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# Potassium Corrosion Test Loop Development
## Topical Report No. 1

# PURIFICATION AND ANALYSIS OF HELIUM FOR THE WELDING CHAMBER

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T. F. Lyon

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Contract NAS 3-2547

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

# POTASSIUM CORROSION TEST LOOP DEVELOPMENT

## TOPICAL REPORT No. 1

# PURIFICATION AND ANALYSIS OF HELIUM FOR THE WELDING CHAMBER

Prepared By
T. F. Lyon

Approved By
J. W. Semmel, Jr.
Manager, Materials and Processes

Prepared For
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Lewis Research Center

Under Contract NAS 3-2547

July 1, 1965

Technical Management

NASA - Lewis Research Center

T. A. Moss and R. L. Davies

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

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# FOREWORD

The work described herein was performed by the General Electric Company under the sponsorship of the National Aeronautics and Space Administration under Contract NAS 3-2547. The purpose was to purify and analyze the helium environment used in welding refractory alloy components for evaluation in a boiling and condensing potassium corrosion test loop.

Dr. T. F. Lyon conducted the experimental work with the assistance of Mr. W. L. Hasty. Dr. R. B. Hand, Manager, Chemistry and Physics, was responsible for the preliminary design of the mass spectrometer system and continuously lent his advice and assistance. Mr. W. R. Young, Manager, Joining and Fabrication, and Mr. P. A. Blanz contributed to the welding aspects of the experimental investigation. This work was administered for the General Electric Company by Dr. J. W. Semmel, Jr., Manager, Materials and Processes. Mr. E. E. Hoffman, Manager, Corrosion Technology, acts as Program Manager of the Potassium Corrosion Test Loop Development Program which will evaluate the material performance. Messieurs T. A. Moss and R. L. Davies are the Technical Managers for the National Aeronautics and Space Administration.

# I. INTRODUCTION

A helium analysis system has been assembled and used to monitor impurities in a vacuum purged, inert gas welding chamber. The analytical system consists of a mass spectrometer and electrolytic hygrometer to monitor the chamber gas and a trace oxygen analyzer and electrolytic hygrometer to detect impurities in the inlet helium. The mass spectrometer is a magnetic deflection instrument with a Nier type of electron bombardment ion source and electron multiplier detector. This type of spectrometer is most frequently used for the analysis of residual gases in an ultrahigh vacuum system.

After design, construction, and initial operation of the mass spectrometer it was soon obvious that reliable analyses for water vapor could not be obtained. Since water vapor was expected to be one of the chief contaminants within the welding chamber, the electrolytic hygrometer system was installed on the welding chamber and utilized for the water analyses.

The impurities in the welding chamber (other than water vapor) of main interest have been oxygen, nitrogen and hydrogen. The mass spectrometer is calibrated for these three gases and the reliable detection limit for each of these is estimated to be between 1 and 2 volume ppm. Qualitative analysis of other impurities is also obtained.

Special cylinder helium has been obtained for use in this program. Further purification of this helium using a molecular sieve dryer and hot titanium furnace has produced inlet gas to the welding chamber having impurity levels consistently below the detection limits of the mass spectrometer. A commercially available trace oxygen analyzer and an electrolytic hygrometer have been used for oxygen and moisture analysis of the inlet helium.

This report describes the layout and operation of the mass spectrometer and other analytical instruments used. Typical impurity levels encountered in the welding chamber and changes in the impurity levels are given.

# II. MASS SPECTROMETER SYSTEM

## A. Apparatus

The mass spectrometer analyzer tube (General Electric Model 22PT110) consists of a Nier type of electron bombardment ion source, a 90-degree sector magnetic analyzer with a 5-cm radius of curvature, and 10-stage electron multiplier detector. The tube envelope is of Type 304L stainless steel with ceramic-metal electrical feedthroughs. The magnetic field is supplied by a 3-kilogauss bakeable permanent magnet (General Electric Model 22PM103). The fixed magnetic field with electrostatic scanning provides adjacent peak separation from 2 to 50 AMU (atomic mass units) which covers the range of the most likely impurities in helium.

Figure 1 is a layout of the mass spectrometer system. The sample gas enters the system through the variable leak valve (Granville-Phillips Series 203) which is used to control the pressure in the system. The system pressure is measured with a Bayard - Alpert type ionization gauge (General Electric Model 22GT104). An ultrahigh vacuum valve is used to isolate the analytical portion of the system from the pumps and traps. The pumping system consists of a glass mercury diffusion pump rated at 80 liters per second which is separated from the analytical system by two liquid nitrogen cooled traps, one glass and one metal. Three stainless steel bellows, located as shown in Figure 1, prevent excessive stress on the components. A molecular sieve trap between the mechanical forepump and the diffusion pump protects the system from backstreaming oil vapor. The foreline pressure is monitored by a thermocouple gauge.

Figure 2 is a photograph of the analytical portion of the system made during initial assembly. The ionization gauge had not yet been attached to the system. As shown in Figure 2, the components are attached with appropriate brackets to a stainless steel baseplate.

That portion of the system shown in Figure 2 may be enclosed with a bakeout oven capable of temperatures as high as 450°C. An overall view of the system with the bakeout oven in place is shown in Figure 3. The metal cold trap is provided with a heating mantle for bakeout. Electronic controls for the system are located on the cart at the left. The recorder is used to monitor the temperature of the various components during bakeout.

With proper precautions, a pressure of 2 to 3 x $10^{-10}$ torr is consistently obtained as indicated by the ionization gauge. However, mass spectra obtained on the residual gases in this system indicate that the true base pressure is in the mid-$10^{-11}$ torr range as will be shown later in this report. The ionization gauge indicates a higher pressure, probably due to being operated near to the x-ray limit which is near 1 x $10^{-10}$ torr, according to the manufacturer.

At the present time, the metal liquid nitrogen trap is not used because adequately low pressures are obtained with only the glass trap cooled. Contamination of the system by condensible vapors such as $H_2O$, $CO_2$, or Hg can usually be quickly eliminated by cooling of the metal trap. However, subsequent warming of this trap to room temperature results in the evolution of fairly large quantities of these vapors back into the system.

The ionization gauge itself has been found to be a source of carbon monoxide and hydrocarbons apparently arising either from the elements of the gauge or from reactions with residual gases in the system. To minimize the quantity of these species on the background spectra, analyses are performed with the ionization gauge off.

The thoria-coated iridium filament in the spectrometer is used and operated with 0.5 ma. emission current and 50 volts ionizing potential. The

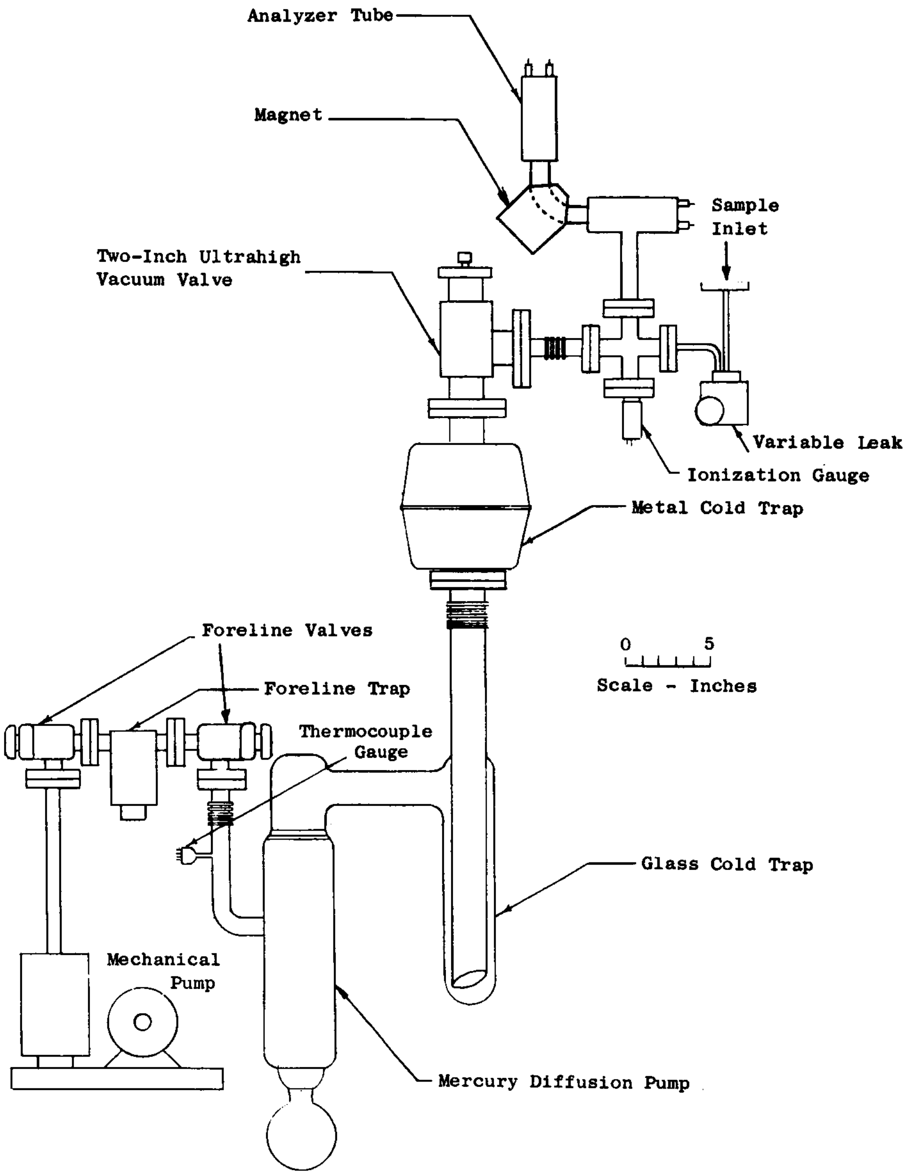


Figure 1. Mass Spectrometer System.

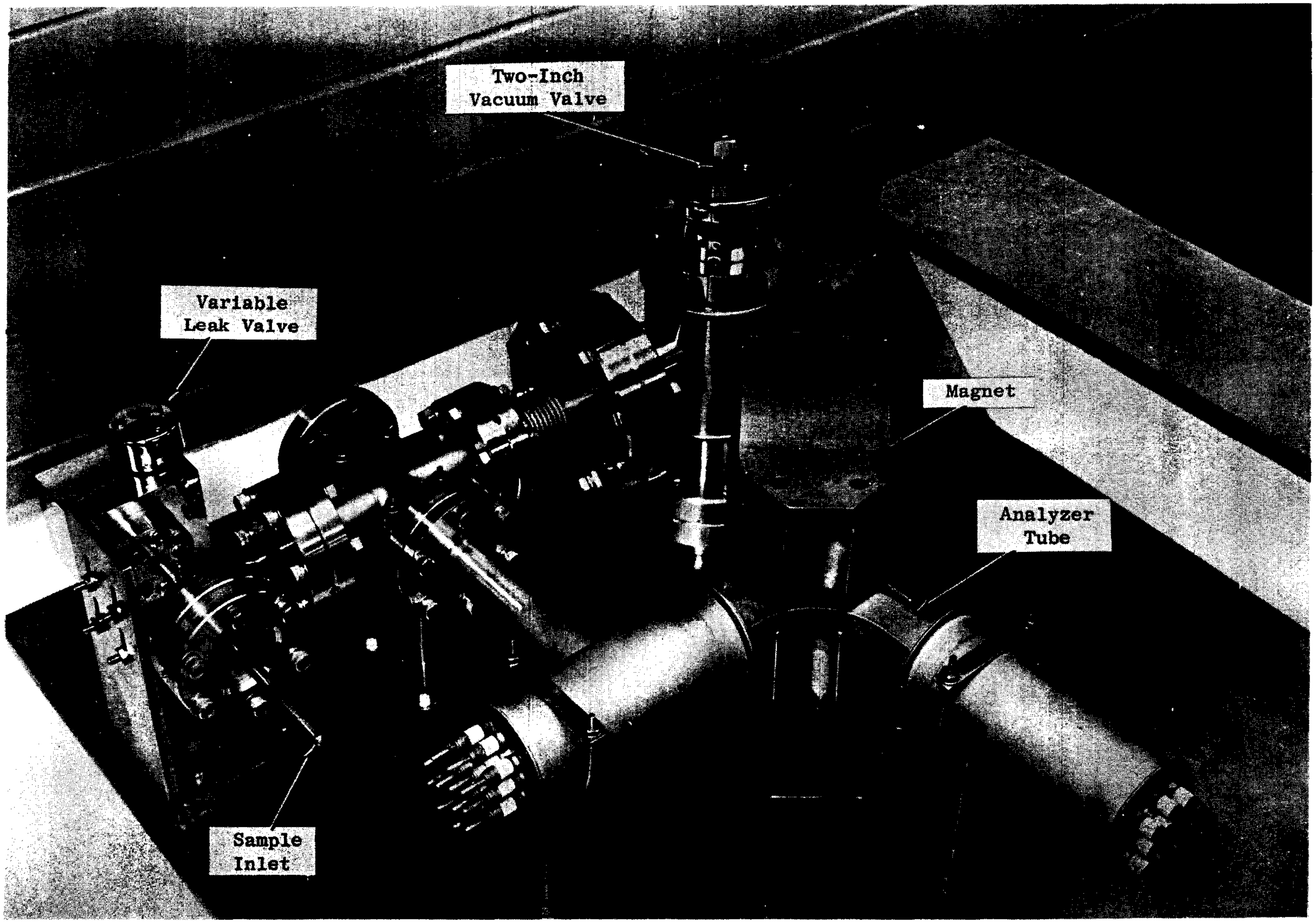


Figure 2. Mass Spectrometer Tube and a Portion of the Vacuum System.

