



Thus, the precision bore was carried all the way to the end of the conductance, where there was an abrupt end with sharp edges. The conductance of this cylindrically-shaped passage can be easily calculated by standard formulas which have been proven experimentally.

In addition, it was desired to avoid the possibility of any deviation from the cosine distribution for molecules leaving the walls, as the standard formulas are based upon this distribution. It has been reported that some small deviations from cosine have been observed with certain molecules on glass(1). The inside wall of the conductance was, therefore, roughened by grit blasting to produce a frosty appearance. The grit blasting removes only a small amount of glass (about 0.01 mm), and it is believed that the roughened surface will assure a cosine distribution.

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(1) B. C. Moore, "Measurement of Vacuum in Nonuniform Temperature Environments", Journal of Vacuum Science and Technology, Vol. 1, Sept/Oct, 1964, p. 10-16.

The calculation of conductance is as follows. The most accurate equation takes into account the "Clausing effect" and can be written(1)

$$C_o = B(T_o/M)^{\frac{1}{2}}$$

where

$$B = 3.638\ K\ \frac{\pi D_o^2}{4}$$

For definition of symbols, see Section 2, "Nomenclature".

There were three different conductances used in the program. One was used on the 10^{-8} system for the first exposure only; after that exposure the conductance tube cracked and was replaced. The second conductance tube was installed and used for the remainder of exposures on the 10^{-8} system. The third conductance tube was installed on the 10^{-10} system and used for all exposures on that system.

An outline of the various dimensions and results of calculations is given below:

Conductance Tube No.	Length, L_o	Diameter, D_o	Clausing Factor, K	Constant, B
1	18.0 cm.	0.9845 cm.	0.0675	0.187
2	18.0 cm.	1.012 cm.	0.0694	0.203
3	16.8 cm.	0.9368 cm.	0.0687	0.172

The conductance, C_o, can be calculated for a given exposure from a knowledge of the constant B, the conductance temperature for that particular exposure, T_o, and the molecular weight which for all exposures was 32 (oxygen).

Values of B and C_o for each exposure are given in Section 6.

9-2. Analysis of Pressure Distribution

The installation of the third ion gage (identified as the "quartz" gage) measuring p_3 in the system, provided an additional means for estimating

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(1) S. Dushman and J. M. Lafferty, "Scientific Foundations of Vacuum Technique", Second Edition, John Wiley and Sons, 1962, p. 93.

the pressure directly over the specimen. In earlier experiments, the specimen pressure (p_2) was estimated by knowing the pressure of the "sample" gage, p_1, calculating the pressure drop through the tube between the sample gage and the specimen, and applying a thermal transpiration correction. In predicting specimen pressure (p_2) from p_3, it should be necessary to use only a thermal transpiration correction as there should be no net flow of gas between the specimen and the p_3 gage.

Unfortunately, there has generally been a very large discrepancy between the specimen pressure as predicted by the sample gage (p_1) and the quartz gage (p_3) calculations. For this reason a detailed analysis of the flows and pressures was undertaken. Such an analysis was presented in the last Final Report (period of 22 January 1964 to 22 January 1965, NASA Contract NAS 3-4169). An improved analysis will be presented herein.

First, the flow rate, Q, is defined by $Q = p\dot{V}$. Since the perfect gas law gives

$$p\dot{V} = \frac{\dot{m}RT}{M} \quad ,$$

the flow rate can be expressed as

$$Q = \frac{\dot{m}RT}{M} \tag{1}$$

Along a tube with <u>axial</u> variations in temperature, the <u>mass</u> flow rate ($\dot{m}$) remains a constant regardless of the temperature. However, the flow rate, Q, varies with temperature. If Q_o = the flow rate where the temperature is T_o, and Q_n = the flow rate where the temperature is T_n,

$$Q_n = \frac{\dot{m}RT_n}{M} = \frac{\dot{m}RT_o}{M} \times \frac{T_n}{T_o}$$

or,

$$Q_n = Q_o \frac{T_n}{T_o} \tag{2}$$

An important assumption, which was justified in the Final Report referenced above, is that gas, as it flows in molecular flow through a tube, immediately comes to the temperature of the wall at any cross-section. (This assumption is based upon the fact that most molecules bounce back and forth between tube walls, and only slowly travel down the tube in the direction of net flow).

