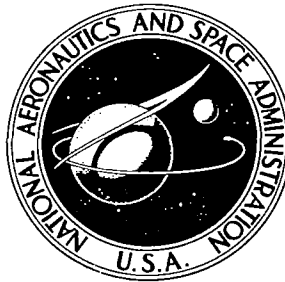


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**THE USE OF THERMODYNAMIC
PROPERTIES OF METAL-GAS SYSTEMS
AS REDUCED-PRESSURE STANDARDS**

by Charles E. Lundin

Prepared by
UNIVERSITY OF DENVER
Denver, Colo.
for Langley Research Center



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SYSTEMS AS REDUCED-PRESSURE STANDARDS

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ABSTRACT

The erbium-hydrogen binary system was investigated by means of a Sievert's apparatus to determine the applicability of such a metal-gas system for a low-pressure standard. A thermodynamic relationship was empirically determined for the two-phase region, erbium solid solution plus erbium dihydride, for the pressure range of 10^{-4} to 10 torr and the temperature range of 550 to 950°C. This relationship, the van't Hoff equation, is a first-order equation representing the dissociation of hydrogen gas from the dihydride phase to form the metal solid solution as a function of temperature. The coefficient of determination for the data presented was computed to be 0.999. Data is presented on the feasibility of using metal-oxygen and metal-nitrogen systems as secondary low-pressure standards.

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by

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SUMMARY

The erbium-hydrogen binary system was investigated by means of a Sievert's apparatus to determine the applicability of such a metal-gas system as a hydrogen reduced-pressure standard. The Sievert's apparatus was specially designed and assembled for this work and incorporated with all of the refinements necessary to conduct precision measurements. A thermodynamic relationship for the two-phase region, erbium solid solution plus erbium dihydride, was established to assess the potential of this system as a secondary reduced-pressure standard. The relationship is the van't Hoff equation representing the reduced pressure-temperature variables of the system over the temperature range, 550 to 950°C, and the low-pressure range, 4×10^{-4} to 10 torr. The van't Hoff equation is a straight line representing the dissociation of hydrogen gas from the dihydride phase to form the metal solid solution. Thus, the logarithm of the hydrogen reduced pressure in equilibrium with the solid varies linearly as a function of the reciprocal of absolute temperature. The equation obtained is as follows:

$$\log_{10}p(\text{torr}) = - \frac{11,490 \pm 18}{T} + 10.668 \pm 0.019$$

The errors in the measurements are represented by the standard deviations in the above relationship. These were determined by a computer least-squares analysis. A total of 252 data points representing twelve independent runs were obtained. The statistical treatment of these data demonstrate the success of the use of a metal-gas system for secondary reduced-pressure standards application.

A feasibility study of metal-oxygen and metal-nitrogen system indicates that the same approach can be used for oxygen and nitrogen reduced-pressure standards. The kinetics of obtaining equilibrium are much lower than with hydrogen; however the initial results of the

feasibility study demonstrate that there is good potential of success. Two metal-nitrogen systems, Cr-N and V-N, and two metal-oxygen systems, Cu-O and Pd-O, were selected on the basis of a literature survey. These systems have all the earmarks of the appropriate characteristics similar to the erbium-hydrogen system.

INTRODUCTION

The progress made to date in conducting absolute reduced-pressure measurement of gases can best be characterized by the lack of significant advance. Compared to the strides made in all other areas of vacuum technology in recent years, the status of such measurements is far behind the pace. The principal difficulty in making these measurements is that current standards of accuracy are not sufficiently reliable. Much attention has been given to this problem, but it is an area fraught with many pitfalls and has resisted successful breakthroughs or unique new techniques.

The demand for a reliable standard exists both in industrial applications and in government defense requirements. Probably of equal importance is the demand of space technology for an accurate measure of reduced pressure. Much research has been conducted both for in-flight and ground-facility programs by National Aeronautics and Space Administration where accurate measurement is a critical need.

The accurate measurement of the reduced pressure of a gas or gas mixture is very difficult according to the present state of the art in the pressure range below 10^{-3} torr pressure. Even above 10^{-3} torr, difficulties beset the scientist in making absolute measurements. There are no really good partial-pressure standards, except for the manometer, and its range is in the high-partial-pressure region where there is reduced interest. The McLeod gauge in the next lower range of partial pressure is fairly satisfactory above 10^{-3} torr, but extreme care must be employed in measurements if one desires any degree of accuracy. In the lowest range which can be measured by present-day techniques from 10^{-4} to 10^{-11} torr, the hot- or cold-filament ionization gauges are in the most common practice, but these leave much to be desired in accuracy and reproducibility. They are simply too sensitive to so many variables which can cause errors. It is this range in which present-day scientists are commonly making measurements, but without much reliability. One of the major problems in partial-pressure measurements is having good primary and secondary standards from which to calibrate

these instruments. Also, the techniques that are used for ionization-gauge calibration are indirect, complicated, and not dependable with time.