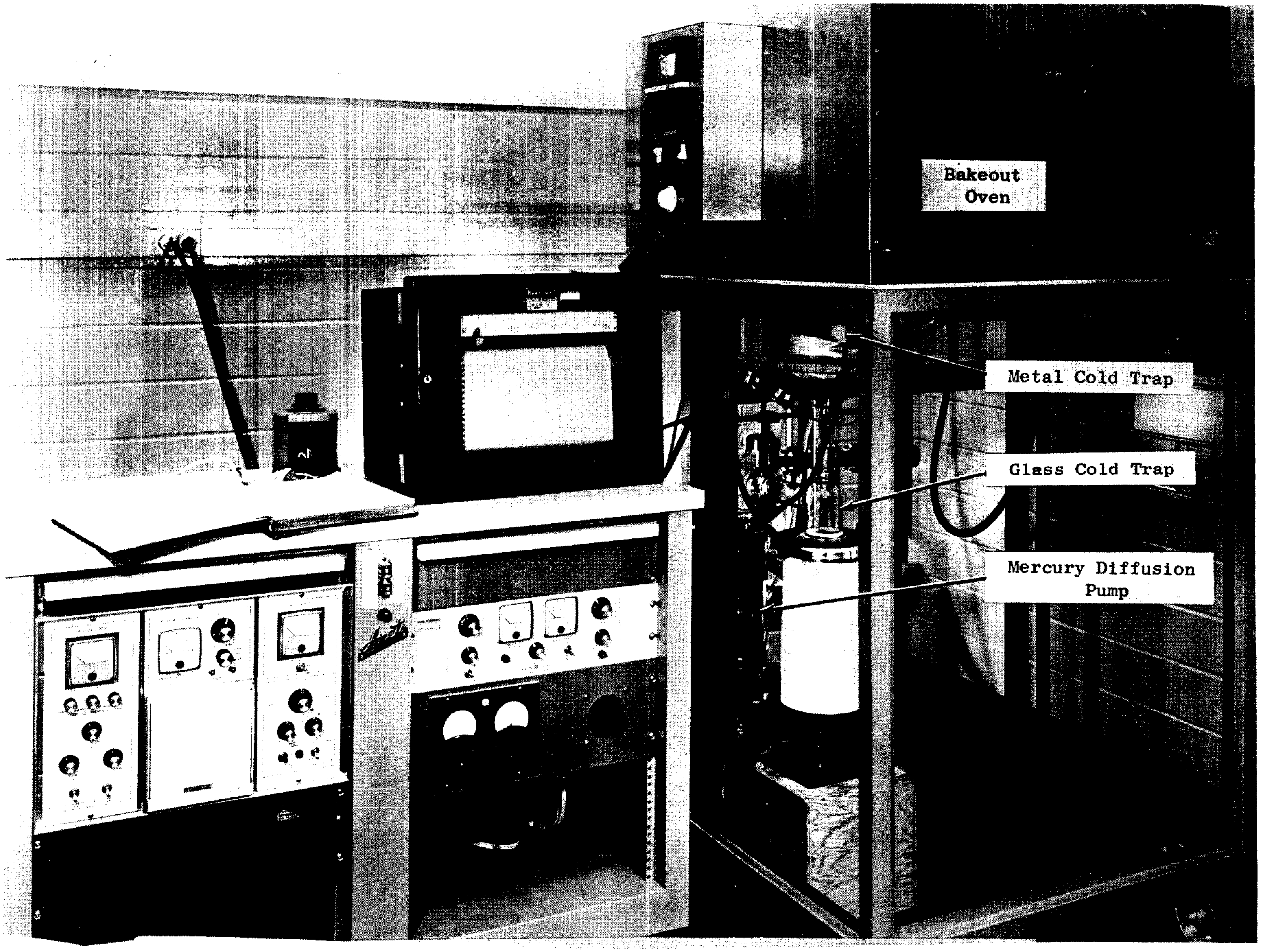


Figure 3. Mass Spectrometer System with Bakeout Oven in Place.

filament is operated continuously at these conditions because considerable gas is liberated when the filament power is first applied after a prolonged period with the filament at ambient temperature. The electron multiplier is operated at 1550 volts.

Mass spectra are recorded on 8 1/2 x 11-inch paper utilizing a moving pen recorder (Houston Instrument Model HR-96T) with time base on the x-axis. The high mass range from 45 to 12 AMU is focused at accelerating potentials between 200 and 800 volts. This range is scanned in approximately 10 minutes. The low mass species, helium at 4 AMU and hydrogen at 2 AMU, are focused with accelerating potentials of about 2300 and 4600 volts respectively and require an additional 5 minutes. It thus requires about 15 minutes to record the total mass spectrum.

## B. Calibration and Data Reduction

The original plan for the analytical system was to calibrate the mass spectrometer against pressures of various pure gases within the system by introducing the pure gases and noting the mass spectrometer ion current produced for a given pressure of the gas as indicated by the system ionization gauge. Some analyses early in the program were actually performed by this method but it soon became apparent that the results were seriously in error due principally to two factors. The first is that oxygen is apparently lost on introduction into the vacuum system. Possible causes for the oxygen loss are adsorption on the clean walls of the system or reaction of the oxygen with the elements of the ion gauge or the spectrometer tube. The result of this was that oxygen analyses were obtained which were less than the true value by a factor of about ten.

The second factor affecting the measurements was associated with the hydrogen analyses. It was found that the mass spectrometer ion current at 2 AMU was a function of both the total gas pressure and of the partial pressure of hydrogen. The reason for this behavior is not completely clear at this time although it may be connected with the liberation of hydrogen at some point in the spectrometer tube due to local heating by the ion beam. The result of this behavior was that hydrogen concentrations calculated from the pure gas calibration were 10 to 20 ppm greater than the true value.

Although this early procedure gave erroneous results for oxygen and hydrogen, analyses for the more inert gases such as argon, neon and nitrogen were at least reasonable.

The response of the mass spectrometer which was obtained for calibration with pure gases shows the very strong mass discrimination which can occur with electrostatic scanning when a fixed magnetic field is employed. Figure 4 shows the results of the pure gas calibration for the four gases $H_2$, He, $N_2$ and A for which the parent peaks occur at mass numbers 2, 4, 28 and 40 respectively. This figure is a semi-logarithmic plot of the ratio of the mass spectrometer ion current to the pure gas pressure as indicated by the ionization gauge, plotted against mass number. Since differences in ionization efficiency affect the mass spectrometer ion source and the ionization gauge to roughly the same extent, this plot reflects the change in response due to mass differences alone. As may be seen from Figure 4, the sensitivity

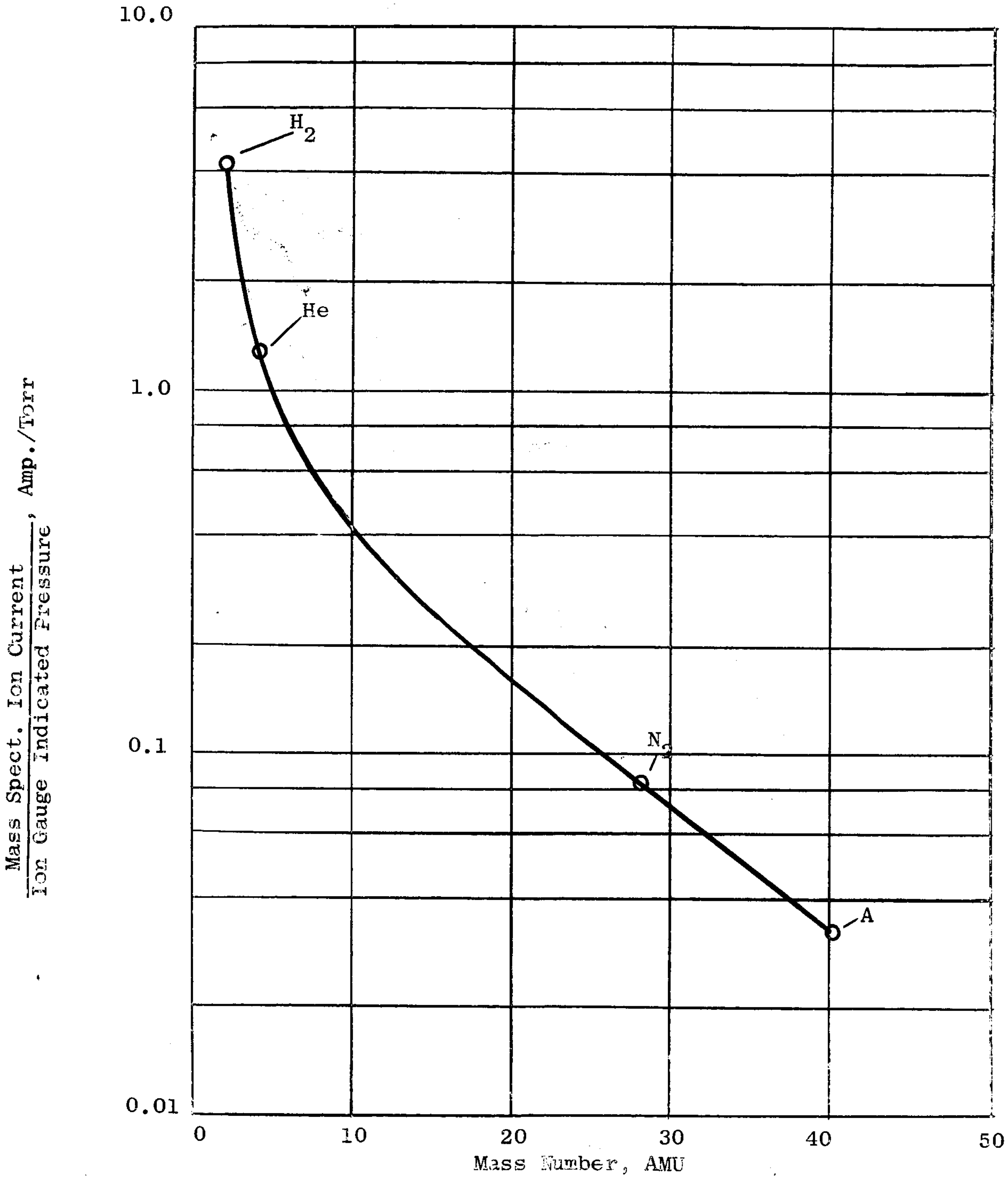


Figure 4. Sensitivity of Mass Spectrometer (Amp. per Torr) Plotted Against Mass Number Showing Strong Mass Discrimination.

of the mass spectrometer to hydrogen is about a factor of 130 greater than the sensitivity to argon. This is due mainly to the large difference in accelerating voltage required to focus the different masses at the detector; 4600 volts for hydrogen as compared to about 220 volts for argon. Much better secondary emission of the more energetic ions occurs at the first dynode of the electron multiplier and, in addition, better withdrawal and focusing of ions occurs in the ion source and better transmission efficiency through the magnetic analyzer is obtained at the higher ion accelerating voltages.

The attempt to obtain reliable analyses by the pure gas calibration procedure has been abandoned due mainly to the difficulty in obtaining reliable analyses for oxygen and hydrogen by this technique, and also due to the rather time consuming procedure involved in the calibration.

An alternate procedure, in which the mass spectrometer is calibrated against helium of known impurity content, has been found to give quite accurate analyses down to impurity levels as low as a few ppm. A "background" mass spectrum is first obtained using a sample of ultrahigh purity helium in which the concentration of each impurity of interest is effectively zero. A spectrum of the calibrating gas is then obtained. The sensitivity factor for each impurity is then obtained from the increase in ion current in the calibrating gas spectrum above that of the pure gas spectrum.

Thus if I is the ion current due to a particular impurity in the calibrating gas, $I_o$ is the ion current at the same mass number for the pure helium, and $I_{He}$ is the ion current for helium in the calibrating gas spectrum, then the sensitivity factor is determined as the increase in the ratio $\frac{I - I_o}{I_{He}}$ per part per million impurity in the calibrating gas. The sensitivity factor thus obtained is used to calculate the various impurity concentrations from the unknown sample spectrum.

Analyses of the ultrahigh purity helium and the calibrating mixture were supplied by the vendor and these analyses are shown in Table I. Analysis of the calibrating mixture for oxygen has also been obtained here using a sensitive Brady apparatus. The result is in very good agreement with the vendor analysis and is given in the last column of Table I.

Use of the above described procedure eliminates the difficulties encountered in the pure gas calibration method. An additional advantage is that the calibration can be performed fairly rapidly. The system is usually calibrated once each day and, in certain instances, both before and after analysis of the unknown samples.

## C. Sampling System

The sampling system for the mass spectrometer is arranged so that helium may be introduced from the inlet line to the welding chamber, from

## TABLE I

## ANALYSES OF ULTRAPURE HELIUM AND CALIBRATING MIXTURE

| | Ultrapure Helium Vendor Analysis, ppm | Calibrating Mixture Vendor Analysis, ppm | Calibrating Mixture Brady Analysis, ppm |
|---|---|---|---|
| $O_2$ | 0.1 | 17 | 18.3 |
| $N_2$ | 0.3 | 12 | |
| $H_2$ | 0.2 | 20 | |
| CO | 0.0 | 15 | |
| $CO_2$ | 0.1 | ND | |
| Ne | 10.9 | ND | |
| A | 0.0 | ND | |
| $CH_4$ | 0.0 | ND | |
| $H_2O$ | 2.5 | ND | |
| He | Balance | Balance | |

ND indicates not detected

the chamber itself, or from either the calibrating gas cylinder or the cylinder of ultrapure helium. A schematic drawing of the sample system is shown in Figure 5. Valves used in this system are of all metal construction, either of the diaphragm or bellows-seal type. Most of the connections are welded or brazed and, where the lines must be uncoupled, Swagelok connections are used. A detail drawing of the inlet to the variable leak valve is also shown in Figure 5. This construction is such that the sample flushes past the valve seat and returns through an annular space.

The sample system is evacuated by a mechanical pump to about 1 micron pressure before the sample is introduced. The sample system is maintained under vacuum when not in use. A molecular sieve trap prevents back diffusion of oil vapor from the pump and a thermocouple gauge is used to monitor the sample line pressure. The sample gas is allowed to flow through the system for several minutes at a rate of about 150 cc per minute in order to flush out traces of contaminants not removed by the pump. Two feet of small-bore tubing between the sample inlet and the flowmeter prevents back diffusion of air from the outlet of the flowmeter.

## D. Performance of the Mass Spectrometer

A mass spectrum of the residual gases in the mass spectrometer system, obtained with the ionization gauge off is shown in Figure 6. The major gaseous species present are $H_2$ (2 AMU) at about $1.6 \times 10^{-11}$ torr; CO (28 AMU) at about $1.5 \times 10^{-11}$ torr; and $CO_2$ (44 AMU) at about $1.0 \times 10^{-11}$ torr. Smaller amounts of water vapor (18 AMU) and helium (4 AMU) are also detectable. These partial pressures are calculated from the calibration curve shown in Figure 4. The total pressure calculated from the sum of the partial presures is $4.4 \times 10^{-11}$ torr. In Figure 6 the peaks at 16.3 and 19.3 AMU probably do not arise from gaseous species within the system. The origin of these peaks has been discussed by Davis (1) who concludes that their most probable source is the ionization of surface impurities by electrons in the ion source region. This seems to be quite a reasonable explanation because the peaks do not occur at integral mass numbers and, in addition, do not vary appreciably with the total pressure in the system. The peak at 16.3 AMU is said to be due to $O^+$ ions from absorbed CO and the peak at 19.3, to $F^+$ ions. For a considerable period after first putting the mass spectrometer into operation, such spurious peaks due to $Na^+$, $Cl^+$ and $K^+$ ions were also observed. These peaks have since disappeared and the $F^+$ peak has also decreased considerably.

These spurious peaks generally cause no difficulty in the interpretation of the mass spectrum. However, in most cases, the 16.3 peak masks the peak at 16 AMU due to $CH_4$ and $O^+$ from $O_2$ and $CO_2$.