As a result of this assumption, a thermal transpiration relationship can be applied across the edge of the furnace, from pressures p_A to p_B (see sketch in Section 2, "Nomenclature"). This relationship can be written(1):

$$(p_A/p_B) = (T_1/T_2)^{1/2} \qquad (3)$$

The following two equations can be written from the definition of conductance, C:

$$Q_1 = C_1 \quad (p_1 - p_A) \qquad (4)$$

$$Q_2 = C_2 \quad (p_B - p_2) \qquad (5)$$

Equations (3), (4), and (5) can be combined, eliminating p_A and p_B to give:

$$p_2 = \left(\frac{T_2}{T_1}\right)^{1/2} p_1 - \frac{Q_1}{C_1}\left(\frac{T_2}{T_1}\right)^{1/2} - \frac{Q_2}{C_2} \qquad (6)$$

Equation (2) can now be used to put $Q_1$and Q_2 in Equation (6) in terms of Q_o to yield

$$p_2 = \left(\frac{T_2}{T_1}\right)^{1/2} p_1 - \frac{Q_o(T_1T_2)^{1/2}}{C_1\ T_o} - \frac{Q_o\ T_2}{C_2\ T_o} \qquad (7)$$

This equation gives the predicted pressure directly over the specimen as a function of the flow rate, temperatures, conductances, and p_1.

The conductance of a tube in molecular flow is given by the following equations(2):

$$C_1 = 3.81\ \frac{D^3}{L_1}\ \left(\frac{T_1}{M}\right)^{1/2} \qquad (8)$$

$$C_2 = 3.81\ \frac{D^3}{L_2}\ \left(\frac{T_2}{M}\right)^{1/2} \qquad (9)$$

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(1) S. Dushman and J. M. Lafferty, "Scientific Foundations of Vacuum Technique", Second Edition, John Wiley and Sons, 1962, p. 59.

(2) Ibid. p. 88.

If Equations 8 and 9 are substituted into 7, there results finally:

$$p_2 = p_1 \left(\frac{T_2}{T_1} \right)^{1/2} - \frac{Q_o L \; (MT_2)^{1/2}}{3.81 \; D^3 \; T_o} \tag{10}$$

where D is the diameter of the quartz tube containing the specimen, and:

$$Q_o = C_o(p_o - p_1) \tag{11}$$

and where, for the 10^{-10} system

$$C_o = 0.172 \; (T_o/M)^{1/2} \tag{12}$$

Thus, the pressure over the specimen can be predicted from a knowledge of p_o, p_1, the various temperatures, and the system geometry.(1).

On the other hand, the pressure p_2 can be obtained from p_3 by a simple thermal transpiration correction:

$$p_2 = p_3 \left(\frac{T_2}{T_3} \right)^{1/2} \tag{13}$$

These equations will now be applied to the data of Exposure 11. The values assumed are:

T_o = 313°K (40°C)

T_1 = 313°K (40°C)

T_2 = 1253°K (980°C)

T_3 = 313°K (40°C)

D = 2.76 cm

L = 36 cm

Pressures are in torr, and numbers in parentheses are powers of ten:

---

(1) The pressure over the specimen was assumed to be that existing at the center of the specimen. No attempt is made here to account for the presence of the specimen itself on the pressure distribution.

Phase	p_o	p_1	p_3	p_2 from Eqn. 10	p_2 from Eqn. 13
2	5.9(-5)	5.0(-6)	5.8(-7)	1.66(-6)	1.16(-6)
3	1.0(-5)	1.08(-6)	1.3(-7)	7.80(-7)	2.6 (-7)
4	5.1(-6)	4.8 (-7)	8.4(-8)	2.45(-7)	1.68(-7)
5	1.94(-6)	2.2(-7)	4.3(-8)	1.74(-7)	0.86(-7)
7	6.7(-7)	8.4(-8)	2.7(-8)	7.74(-8)	5.4 (-8)
8	3.2(-7)	5.0(-8)	1.75(-8)	5.83(-8)	3.5 (-8)
9	1.5(-7)	3.9(-8)	1.48(-8)	6.08(-8)	2.96(-8)

It is apparent that predictions of p_2 from Equation 10 tend to be higher than those predicted from Equation 13, by a factor of from 1.6 to 3.0, but generally around 2.0. This is considerably better than results of last year, possibly because of the larger diameter of quartz tube in this year's apparatus, reducing the pressure drop between p_1 and p_2.

9-3. Calibration of Secondary Standard Gages

In December, 1964, the two secondary standard gages were sent to the AVCO Corporation, Wilmington, Massachusetts, for recalibration. Calibrations were obtained with both oxygen and nitrogen, in case nitrogen would be used later in the program.