Figure 7 shows the mass spectrum of the ultrahigh purity helium used for background in the calibration procedure. The vendor analysis of this helium has been given in Table I. This spectrum was also obtained with the ionization gauge off. The helium pressure is estimated from the data of Figure 4 to be about $1.1 \times 10^{-6}$ torr indicated pressure on the ionization

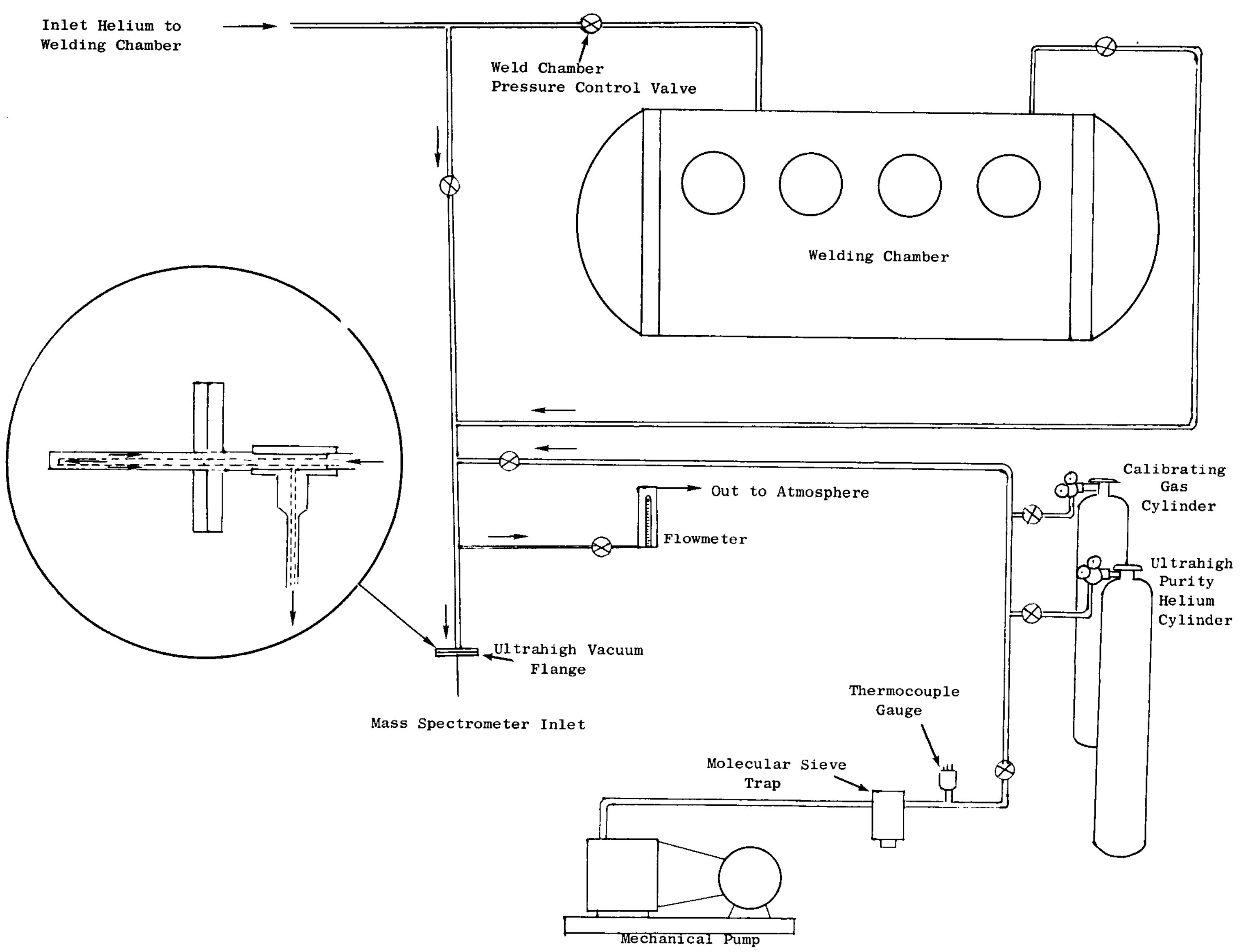


Figure 5. Sampling System for Mass Spectrometer. Inset shows Detail of Sample Inlet Flow.

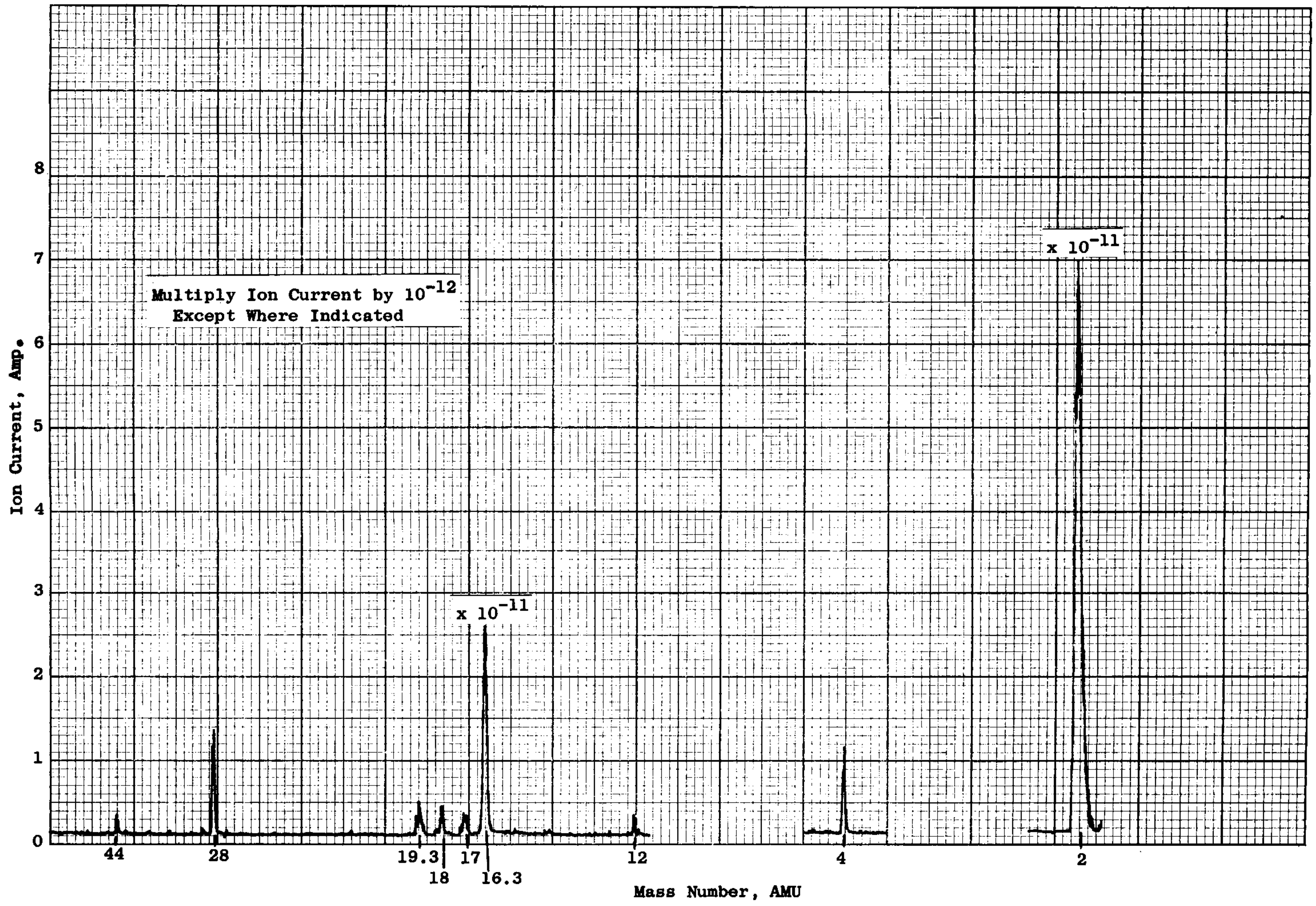


Figure 6. Residual Gas Mass Spectrum. Total Pressure is about 4 x $10^{-11}$ Torr.

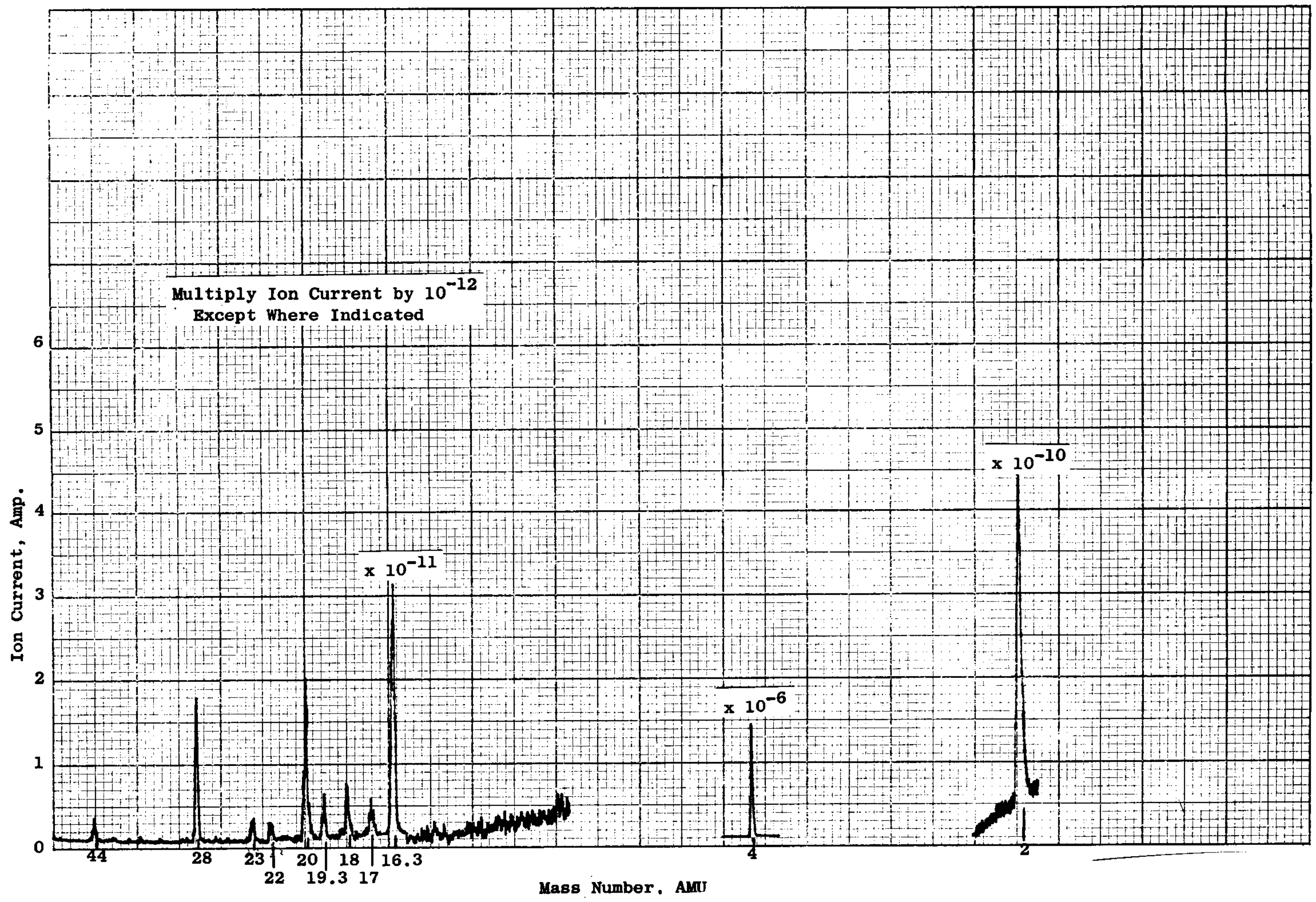


Figure 7. Mass Spectrum of Ultrahigh Purity Helium.

gauge. This corresponds to a true helium pressure of about 5.6 x $10^{-6}$ torr. Several peaks occur in the ultrapure helium spectrum which are not detectable on the residual gas spectrum shown in Figure 6. The peak at mass number 20 is due to $Ne^+$ and the peak at 22 is due to the neon isotope. It may be noted that the ratio of the ion currents at 22 AMU to that at 20 AMU is the same as the ratio of the isotopic abundances. The peak at 23 AMU is probably due to $Na^+$. A slight increase in all peaks over that obtained in the residual gas spectrum may also be noted. The rather drastic increase in the hydrogen peaks (2 AMU) is an indication of the dependence of this peak on total pressure as has been mentioned previously. An increase in the base line signal and also the noise on the trace may be noted starting at about 20 AMU and becoming more severe at the lower mass numbers as the accelerating voltage increases. The peaks at 12 to 15 AMU are practically lost in the noise at this signal level.

Figure 8 is a mass spectrum of the calibrating mixture, the analysis of which is shown in Table I. The sensitivity to the various impurities may be noted by comparing the ion currents with those at the corresponding mass number in Figure 7. The rather low sensitivity to oxygen (32 AMU) is apparent. The sensitivity factor for nitrogen is obtained from the increase in the peak at 14 AMU due to $N^+$. Contribution to this peak from $CO^{++}$ and $CH_2^+$ is assumed to be negligible. In Figure 8, ion currents due to $Ne^+$ (20 AMU) and $A^+$ (40 AMU) are also present. Concentrations of these inert gases were not reported in the analysis shown in Table I.

The accuracy of helium analysis by the presently used procedure depends mainly on three factors; (1) the linearity of response of the mass spectrometer, (2) the reproducibility of the analyses and (3) the accuracy of the analysis of the calibrating mixture. Some consideration of these three factors follows.

For linear response, the increase in ion current over the background ion current at a particular mass number should be proportional to the concentration of the impurity in the helium and also proportional to the total helium pressure within the ion source region. In Figure 9, the oxygen ion current is plotted against helium ion current for a series of mass spectra obtained with the calibration mixture at various pressures within the ion source. These data show the linearity of the oxygen peak with helium pressure over the range of helium pressures used for the analyses. It is thus demonstrated that proportionality is obtained at least up to an oxygen concentration of 17 ppm which is the concentration of oxygen in the calibrating mixture. It is known, however, that at much higher oxygen concentrations, nonlinearity exists and the result is an apparent increase in sensitivity to oxygen. Apparently, this is due to the fact that when the system is very clean, a small quantity of oxygen introduced may be partially lost within the system by some mechanism. When a larger quantity of oxygen is admitted, partial saturation occurs with a consequent increase in oxygen sensitivity. At times, it has been found useful to introduce air at about $10^{-8}$ torr pressure and allow the system to soak for about one hour. This process usually results in an increase in oxygen sensitivity of a factor of at least two which will persist for a period of weeks.

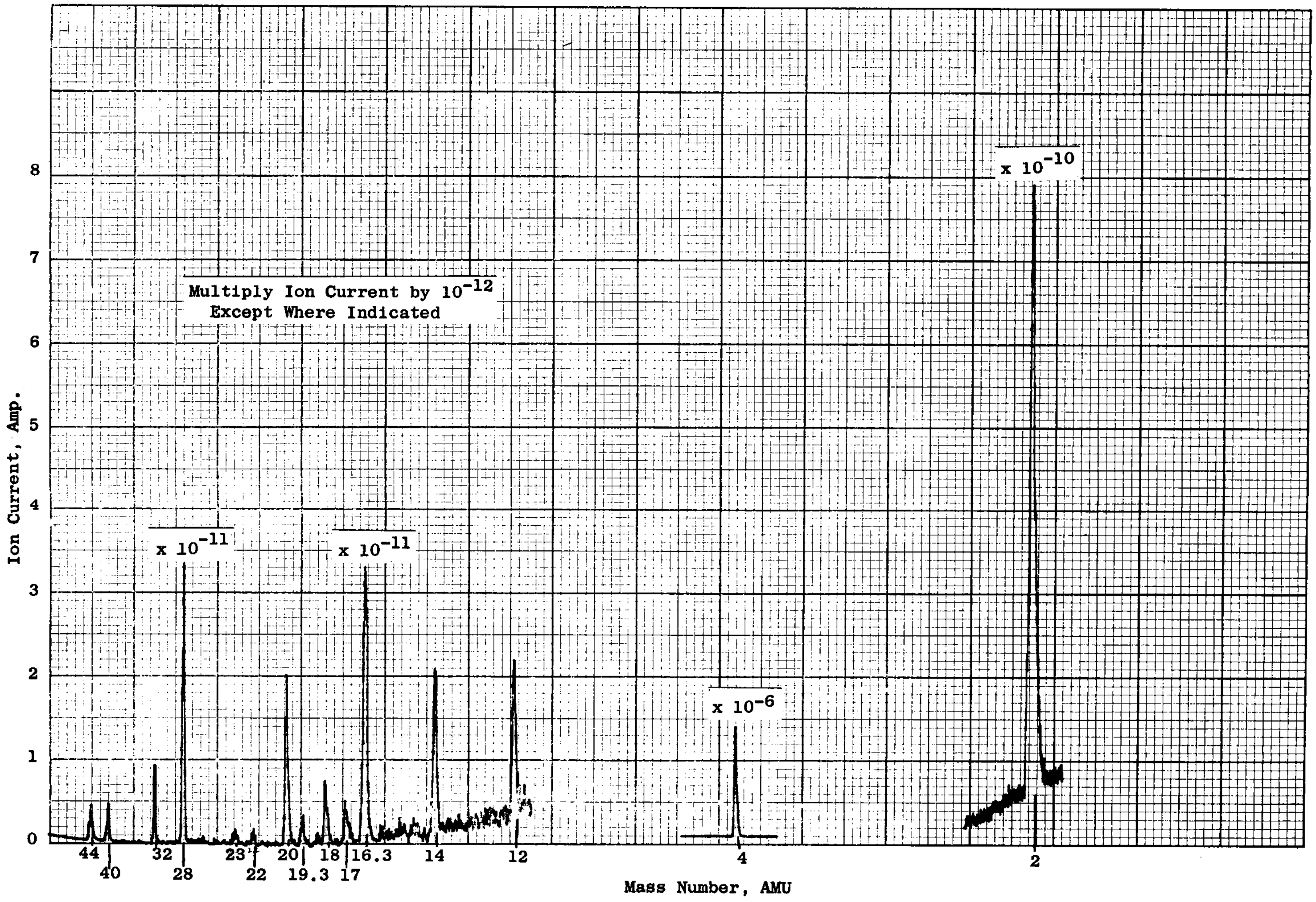


Figure 8. Mass Spectrum of the Calibrating Gas Mixture.

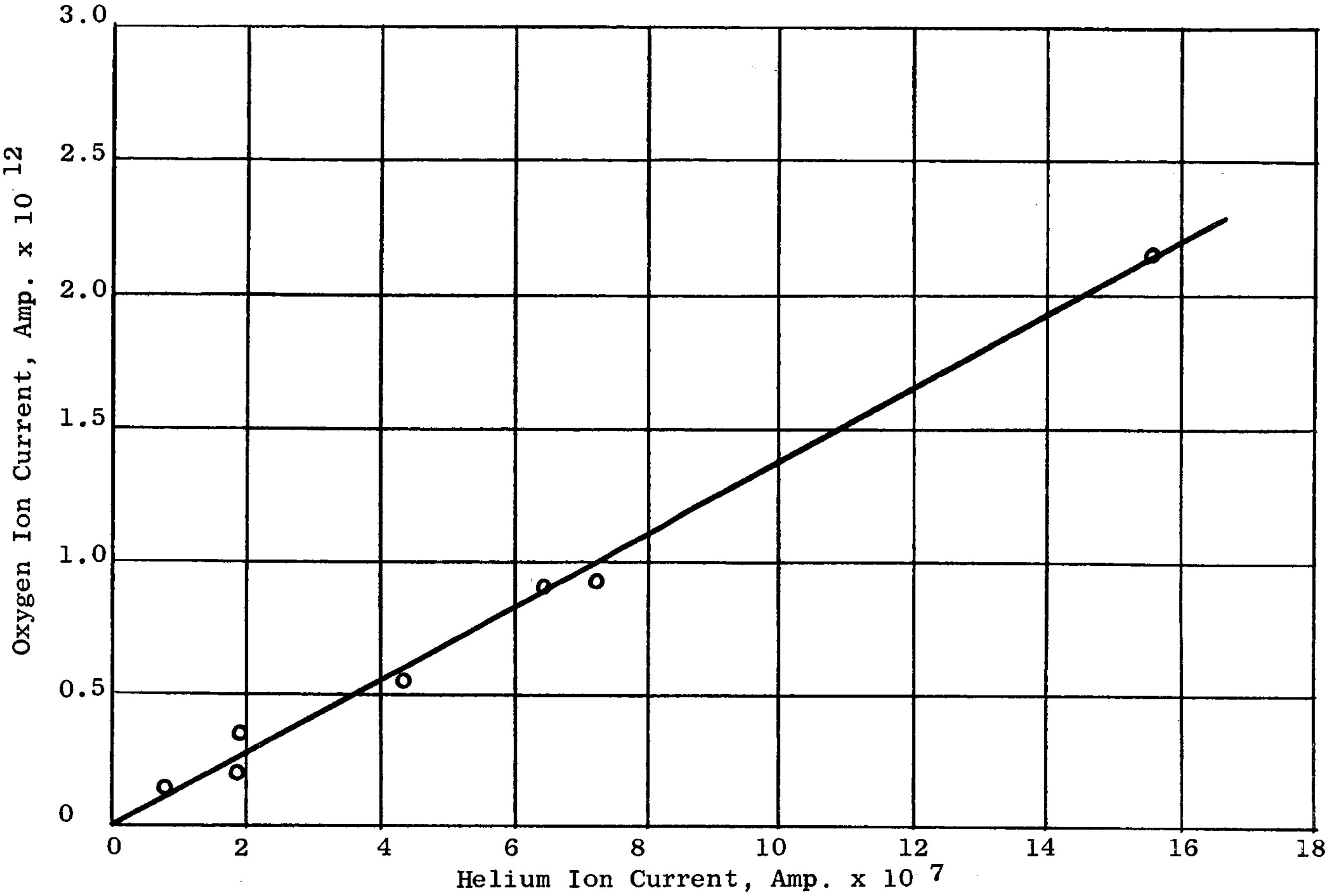


Figure 9. Oxygen Ion Current vs. Helium Ion Current for the Calibrating Gas Mixture.

Figure 10 shows the hydrogen ion current plotted against helium ion current at various helium pressures for both the ultrahigh purity helium and the calibration mixture. It may be noted that an increase in hydrogen pressure is obtained even with the ultrapure helium. The reason for this behavior is not clear, but it is thought to be associated with the very high accelerating voltage necessary to focus the hydrogen ions. In calibrating the mass spectrometer, the difference between the hydrogen ion current from the calibration mixture spectrum and the hydrogen ion current from the ultrapure helium is utilized. As may be seen in Figure 10, this difference is fairly linear with helium ion current at the values for helium ion current normally used in the analyses.

In order to determine the reproducibility of the data obtained with the mass spectrometer, a number of analyses have been made on a cylinder of commercial helium of rather low purity. These analyses were made at various times over a period of about two months. The results are shown in Table II. For the 18 separate analyses the average values obtained were 30.7 and 193 ppm for oxygen and nitrogen, respectively. The corresponding precision, as measured by the standard deviation from the mean, is 19% and 18%. For 12 of the 18 analyses, hydrogen was not detectable. The remaining 6 values varied between 1 and 7 ppm.

Some additional data on the accuracy of the mass spectrometer analyses are available from the welding contamination study which will be discussed in more detail later in this report. For this study, the welding chamber was first filled with pure helium and then various quantities of air were added from a burette. From the volume of the chamber, the quantity of air added, and the known composition of air, the concentrations of oxygen and nitrogen were calculated. A comparison between these measured and calculated values is shown in Table III. Generally good agreement may be noted. The standard deviation between the measured and calculated values for oxygen is 1.3 ppm and for nitrogen, 6.5ppm. For these analyses, the mass spectrometer was calibrated twice each day and the calibration factor was averaged for the analyses calculation. Thus, as might be expected, better precision was obtained than could be anticipated from the data of Table II for which only one calibration was performed each day. The good agreement shown in Table III is further indication of the reliability of the vendor analysis for $O_2$ and $N_2$ in the calibration mixture.

The performance of the mass spectrometer may be summarized as follows: The instrument is normally calibrated for oxygen, nitrogen and hydrogen in helium and the detection limit for each, as estimated from the minimum signal that may be reliably measured, is between 1 and 2 ppm. The accuracy of the analyses of oxygen and nitrogen, as measured by the standard deviation of the various data available, is better than 20%. This value applies to oxygen in helium between 5 and 50 ppm and to nitrogen in helium greater than 5 ppm, with no apparent upper limit. No comparisons are available to estimate the accuracy of the hydrogen analyses. Qualitative analyses of other impurities in the few ppm range can be obtained from interpretation of the mass spectra although no direct calibration is available.

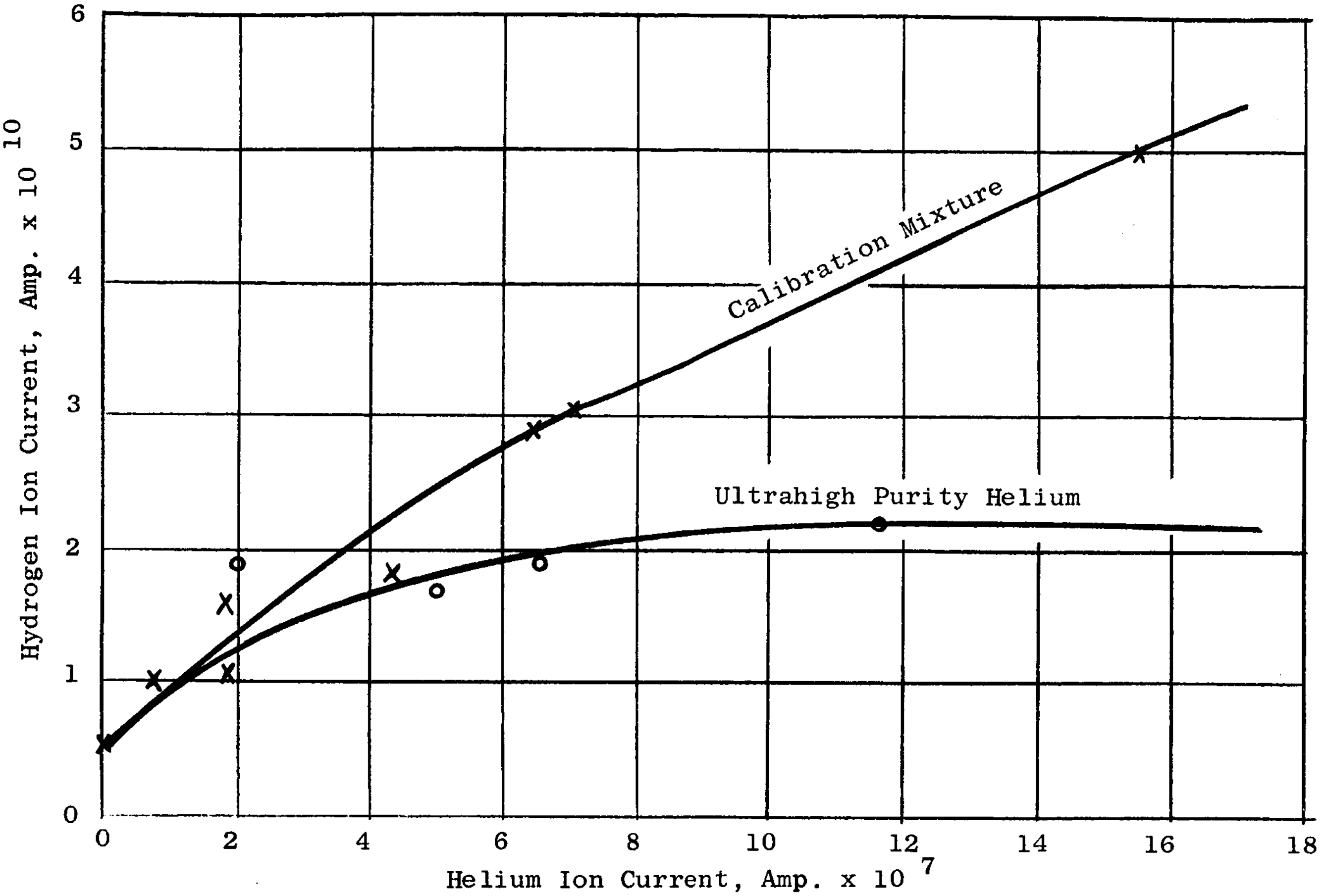


Figure 10. Hydrogen Ion Current vs. Helium Ion Current for the Calibrating Gas Mixture and for the Ultrahigh Purity Helium.

## TABLE II

## ANALYSES OF A CONTAMINATED HELIUM CYLINDER TO DETERMINE REPRODUCIBILITY

| | DATE | $O_2$, ppm | $N_2$, ppm | $H_2$, ppm |
|---|---|---|---|---|
| | 10-20-64 | 31 | 147 | 1 |
| | 10-20-64 | 31 | 168 | ND |
| | 10-26-64 | 46 | 169 | ND |
| | 10-30-64 | 30 | 200 | ND |
| | 10-30-64 | 29 | 200 | ND |
| | 11-2-64 | 30 | 240 | 7 |
| | 11-2-64 | 25 | 234 | ND |
| | 11-4-64 | 34 | 128 | 2 |
| | 11-4-64 | 35 | 160 | ND |
| | 11-5-64 | 22 | 214 | 4 |
| | 11-5-64 | 31 | 202 | 1 |
| | 11-6-64 | 35 | 223 | 2 |
| | 11-6-64 | 35 | 205 | ND |
| | 11-9-64 | 22 | 125 | ND |
| | 11-9-64 | 34 | 210 | ND |
| | 12-23-64 | 22 | 212 | ND |
| | 12-28-64 | 25 | 214 | ND |
| | 12-30-64 | 36 | 227 | ND |
| Average | | 30.7 | 193 | |
| Std. Dev., ppm | | 5.9 | 34 | |
| Std. Dev. | | 19% | 18% | |

ND indicates not detected

## TABLE III

## COMPARISON OF MEASURED AND CALCULATED OXYGEN AND NITROGEN CONCENTRATIONS FOR AIR ADMITTED TO THE WELDING CHAMBER

| Air Added, cc | Oxygen, ppm Calculated | Oxygen, ppm Measured | Nitrogen, ppm Calculated | Nitrogen, ppm Measured |
|---|---|---|---|---|
| 36.3 | 5.7 | 5 | 21.3 | 18 |
| 36.3 | 5.7 | 5 | 21.3 | 20 |
| 92.6 | 14.6 | 14 | 54.4 | 51 |
| 16.1 | 2.5 | 5 | 9.5 | 15 |
| 28.1 | 4.4 | 5 | 16.5 | 20 |
| 42.0 | 6.6 | 7 | 24.6 | 21 |
| 69.2 | 10.9 | 10 | 40.6 | 37 |
| 96.5 | 15.2 | 17 | 56.7 | 60 |
| 144.3 | 22.7 | 21 | 84.7 | 68 |

## III. ELECTROLYTIC HYGROMETER FOR THE WELDING CHAMBER

Due to the difficulty in obtaining reliable analyses for water with the mass spectrometer, an electrolytic hygrometer system has been designed and constructed for the determination of moisture within the welding chamber. Figure 11 shows a schematic diagram of the system. Sample gas from the welding chamber is continuously drawn through the electrolytic cell and the flowmeter transducer. The electrolytic cell is used to determine the water vapor flow rate and the flowmeter measures the total gas flow. To decrease the response time of the system, the length of sample line and the number of fittings between the cell and the welding chamber have been minimized. With the welding chamber helium pressure automatically regulated and critical flow through the metering valve, no additional flow regulation has been found to be necessary. The electrical circuit for the cell consists of a 67.5-volt battery connected in series with the cell, a 10,000-ohm current limiting resistor, the milliammeter, a recorder, and a switch. The multi-range milliammeter gives full scale indication for 0.1, 0.3, 1.0, 3.0, 30 or 100 ma. The recorder has a full scale range of 0.2 ma.