The calibrations were obtained in the 10^{-7}, 10^{-6} and 10^{-5} torr ranges. The low 10^{-7} torr pressure was as low as the AVCO apparatus would permit. The estimated limit of error was ± 10%.

Results are presented in Figures 22, 23, and 24. One secondary standard gage tube is designated No. 10, and the other is No. 11. Each gage tube was always used only with its individual control panel. In order to reduce the possibility of inaccuracies, the secondary standard gage tubes and control panels were used only for calibration of the system gages, and not for any other purpose.

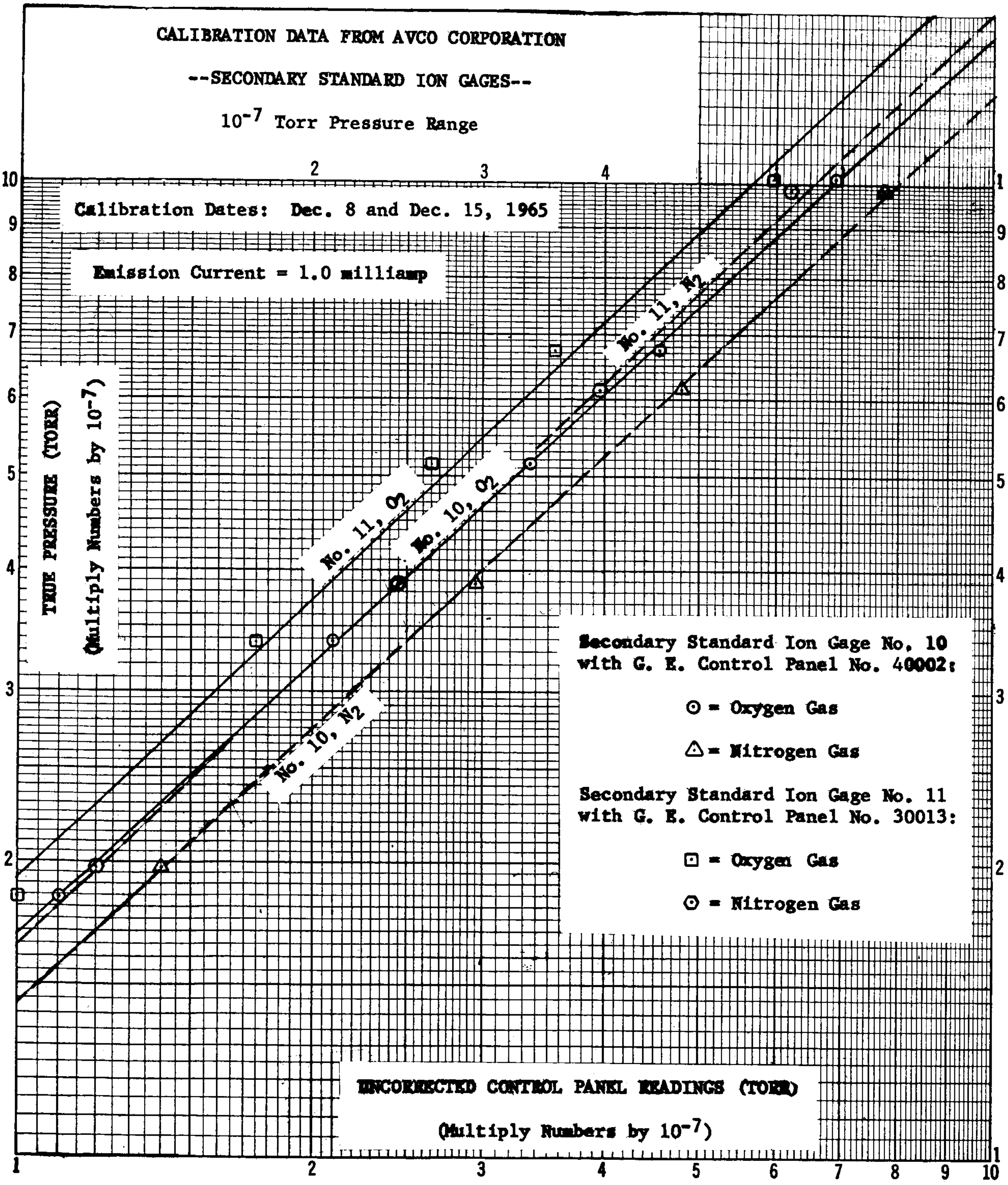

Figure 22. Secondary Standard Calibration, 10^{-7} Range.