The operation of the hygrometer is based on Faraday's law which when applied to the electrolysis of water, states that for each Faraday (96,500 coulombs), 9.008 grams of water (one gram equivalent weight) dissociate into hydrogen and oxygen gas. If water vapor is being electrolyzed at a constant rate, this rate is $\frac{9.008}{96,500} = 9.34 \times 10^{-5}$ grams per second of water vapor per ampere of electrolyzing current. This value may be converted to standard volume units to obtain 0.1266 std cc per second per ampere of electrolyzing current when standard conditions are taken as 25°C and 760 torr pressure. Using this factor, it may readily be shown that moisture content (ppm by volume) = $\frac{7.60 \times \text{electrolyzing current (microamperes)}}{\text{total flow rate (std cc/minute)}}$.

It has been found quite convenient to set the total flow rate at 76.0 std cc/minute so that the moisture content in ppm by volume is just 0.1 times the cell current in microamperes. This hygrometer system can detect changes in moisture of less than 0.1 ppm at the 1 ppm level.

The Hastings-Raydist type LF-100 mass flowmeter reads directly 0-100 cc per minute of air at standard conditions. Calibration was performed here for helium using a "bubble flowmeter" and several calibration points were also obtained using a wet test meter. All calibration points have been corrected to standard conditions (i.e., 25°C and 760 torr pressure). The results of this calibration are shown in Figure 12. The manufacturer of the flowmeter states that the instrument is accurate within 2% at pressures from 0.01 psia to 250 psia, and temperatures to 200°F. The calibration points of Figure 12 were obtained with helium pressures at the transducer between atmospheric and 10 psig and no particular trend with pressure was noted.

Figure 13 is a photograph of the electrolytic hygrometer system and the welding chamber. It may be noted that the hygrometer sample line is independent of the mass spectrometer sample system because the samples

(1) Inlet Shutoff Valve - Whitey Ball Valve Type 43S4-316
(2) Electrolytic Hygrometer Cell - Beckman Part #76285
(3) Mass Flowmeter Transducer - Hastings-Raydist Type F-100
(4) Metering Valve - Nupro Type SS2S
(5) Vacuum Gauge - USG Type 19931-1; 0-30 in. Hg vac.
(6) Outlet Shutoff Valve - Hoke Toggle Type 1252
(7) Vacuum Pump - Welch #1399B
(8) Hygrometer Cell Milliammeter - Weston Model 911 (Multirange)
(9) Flowmeter Control - Hastings Type LF-100
(10) Recorder - Rustrak Model A (0-0.2 ma.)

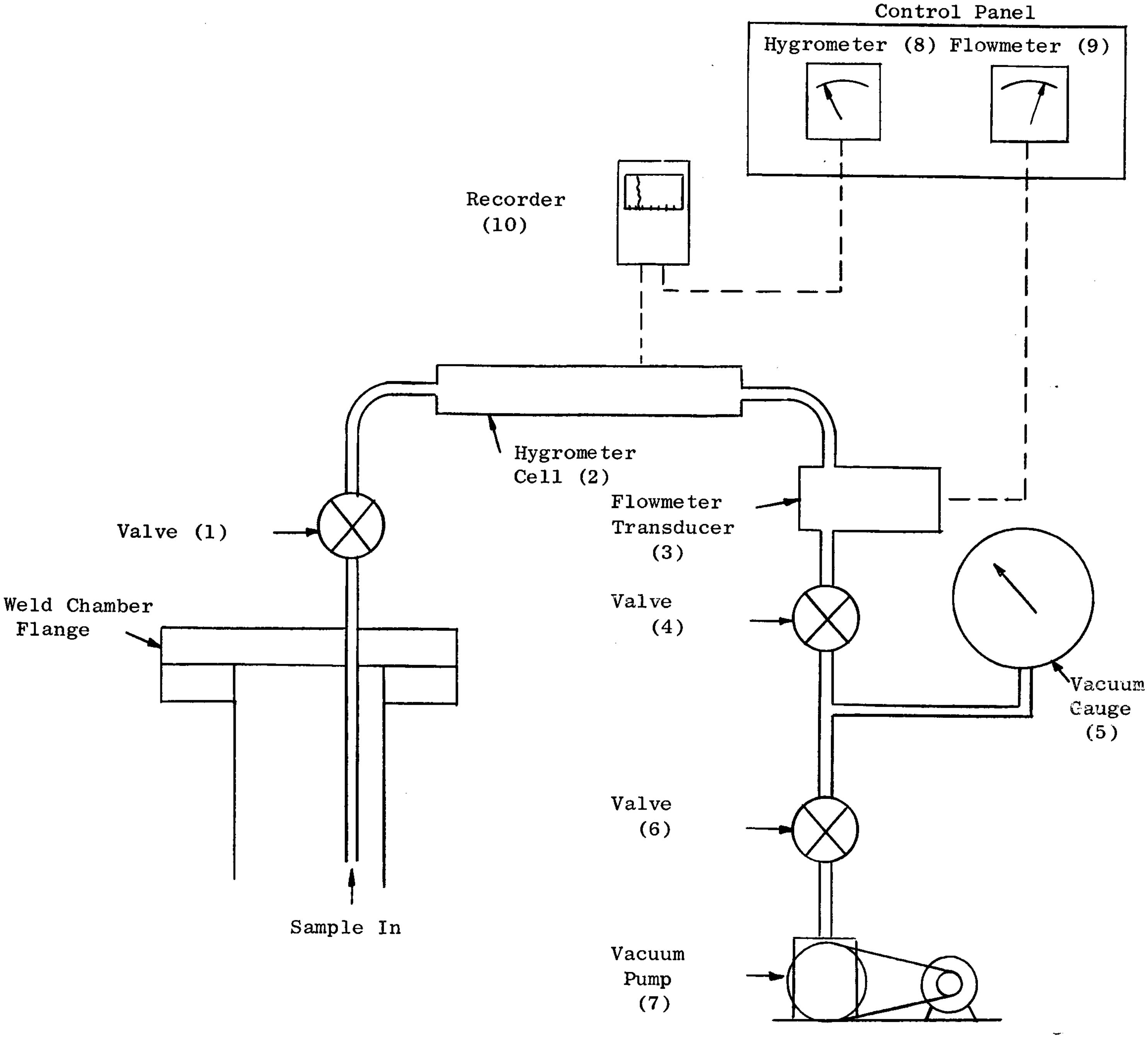


Figure 11. Schematic of Electrolytic Hygrometer System for Moisture Measurement in Welding Chamber.

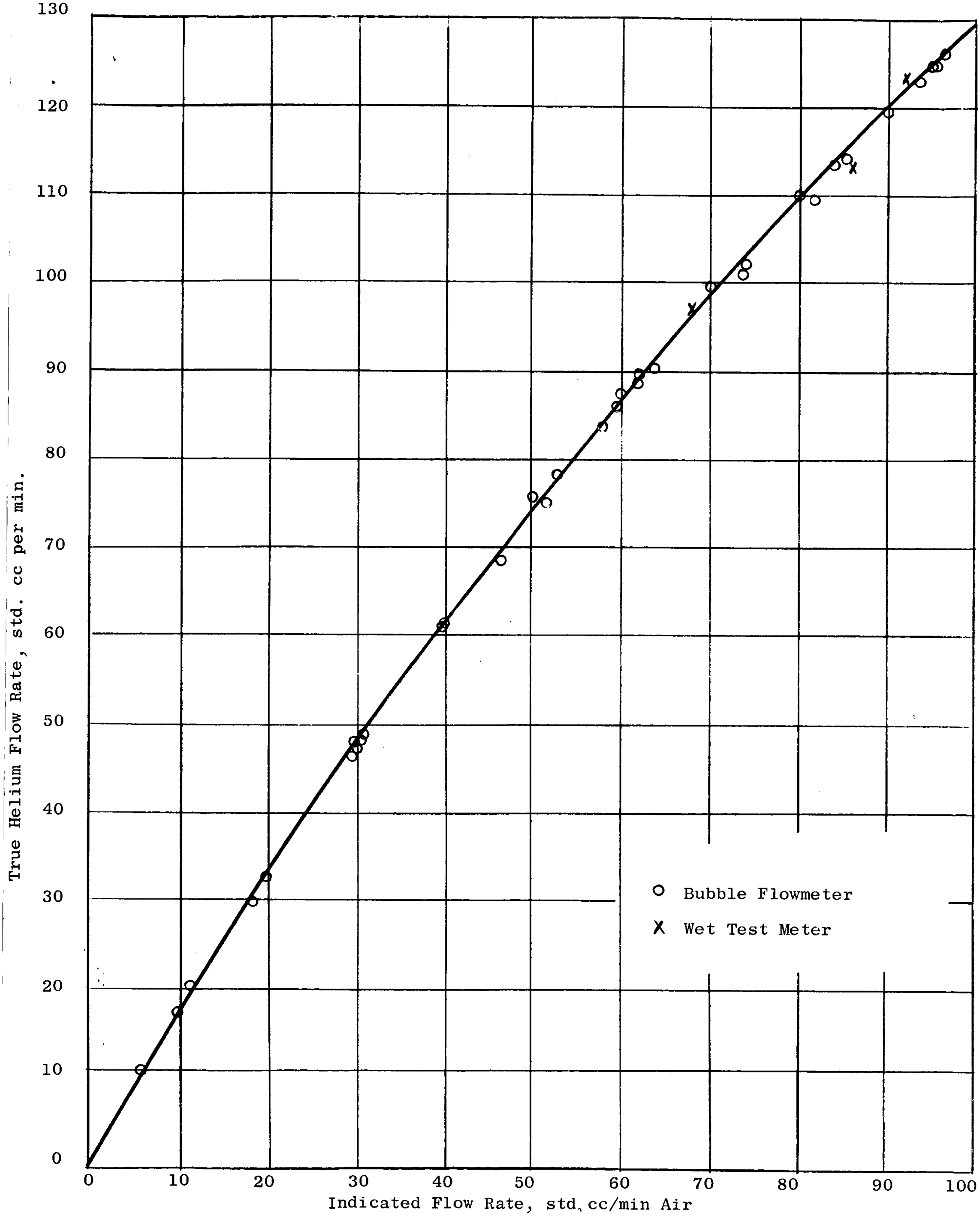


Figure 12. Calibration of Hastings Mass Flowmeter for Helium.

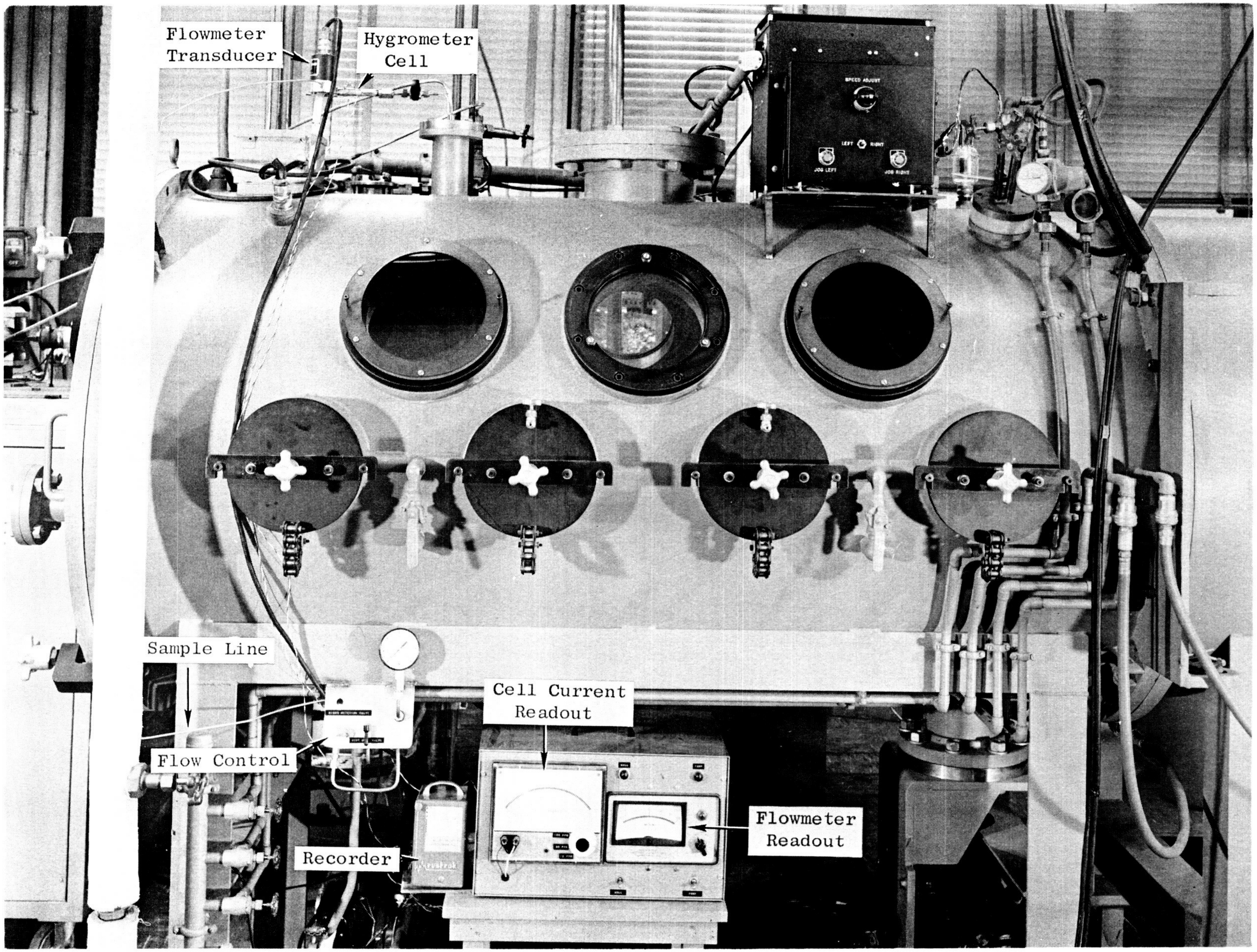


Figure 13. Electrolytic Hygrometer System on the Welding Chamber.

are taken intermittently for mass spectrometric analysis and the hygrometer operates continuously when welding is in progress. The hygrometer controls are placed near the station usually used for welding so that the moisture indication is visible to the welder.

## IV. BECKMAN TRACE OXYGEN ANALYZER AND ELECTROLYTIC HYGROMETER FOR INLET HELIUM ANALYSIS

The most important impurities in the helium admitted to the welding chamber are oxygen and water. Because reliable indication for water vapor cannot be obtained with the mass spectrometer system and because the inlet helium has an oxygen content consistently below the detection limit of the mass spectrometer, a separate system has been used to monitor the inlet gas purity. This system consists of Beckman Model 27901 electrolytic hygrometer and a Beckman Model 80 Trace Oxygen Analyzer which are panel mounted with the associated gas handling system. A photograph of this system is shown in Figure 14. The electrolytic hygrometer has five ranges: 0-10, 0-30, 0-100, 0-300, and 0-1000 ppm water vapor full scale. The oxygen analyzer has two ranges; 0-10 and 0-50 ppm full scale. The detection limit for the hygrometer is about 0.05 ppm water vapor, and for the oxygen analyzer, about 0.1 ppm $O_2$.

## V. OPERATION OF WELDING CHAMBER AND ANALYTICAL SYSTEM

### A. Inlet Helium Supply and Purification Train

Quite early in the Potassium Corrosion Test Loop Development Program, it was found that certain commercially available helium was not of consistently high purity. To guard against the possibility of obtaining a highly contaminated cylinder, arrangements were made with the Air Reduction Company to have a total of sixteen cylinders marked and reserved for exclusive use on this program. Proper precautions are taken here to avoid contamination of these cylinders. In addition, the helium is specified to contain maximum impurities of 2 ppm $O_2$ and 2 ppm $H_2O$ as analyzed by the Air Reduction Company after filling. The helium cylinders are connected to an 8-cylinder manifold with one bank of 4 cylinders in use at one time.

This helium, although of quite high purity, is further purified by passing through a molecular sieve dryer and a titanium furnace. The dryer consists of an 8-inch diameter stainless steel pipe, 4 feet long, containing 60 pounds of Linde Type 13X molecular sieve. The titanium furnace is a 4-inch diameter Inconel pipe, 54 inches long, and contains about 10 pounds of titanium turnings. The helium supply system is of welded construction except for the manifold, pressure regulator, pressure relief valve and the inlet valve to the welding chamber. The dryer operates at room temperature and the titanium furnace at about 1450°F. Normal flow through the purification system is about 5 cfm so that the welding chamber is filled in approximately 10 minutes.

Excellent helium purity has been obtained from this system. The electrolytic hygrometer has consistently shown water vapor concentration of about

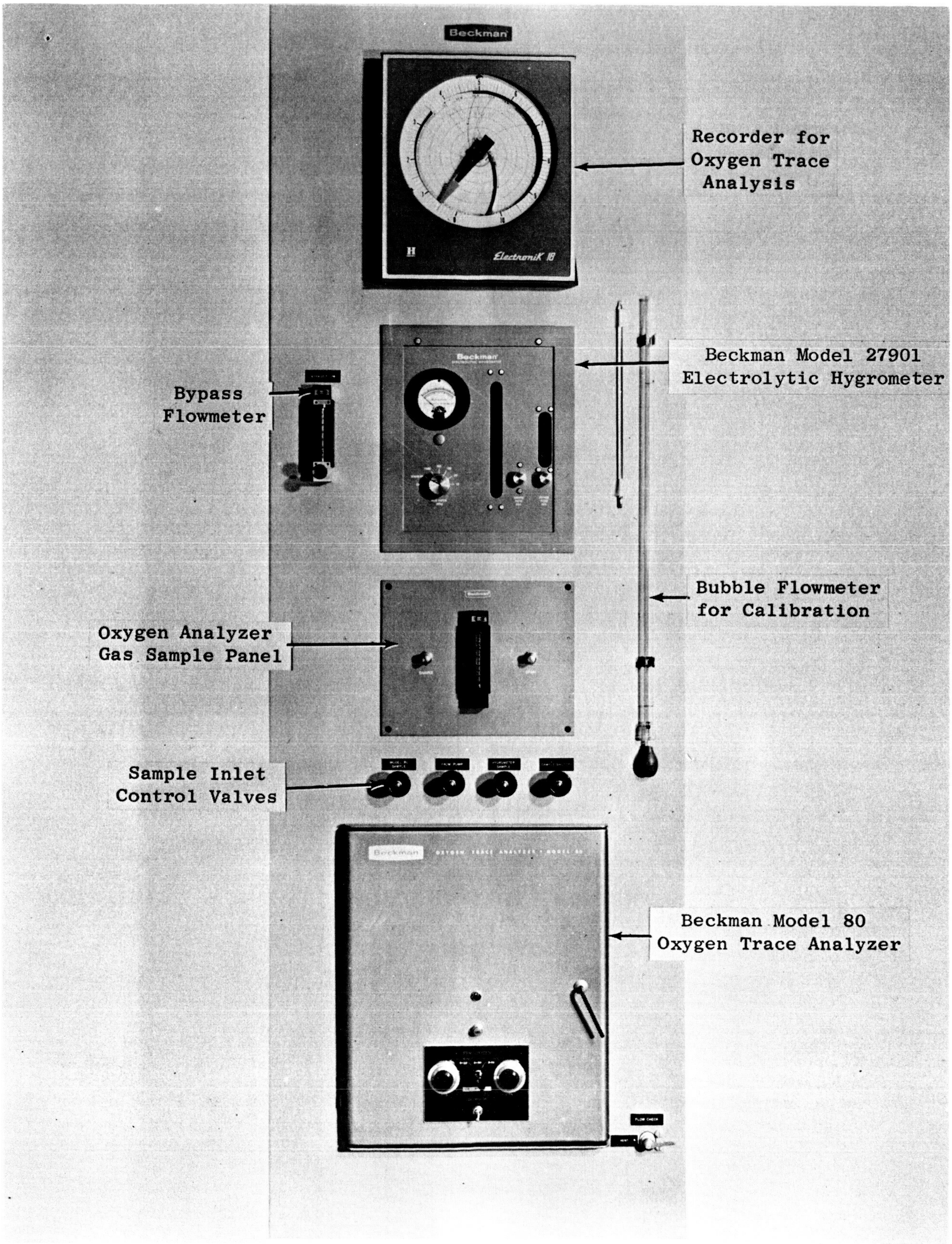


Figure 14. Beckman Electrolytic Hygrometer and Oxygen Trace Analyzer for Monitoring Water and Oxygen Content in Helium.

0.05 ppm. Oxygen content, as measured by the trace oxygen analyzer, has been about 0.6 ppm. This indicates appreciable reduction in moisture and oxygen content from the as-received level as shown in the following table.

## TABLE IV

## TYPICAL OXYGEN AND WATER VAPOR CONTENT OF HELIUM BEFORE AND AFTER PURIFICATION

| As-Received* | | After Purification | |
|---|---|---|---|
| $O_2$ | $H_2O$ | $O_2$ | $H_2O$ |
| 2 ppm | 1 ppm | 0.6 ppm | 0.05 ppm |

*Obtained from Air Reduction Company to a specification requiring less than 2 ppm $O_2$ and less than 2 ppm $H_2O$

Analysis of the purified helium with the mass spectrometer shows typically 2 ppm $N_2$ and 2 ppm $H_2$. No other active impurities have been detected.

### B. Welding Chamber and Filling Procedure

The welding chamber is 3 feet in diameter and 6 feet long and is of stainless steel, double-walled construction. Full opening doors are provided at each end of the chamber. All vacuum seals are made with neoprene O-rings. For welding very large assemblies, an 8-foot diameter extension chamber is also used. The chamber is evacuated with a 10-inch oil diffusion pump backed by a 290 cfm Roots Blower and a 100 cfm mechanical pump. The diffusion pump is equipped with a liquid nitrogen cooled baffle. The lowest pressure that has been obtained in the chamber is about 5 x $10^{-6}$ torr. The chamber contains seven glove ports. Each port is equipped with a "Neo-Sol" sulfur-free dry box glove (Charleston Rubber Co.). As stated by the manufacturer, the water vapor permeability of these gloves is less than 1.5 grams per day per square meter by test ASTM E-96-53T Method B. Before use in welding, these gloves are treated by heating at about 120°F for 15 hours under vacuum.

Figure 15 is a photograph of the mass spectrometer and its associated sampling system. The welding chamber and helium supply are visible in the background.

The procedure that is used in filling the chamber is defined in the specification for welding of columbium-1% zirconium alloy by the inert-gas tungsten arc process. This specification is identified as SPPS 03-0005-00-A, dated 24 September 1963. The chamber is first evacuated to less than 1 x $10^{-5}$ torr and the pressure rise rate, with the pumps valved off, is measured with the ionization gauge. This rate must be less than 5 microns per hour, according

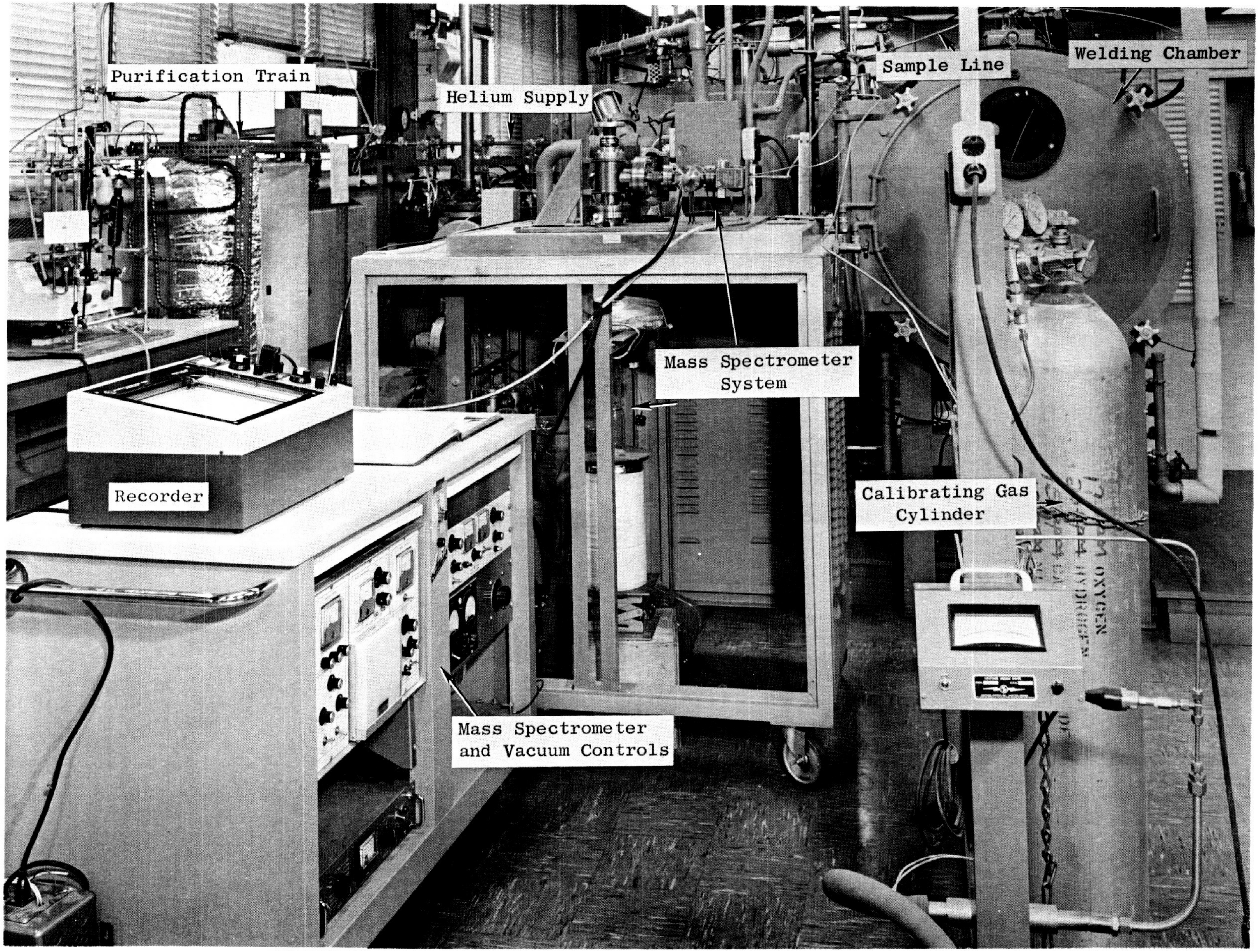


Figure 15. Mass Spectrometer System, Welding Chamber, and Helium Supply.

to the specification, and a pressure increase of about 3 microns per hour is frequently obtained. The chamber is then filled with helium from the purification train while a portion of the flow is analyzed by the Beckman electrolytic hygrometer and Trace Oxygen Analyzer. As mentioned above, the measured purity is typically 0.05 ppm water vapor and 0.6 ppm oxygen. The specification requires that the helium contain less than 1 ppm active impurities. A sample of the helium is then withdrawn from the chamber and analyzed with the mass spectrometer. Generally, this analysis shows about 2 ppm $N_2$ and 2 ppm $H_2$. All other active impurities are below the detection limits of the system, estimated to be about 2 ppm.

The chamber is then re-evacuated to less than $1 \times 10^{-5}$ torr and refilled with helium. The gas within the chamber is again analyzed with the mass spectrometer, the sample flow through the electrolytic hygrometer is started and the moisture content in the chamber is measured. As a further check on chamber purity, the welding specification requires that, before welding the columbium parts, fusion beads be made on a titanium bar. If the titanium bar shows any discoloration, the atmosphere is considered unsuitable for welding columbium alloys.

In addition, the welding specification requires that, prior to welding the first piece and subsequent to the last piece welded in each inert gas environment, weld bend specimens be prepared using the same filler wire material used to weld the intervening pieces. The specimens are retained for subsequent chemical analysis should an investigation of welding contamination be desired.

Also required by the welding specification, is a chamber qualification test which is performed prior to any columbium welding in the chamber and at approximately 3-month intervals throughout the Potassium Corrosion Test Loop Development Program. In order to qualify the chamber, butt-welded joints are prepared with the chamber being filled according to the specification. The weld samples are subjected to bend tests and chemical analyses. The analyses of the weld metal are compared to that of the parent metal and must demonstrate that the welding environment is such that contamination does not exceed the following limits: increase in oxygen content - less than 50 ppm; increase in nitrogen content - less than 50 ppm; increase in hydrogen content - less than 5 ppm; increase in carbon content - less than 10 ppm.

In spite of the rather elaborate procedure required by the present welding specification to maintain a high purity welding environment, contamination of the chamber has occurred on a number of occasions. In every case, the contamination was detected by analysis of the helium within the chamber, the source of the contamination was located, and the leakage was remedied <u>before</u> welding proceeded.

Several instances of chamber contamination may be of interest. In one case, the flange on the large extension chamber had not been properly tightened and air leakage occurred during filling when the chamber pressure was near ambient pressure. The flange did not leak under vacuum since the force of atmospheric pressure kept the flange closed. In this case the chamber was highly contaminated and the mass spectrometer showed about 1% air in the chamber. In addition, the fusion bead on the titanium bar showed marked discoloration.

Another instance of contamination detected by chamber analysis occurred from leakage of the stem seal on the helium inlet valve to the chamber. This valve was leaking only in the open position and thus showed no leakage under vacuum. In this instance, the mass spectrometer showed about 100 ppm air in the chamber and the fusion bead on titanium showed no discoloration. This faulty valve has since been replaced with a solenoid actuated valve which contains no motion seals.

On one occasion, a slight leak occurred in a connection in the water cooling line to the torch. This leakage was immediately detected on the electrolytic hygrometer as a rapid rise in the moisture level within the chamber.

In still another case, air contamination occurred through a pinhole leak in a glove. This leakage was not detected by the vacuum checks since both sides of the gloves are evacuated.

Due to these numerous and varied ways in which weld chamber contamination can occur, the routine practice of frequent analysis of the chamber gas with the mass spectrometer has been adopted along with the continuous analysis with the electrolytic hygrometer during welding. These analyses are performed after the first filling of the chamber with the glove ports closed, after the second filling of the chamber (before welding) with the glove ports open, and again after welding is completed. If welding extends over an unusually long period of time, additional analyses are made during the period.

## C. Some Typical Results of Weld Chamber Analysis

In order to discuss typical impurities found in the welding chamber, the period from December 23, 1964 to June 6, 1965 has been chosen. This is the period of time during which the electrolytic hygrometer was in use on the welding chamber and also is the period during which most of the welding on the Prototype Loop was accomplished. During this time, the chamber was filled and welding was performed according to the welding specification on 28 separate occasions.

Oxygen was below the detection limit of the mass spectrometer in most instances, regardless of whether the analysis was performed immediately after filling the chamber or at the conclusion of welding. No particular trend of increasing oxygen content with time was noted. The maximum oxygen concentration found was 3 ppm.

Typically, the nitrogen content found immediately after filling the chamber was 2 ppm and on one occasion, 11 ppm was detected. An increase in nitrogen of about 5 ppm was generally observed during welding. The maximum increase found was 12 ppm and the maximum nitrogen content found after welding was 16 ppm. Several obvious sources of nitrogen are leakage from the atmosphere through the seals and diffusion through the gloves. Another possible source is the metal being welded. For example, if we assume that during a particular welding period, 1000 grams of metal are heated to a temperature high enough so that, on the average, the nitrogen content of the metal is reduced by 10 ppm by weight, then the resulting nitrogen increase in the welding chamber would be about 6 ppm by volume.