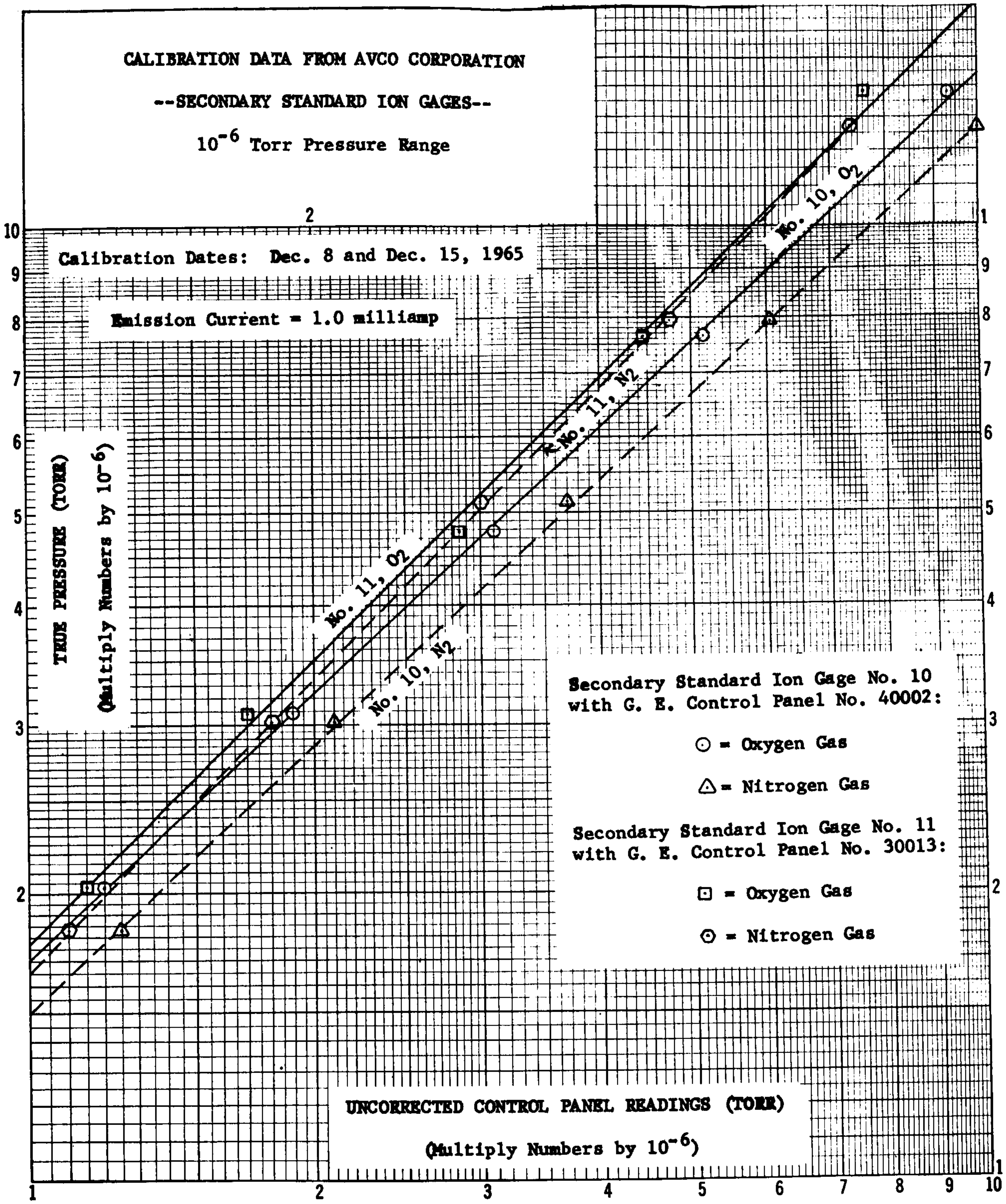

Figure 23. Secondary Standard Calibration, 10^{-6} Range.