The water content of the gas within the chamber has been found to depend on the time since filling the chamber, the type of welding performed, and the previous treatment of the chamber. Upon initially filling the chamber the moisture content is consistently well below the 1 ppm level and will remain near 1 ppm for a period of about an hour if no heat is applied to the chamber and no welding performed. After this initial period, a more rapid increase in moisture is observed. This increase is most severe if heavy parts are being welded.

Figure 16 shows the change in moisture content with time during typical welding operations. It may be noted that the rate of moisture increase was most rapid during welding and more gradual during periods of no welding. This rapid increase in moisture during welding is attributed to desorption of water from the parts being welded, from the fixturing, or from the chamber walls. A drastic increase in moisture content is obtained if the chamber is heated by hot water flow through the jacket.

An overnight "bakeout" of the chamber with hot water while the chamber is maintained under vacuum results in a decrease in the quantity of moisture observed upon subsequent filling of the chamber. It has also been observed that a low pressure rise rate under vacuum is associated with lower moisture content when the chamber is subsequently filled with helium.

These observations indicate that there are both sources and sinks for water vapor within the chamber and that the net change in moisture content depends on which process predominates. It also would appear that diffusion of moisture through the gloves is not the primary source of water vapor within the chamber. The behavior of the moisture content within the welding chamber and the tentative conclusion reached here are quite similar to results obtained in the study conducted at the Westinghouse Astronuclear Laboratory (2).

Hydrogen has been found to be typically about 2 ppm immediately after filling the chamber, and hydrocarbons are not detected (probably less than 2 ppm). The highest hydrogen found after filling the chamber was 10 ppm. An increase to about 12 ppm was generally observed during welding. However, on several ocassions, much higher hydrogen increases were measured, the

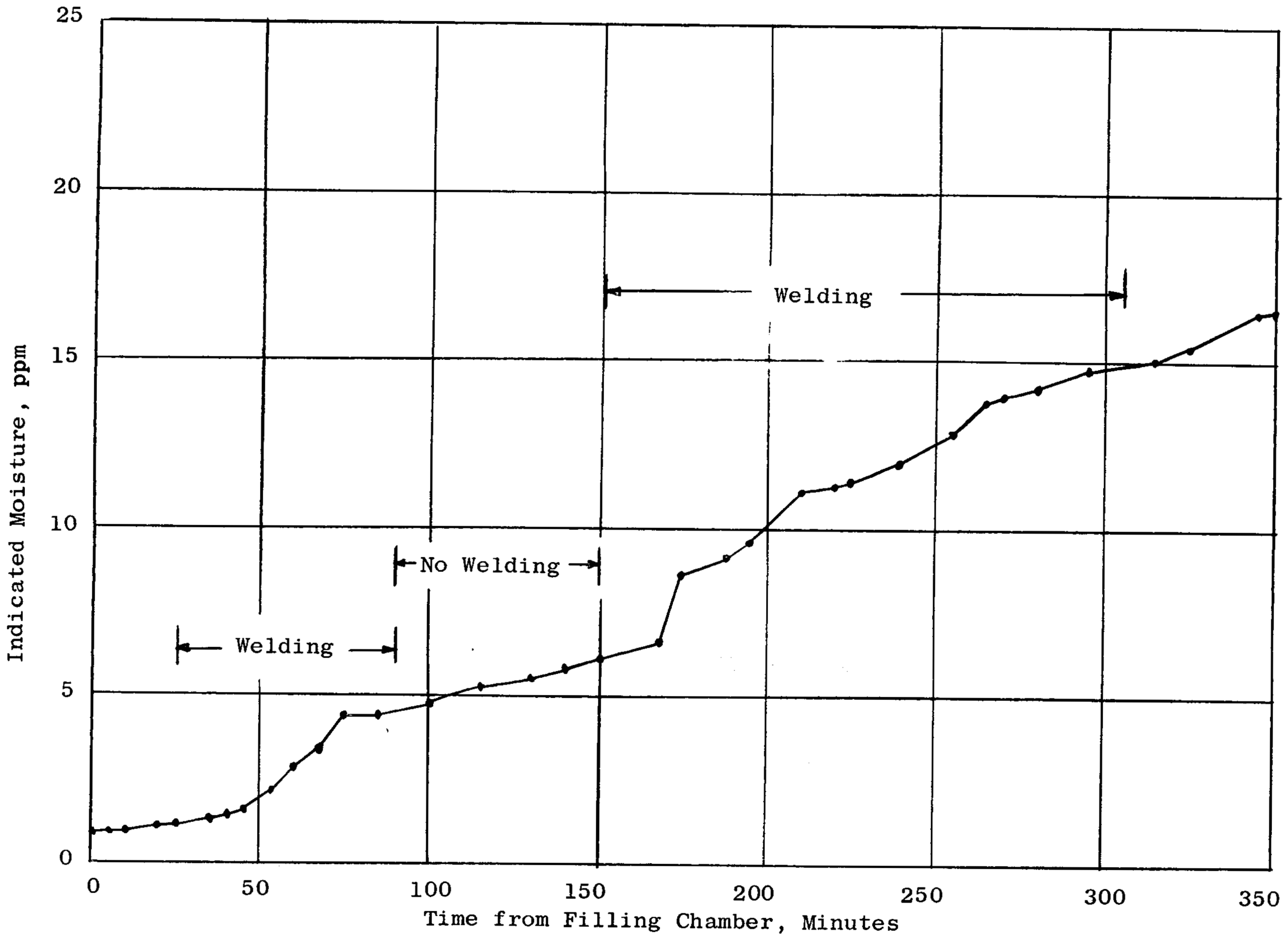


Figure 16. Typical Change in Moisture with Time in the Welding Chamber.

maximum being 46 ppm. In each case of unusually high hydrogen content, hydrocarbon peaks occurred in the mass spectrum. These peaks are due to methane (16 AMU), $CH_3$ (15 AMU), and CH (13 AMU) and in some cases, the hydrocarbon peaks appeared at 27, 29 and 30 AMU. The high hydrogen and hydrocarbon peaks usually occurred under the same conditions for which the water vapor content was high; that is, after prolonged periods of welding or when unusually heavy pieces are being welded. A possible source of these light hydrocarbons is thermal decomposition of residual pump oil within the chamber or decomposition of the neoprene gloves. The possibility is not discounted that the high hydrogen content is ultimately responsible for the hydrocarbons through some sort of chemical reactions. The hydrogen is most likely evolved from the metal surfaces which are heated due to their proximity to the arc.

Hydrogen contamination, at the levels mentioned above, is not expected to cause appreciable increase in the hydrogen content of the weld. In a separate study conducted here (3), several butt-welded samples of columbium alloy D-43, 0.040-inch thick, were prepared in a helium atmosphere containing 1% hydrogen which had been deliberately added to the chamber. Even at this relatively high hydrogen level, the hydrogen content of the weld metal increased only 6 ppm (by weight).

## VI. WELD CONTAMINATION STUDY ON Cb-1Zr

After sufficient experience had been obtained to determine the reliability and sensitivity of the analyses and to determine what level of purity could be maintained within the chamber, a study of the relation between gaseous impurity levels and weld metal pickup for Cb-1Zr welds was undertaken. It should be pointed out that a number of factors other than gaseous impurities in the chamber could affect the apparent change in weld metal purity and these extraneous effects should be eliminated as far as possible. Thus for each series of welds, the thickness of the weld specimen, fixturing, current voltage, welding speed, preparation of weld specimens for analsis, and the analytical procedure were the same for each specimen.

Various quantities of air were introduced to the chamber, after initially filling the chamber with pure helium. The air was added from a 100 cc, oil filled gas burette. The burette was initially charged with room air drawn through a drying tower to remove moisture. In one test, analyses of the chamber were made after allowing one-half hour and one hour for mixing of the air with helium. No difference in the analyses was detected and so about one hour was allowed for homogenization between adding the air and making the test welds. The sample lines for the mass spectrometer and the electrolytic hygrometer were extended within the chamber to a point about 13 inches from the torch. The chamber was initially filled with helium by the procedure defined in the welding specification. The geometrical volume of the chamber was estimated to be 46.9 cubic feet so that 1.33 cc is equal to one ppm by volume.

The quantity of moisture in the chamber was varied at the low levels simply by allowing sufficient time for the spontaneous moisture increase to occur. To obtain the higher moisture levels, hot water was passed through the chamber jacket to accelerate the process.

The majority of the test welds were made on 0.062-inch thick sheet, approximately one inch wide and two inches long. This sheet has been cold-rolled from the 0.125-inch thick as-received material. Several tests were also conducted on the 0.125-inch sheet. All weld specimens were pickled before welding in an HF-$HNO_3$ acid solution as defined in the specification 03-0010-00-B. All welding was done automatically at 2 inches per minute. This rather slow speed was selected to be representative of the manual welds which are frequently made in the chamber. Welding current and voltage were 95 amperes and 19 volts for the 0.062-inch sheet, and 160 amperes and 19 volts for the 0.125-inch sheet. The fusion zone, approximately 0.25 inch wide, was cut from each specimen and submitted for analysis. No surface material was removed from the specimens after welding, the samples being only degreased in ether prior to the analyses. The two 0.125-inch thick weld specimens were bend tested through 105 degrees over a 1T radius at room temperature with no evidence of cracking. Bends were made perpendicular to the weld axis to insure bending of parent metal, weld, and heat affected zone. The bend rate was 0.44 inch per minute, and the weld root was in tension.

Analyses by the vacuum fusion method were made on each sample along with duplicate analyses on the control (unwelded) samples. Analyses for nitrogen by the Kjeldahl method were also made on the samples welded with air contamination and on the control samples.

The results of the weld contamination study are summarized in Table V. The difference in oxygen between the unwelded 0.062-inch specimen and the unwelded 0.125-inch specimen is apparently due to inhomogeneity in the material taken from remote parts of the same sheet since the oxygen content of the unwelded 0.062-inch specimen is in excellent agreement with the as-received (G.E.) analysis while the unwelded 0.125-inch specimen is considerably lower (by about 130 ppm) than the as-received analysis.

The data in Table V show that for the highest air contamination level in the helium (109 ppm), the 0.062-inch sheet had an oxygen increase of 19 ppm during the welding and the maximum oxygen increase of 29 ppm occurred with the specimen welded in 21 ppm air. The 0.125-inch specimen welded in helium contaminated with 109 ppm air indicated an oxygen increase of 35 ppm. There is no particular trend of increasing oxygen in the weld metal with increasing oxygen contamination in the chamber up to 109 ppm. In view of the uncertainty involved in the local homogeneity of the specimens, there is some doubt as to whether or not there is a real increase at all.

Table V shows also an interesting comparison between the vacuum fusion results for nitrogen and the corresponding Kjeldahl results. As might be

## TABLE V

## RESULTS OF WELD CONTAMINATION STUDY ON Cb-1Zr

| Nominal Contam. Vol. ppm | Air Added Vol. ppm | Chamber Analysis, Vol. ppm $O_2$ | $N_2$ | $H_2$ | $H_2O$ | Weld Metal & Control Specimen Analysis, Wt. ppm Vacuum Fusion O | H | N | Kjeldahl N |
|---|---|---|---|---|---|---|---|---|---|
| **0.062-Inch Sheet** | | | | | | | | | |
| Control | | | Not Welded | | | 227 | 2 | 92 | 185 |
| | | | | | | 232 | 2 | 91 | |
| 0 Air | 0 | <2 | <2 | 2 | 2.8 | 247 | 2 | 106 | 118 |
| 10 Air | 12.0 | 5 | 15 | <2 | 9.0 | 255 | 1 | 105 | 152 |
| 20 Air | 21.1 | 5 | 20 | 6 | 13.3 | 258 | 2 | 103 | 85 |
| 30 Air | 31.6 | 7 | 21 | <2 | 3.0 | 242 | 1 | 103 | 107 |
| 50 Air | 52.0 | 10 | 37 | <2 | 7.6 | 232 | <1 | 96 | 110 |
| 70 Air | 72.6 | 17 | 60 | 2 | 13.2 | 239 | 3 | 110 | 91 |
| 100 Air | 108.5 | 21 | 68 | 5 | 18.3 | 248 | <1 | 108 | 146 |
| 5 $H_2O$ | 0 | <2 | <2 | <2 | 5.3 | 238 | 1 | 100 | --- |
| 10 $H_2O$ | 0 | <2 | <2 | <2 | 10.0 | 232 | <1 | 82 | --- |
| 20 $H_2O$ | 0 | <2 | 2 | 3 | 20.1 | 249 | 1 | 106 | --- |
| 30 $H_2O$ | 0 | <2 | 2 | 2 | 30.2 | 247 | 2 | 97 | --- |
| 50 $H_2O$ | 0 | <2 | 6 | 10 | 51.7 | 254 | 1 | 75 | --- |
| **0.125-Inch Sheet** | | | | | | | | | |
| Control | | | Not Welded | | | 102 | <1 | 55 | 72 |
| | | | | | | 117 | 4 | 54 | |
| 100 Air | 108.5 | 21 | 68 | 5 | 21.0 | 144 | 2 | 73 | 100 |
| 50 $H_2O$ | 0 | <2 | 6 | 10 | 55.1 | 122 | 3 | 59 | --- |
| MCN 415-1 Vendor Analysis | | | | | | 170 | 2.7 | 80 | |
| MCN 415-1 As-Received - G.E. Analysis | | | | | | 243 | <1 | 74 | |

expected, the Kjeldahl results are higher in most cases than the vacuum fusion analyses. This is due to the fact that nitrogen may be picked up during the sample preparation and dissolution procedure for the Kjeldahl analysis, while for the vacuum fusion analyses, low values may occur due to imcomplete release of nitrogen during the fusion process. It may also be noted that the vacuum fusion results are more consistent than the Kjeldahl values.

## VII. WELD CONTAMINATION STUDY ON T-111

A similar weld contamination study has been made on T-111, the alloy selected for use in fabrication of an advanced refractory alloy corrosion loop under Contract NAS 3-6474. This is a tantalum base alloy with nominally 2% hafnium and 8% tungsten. The methods of addition of contaminants and general analytical techniques employed were the same as for the Cb-1Zr study described above.

A total of 27 test welds were made at nominal contamination levels of 0, 10, 20, 50, 100 and 200 ppm air and 10, 20 and 50 ppm water vapor. Welds were made on sheet of 3 thicknesses; 0.040, 0.080 and 0.160 inch. Chemical analyses were obtained for O, N and H by the vacuum fusion method and carbon was determined conductimetrically. Weld metal analyses were made on only the 0.040 and 0.160-inch thick specimens and in addition, unwelded control specimens of the 0.040 and 0.160-inch thick material were analyzed.

All 0.080-inch thick weld specimens were bend tested through 105 degrees over a 1.2T radius at room temperature with no evidence of cracking. Bends were made perpendicular to the weld axis to insure bending of parent metal, weld, and heat affected zone. The bend rate was 0.44 inch per minute, and the weld root was in tension.

Weld specimens were prepared from the same heat of material and were pickled in a $HF$-$HNO_3$-$H_2SO_4$ solution before welding. No surface material was removed after welding. Approximate welding conditions were: for 0.040-inch sheet, 110 amps, 17 volts; for 0.080-inch sheet, 185 amps, 19 volts; for 0.160-inch sheet, 270 amps, 21 volts. Welding was done automatically at 2 inches per minute.

The results of the T-111 weld contamination study are summarized in Table VI. As may be seen from these data, reasonably good agreement between the mass spectrometric analysis for $O_2$ and $N_2$ and the quantity of air added has again been obtained except for the welds made at the highest air contamination level. As in the case of the Cb-1Zr study, some inhomogeneity in the material is evidenced by the difference in oxygen content of the unwelded thin sheet compared to the unwelded 0.160-inch thick specimens.

There is slight evidence of increasing nitrogen content with increasing air contamination level in the case of the welds in the 0.160-inch sheet. However, the overall increase at the highest contamination level (178 ppm air)

## TABLE VI

## RESULTS OF WELD CONTAMINATION STUDY ON T-111

| Nominal Contam. Vol. ppm | Air Added Vol. ppm | Chamber Analysis, Vol. ppm | | | | Weld Metal & Control Specimen Analysis, Wt. ppm | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | $O_2$ | $N_2$ | $H_2$ | $H_2O$ | O | H | N | C |
| **0.040-Inch Sheet** | | | | | | | | | |
| Control | | Not Welded | | | | 16 | <1 | 23 | 5 |
| 0 Air | 0 | <2 | <2 | <2 | 1.5 | 19 | <1 | 23 | 14 |
| 10 Air | 11.8 | <2 | 12 | 6 | 3.0 | 16 | 1 | 21 | 4 |
| 20 Air | 21.7 | 4 | 14 | <2 | 9.9 | 17 | <1 | 24 | 7 |
| 50 Air | 51.2 | 8 | 35 | 7 | 17.4 | 12 | <1 | 14 | 10 |
| 100 Air | 101.0 | 23 | 78 | <2 | 9.5 | 25 | 1 | 28 | 19 |
| 200 Air | 177.6 | 23 | 200 | 4 | 19.0 | 20 | 1 | 15 | 18 |
| 10 $H_2O$ | 0 | <2 | <2 | <2 | 10.4 | 28 | 1 | 24 | 17 |
| 20 $H_2O$ | 0 | <2 | 2 | 11 | 20.1 | 15 | 1 | 16 | 14 |
| 50 $H_2O$ | 0 | <2 | <2 | 18 | 52.3 | 26 | 1 | 17 | 10 |
| **0.160-Inch Sheet** | | | | | | | | | |
| Control | | Not Welded | | | | 40 | 1 | 13 | 18 |
| 0 Air | 0 | <2 | <2 | <2 | 0.9 | 34 | 2 | 17 | 24 |
| 10 Air | 11.8 | <2 | 12 | 6 | 4.5 | 35 | 3 | 22 | 17 |
| 20 Air | 21.7 | 4 | 14 | <2 | 12.0 | 39 | 3 | 23 | 16 |
| 50 Air | 51.2 | 8 | 35 | 7 | 19.4 | 33 | 12 | 24 | 15 |
| 100 Air | 101.0 | 23 | 78 | <2 | 12.0 | 35 | 2 | 29 | 18 |
| 200 Air | 177.6 | 23 | 200 | 4 | 21.0 | 34 | 2 | 28 | 15 |
| 10 $H_2O$ | 0 | <2 | <2 | <2 | 14.5 | 29 | 2 | 23 | 18 |
| 20 $H_2O$ | 0 | <2 | 2 | 11 | 22.0 | 24 | 2 | 21 | 17 |
| 50 $H_2O$ | 0 | <2 | <2 | 18 | 54.4 | 33 | 2 | 21 | 19 |
| Vendor Analysis NRC Heat No. 2511 | | | | | | 37 | | 15 | 29 |

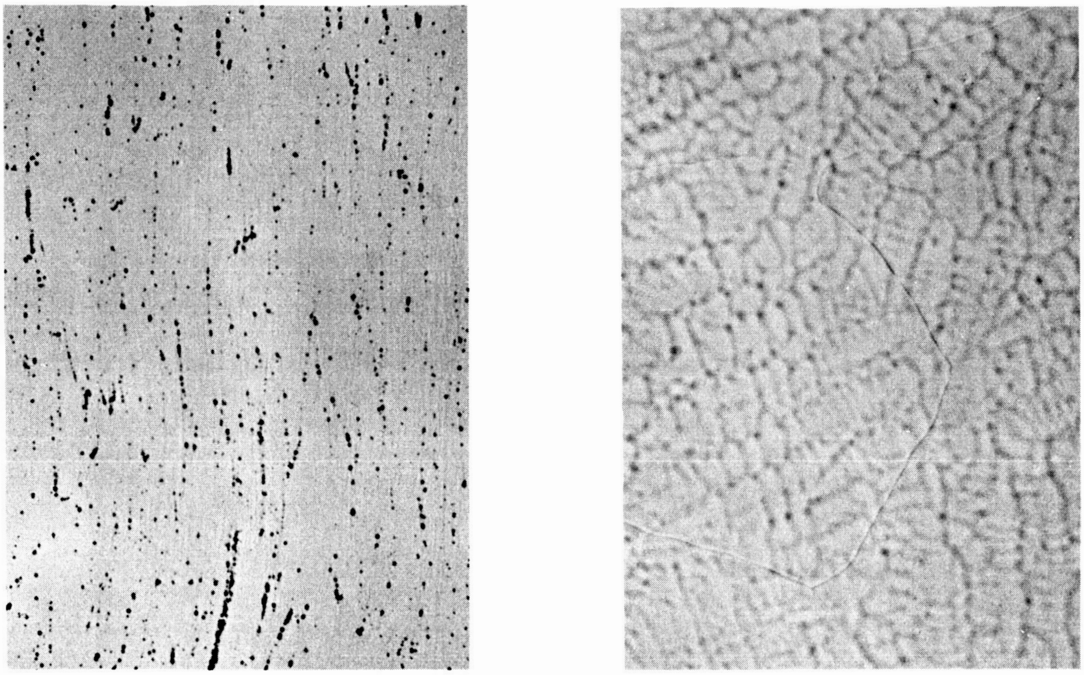
is only 16 ppm nitrogen. There is also an indication that the hydrogen content of the thick specimens is slightly greater than that of the thin specimens. An apparent hydrogen increase was obtained in the 0.160-inch thick specimens at the 50 ppm air level; however, no explanation for this increase is obvious and it is not consistent with the other data. The apparent variations in carbon content are not considered significant due to the uncertainties in the carbon analyses at these very low levels. With the uncertainty involved in the local homogeneity of the specimens and possible variations in the chemical analyses, it is doubtful as to whether or not there was any real increase in the interstitial element concentration of the welded specimens.

The 0.080-inch thick weld specimens made in pure helium and at the highest contamination levels, 200 ppm air and 50 ppm water vapor, were examined metallographically. These weld microstructures and that of the parent metal are shown in Figure 17. No increase in the quantity of precipitating phases was noted at these highest levels of contamination investigated.

The results of the weld contamination study on T-111 thus indicate that contamination of the weld, if any occurs at all, is not readily detectable by vacuum fusion analyses, by room temperature bend tests, or by metallographic examination of specimens welded in helium containing up to 200 ppm air or 50 ppm water vapor. Similar results were obtained in the study of the Cb-1Zr alloy although metallographic examination was not performed.

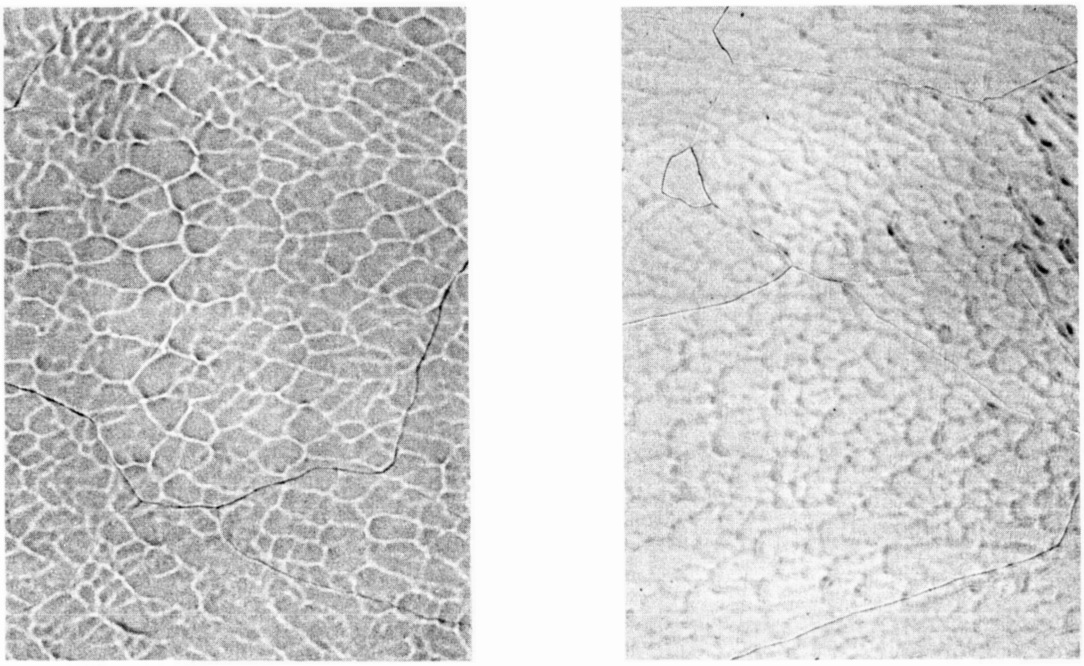
It is pertinent to note that Sinclair (4) has recently reported an investigation of the contamination and bend ductility of selected refractory metal weldments. Alloys investigated were Cb-1Zr, Cb-752, FS-85, B-33, D-43 and T-111. Specimens of the six alloys were TIG welded using clean laboratory conditions and all withstood bend testing (1T at room temperature) in the as-welded condition without cracking. The "clean" welding was done in argon with 2 to 4 ppm $O_2$ and 17 to 19 ppm $H_2O$. Similar specimens of the six alloys were also welded under "field" conditions, i.e., in argon containing 95 to 100 ppm $O_2$. The Cb-752, B-33, FS-85 and D-43 specimens welded in this environment cracked at bend angles of 35 degrees or less, while Cb-1Zr and T-111 specimens showed cracks under microscopic examination (30X) after 90-degree bends. Oxygen analyses of the weld specimens showed a small, but consistent increase in oxygen content of the alloys welded in argon with 100 ppm $O_2$ as compared to the alloys welded under clean laboratory conditions. This increase ranged from 50 to 90 ppm oxygen in the six alloys. Nitrogen was not monitored in the welding atmosphere and the nitrogen content of the field welded specimens was not reported. However, if one assumes that the oxygen resulted from air leakage, then the corresponding nitrogen content of the argon would be about 400 ppm, and nitrogen contamination of the welds also could have been a contributing factor in the decreased weld ductility.

Incorporating the results of the presently reported study with those of Sinclair (4) summarized above, one might cautiously conclude that at some contamination level between roughly 200 and 500 ppm air, sufficient contamination of the weld occurs to be readily detectable by vacuum fusion



Parent Metal

Pure Helium
($\ll 2$ $O_2$, $<2$ $N_2$, $<2$ $H_2$, 0.9 $H_2O$)



50 ppm Water Vapor Nominal
($<2$ $O_2$, $<2$ $N_2$, 18 $H_2$, 53 $H_2O$)

200 ppm Air Nominal
(23 $O_2$, 200 $N_2$, 4 $H_2$, 20 $H_2O$)

Figure 17. Microstructures of 0.080-Inch Thick T-111 Alloy Parent Metal and Weldments Made in Pure and Contaminated Helium Welding Environments.

Mag: 150X

Etchant: 30 HCl - 30 HF - 15 $HNO_3$ - 25 $H_2O$

analytical methods and to appreciably affect the as-welded ductility of both Cb-1Zr and T-111 at room temperature. This information, of course, should not be used to conclude that such a high level of welding environment contamination is acceptable for general applications; it merely identifies a range of contamination which corresponds to the onset of readily observable changes in chemical composition and room temperature ductility of two of the more weldable refractory alloys.

## VIII. SUMMARY AND CONCLUSIONS

A relatively simple mass spectrometer system has been assembled and used to measure the concentrations of $O_2$, $N_2$ and $H_2$ impurities in a vacuum purged, inert gas welding chamber. Qualitative analyses of other impurities are obtained by interpretation of the mass spectra. It has been found that calibration against pure gas pressures within the analysis system leads to erroneous results, especially for $O_2$ and $H_2$. Calibration of the system has thus been performed against helium of known impurity content. The detection limits of the mass spectrometer system have been found to be between 1 and 2 ppm for $O_2$, $N_2$ and $H_2$.

Although the mass spectrometer system appears to be adequate for the present purposes, there are several modifications which could be made to improve the sensitivity and stability of the system. The use of electromagnetic scanning rather than electrostatic, should result in increased sensitivity to oxygen due to the higher accelerating voltage at which oxygen would be focused. In addition, hydrogen would be focused at a much lower accelerating voltage than is necessary with the permanent magnet, and thus the difficulties with hydrogen analyses should be reduced considerably. Other probable advantages would be lack of serious mass discrimination, greater ion current stability, and increased resolution at the higher mass numbers.

Modification of the sample inlet system so that the sample is introduced directly into the ion source region should decrease the loss of the more active impurities by adsorption on the walls of the system. Such a modification would now be convenient with the presently available spectrometer tubes having an exposed ion source.

Other changes could be made to increase the sensitivity and accuracy of the system. These might include a molecular leak sample inlet and differential pumping in the spectrometer tube. Such features are normally supplied on the commercially available analytical type mass spectrometers. Their use on the present system would, however, considerably increase its complexity.

An electrolytic hygrometer system has been designed and constructed specifically for use on the welding chamber. During welding, a continuous record is made of chamber moisture content.

Special helium having both moisture and oxygen content less than 2 ppm is used to fill the welding chamber. This is further purified by use of a molecular sieve dryer and titanium furnace. Typical purity from the purification train is 0.6 ppm $O_2$ and 0.05 ppm $H_2O$ as measured by a commercially available trace oxygen analyzer and electrolytic hygrometer.

Analyses of the chamber gas immediately after filling with pure helium show typically less than 2 ppm $O_2$, 2 ppm $N_2$, 2 ppm $H_2$, and less than 1 ppm $H_2O$. Examination of mass spectra between 2 and 44 AMU shows no indication of other active impurities in the inlet helium at concentration greater than 2 ppm, which is estimated to be the detection limit of the mass spectrometer. During the course of welding, a slight increase in $O_2$ and $N_2$ occurs. Increases in $H_2$ and $H_2O$ are greater and depend on the type of welding performed, the length of time since filling the chamber and the previous treatment of the chamber. Typical values found after welding are 12 ppm $H_2$ and 13 ppm $H_2O$. Some light hydrocarbons are apparently produced within the chamber, especially when heavy parts are welded. On several occasions, gross contamination of the welding chamber was detected through analyses of the chamber gas and welding in such impure helium was thus prevented.

A study has been made of the contamination of Cb-1Zr welds in helium to which known quantities of air as high as 108 ppm or water vapor as high as 52 ppm had been added. Considering the range of homogeneity of the specimens and the uncertainties of the chemical analyses, contamination of the welds could not be detected. A similar study has been made of contamination of T-111 welds at impurity levels as high as 178 ppm air or 52 ppm water vapor. Again, contamination of the weld metal by reaction with gaseous impurities could not be detected. This information should not, of course, be used to conclude that such high levels of welding environment contamination are acceptable for critical applications.

With the experience gained thus far, beneficial revisions can be made to the welding specification which was originally prepared for the Potassium Corrosion Test Loop Development Program. In particular, requirements will be included in the specifications to monitor the gas composition in the chamber during welding.

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