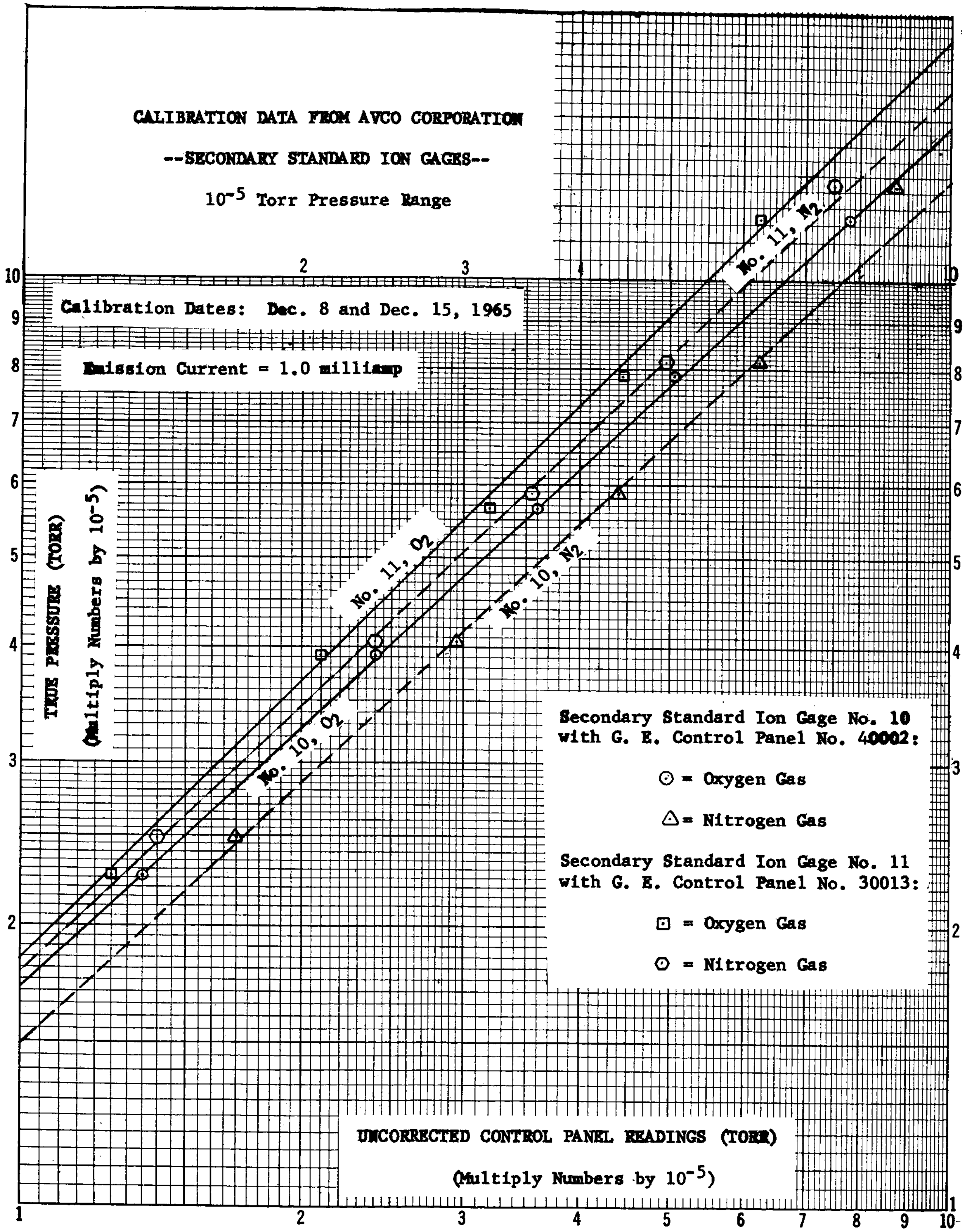

Figure 24. Secondary Standard Calibration, 10^{-5} Range.

9-4. List of Previous Reports

The previous reports on this subject which were prepared by the General Electric Company for the National Aeronautics and Space Administration are listed below.

1. Contamination Exposures of Cb-1Zr Specimens, Final Report for NASA Contract NAS 3-1903, Covering the Period Sept. 8, 1961 to June 22, 1962.
2. Contamination Exposures of Cb-1Zr Specimens, Final Report for NASA Contract NAS 3-1903, Covering the Period Jan. 2, 1963 to July 3, 1963, Rept. No. GE63FPD306.
3. Contamination Exposures of Columbium and Cb-1Zr Alloy Specimens, Final Report for NASA Contract NAS 3-4169 Covering the Period Jan. 22, 1964 to Jan. 22, 1965, Rept. No. DMS65-2.

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