Complementary to the problem of accurate measurement of reduced pressure is the difficulty in establishing a known and specific gas atmosphere at a required low pressure. The ability to simulate space conditions for the testing of materials, components and systems under the environment they will encounter between earth's atmosphere and a hard space atmosphere is the best example. This requirement arises many times in research and has resisted an easy and practical solution.

With both of these critical needs in mind, it was the objective of the research program described in this report to attempt a new and fresh approach to these problems. The essence of the approach was to employ the thermodynamic properties of a metal-gas system wherein the gas in equilibrium with the metal obeyed precise and predictable laws of low-pressure behavior. Hydrogen was selected as the specific gas to be studied because there was considerably more experience available both in the literature and in this investigator's background. The potential for hydrogen existed; it just needed additional development and refinement. This goal has been accomplished, and it is felt by the author that metal-gas systems, per se, have considerable promise as secondary reduced-pressure standards, for calibration of reduced-pressure sensing devices, and for simulation of predetermined reduced pressures.

BACKGROUND

One of the most serious difficulties with vacuum technology today is that of the measurement of the quantitative degree of vacuum.^{1-7*} It can be seriously estimated that as much research has gone into the development of reliable means and methods to measure vacuum than in the rest of all vacuum technology. Not only is there doubt about the accuracy of gauges for low-pressure measurements, but even more so with pressure standards or gauge-calibration techniques.⁸⁻¹⁰ It is the objective of this proposal to come to grips with the measurement and standards problem with a new and unique approach.

The calibration of a vacuum gauge requires either that the gauge itself is absolute or that a comparison standard is employed with which

* Citation of references.

to reference the gauge. The only two gauges which can be classified as absolute are the manometer and the McLeod gauge. The manometer is reliable, but the McLeod gauge requires so many corrections to be made and precautions to be taken that the absolute accuracy is only relative. The significance of the term absolute is that the result of a measurement of pressure can be quantitatively derived using only the basic physical parameters of the gauge itself and accepted physical laws of behavior of gases. The ionization gauge is not absolute since it does not determine pressure directly, but a quantity proportional to pressure. Thus, it demands calibration against either a primary standard or a secondary standard. Secondary standards do not have to be absolute, but their calibration must be obtained from a primary standard. The only sound basis for accurate measurements to be made with ionization gauges is to require its calibration against either a secondary or primary standard. The secondary standard is more practical because it is a standard which is easily accessible.

The technique to be described herein is a whole new and unique approach to setting up partial-pressure standards by which calibrations of ionization gauges can be conducted in a much simpler and more reliable manner. Furthermore, the technique can be employed as a pressure standard in the McLeod gauge and manometer region. Thus, with only one technique, the whole range of partial pressures can be bracketed. However, the need is most serious in the ionization-gauge region.

The selection of hydrogen as the gas to produce a standard is appropriate because it is probably the most difficult of all the gases to obtain accurate reduced-pressure measurements. If this gas can be pressure standardized, the other active gases such as oxygen and nitrogen will be much simpler to develop at a later date. Particularly with the ionization gauge, measurements of hydrogen reduced pressures are beset with many problems. Foremost of the problems is reaction of hydrogen with the hot tungsten filament to produce atomic hydrogen. This dissociation is accelerated as the temperature increases. The reaction is further affected by the condition of the surface of the tungsten. If other gases are absorbed, the reaction rate is modified, accordingly. Various auxiliary reactions can occur. Thus, it is impossible to obtain reproducible results because the temperature and surface condition of the tungsten filament are not subject to control. Several other artifacts occur as a result of the production of atomic hydrogen. First, the atomic hydrogen reacts with either glass or metal container walls to form other contaminating gas species, such as carbon monoxide. Second, erroneous pumping rates occur, because the atomic hydrogen

is readily absorbed on the walls. The only way one can avoid these problems is to keep the temperatures in the gauge tube as low as possible. This reduces the amount of dissociated hydrogen to a minimum. However, most hot-filament ionization gauges operate standardly at temperatures where the reaction of hydrogen with the filament is a serious problem.

The technique to be described relies on the thermodynamic properties of metal-gas systems as a basis for its success. The thermodynamic properties of these systems are fixed and immutable under certain controllable conditions. The property of interest is the reduced pressure of the gas over the solid, which varies as a function of temperature and composition. The manner in which it varies can be determined experimentally and fit to an analytical relationship. Thus, by using a primary standard to measure the pressure as a function of temperature and composition, the system itself becomes a standard. One simply has to establish the temperature and the composition, and the reduced pressure is exactly reproduced. This in turn can be used as a secondary standard to calibrate other gauges. The advantage is that the thermodynamic properties will not change with time or are they affected by other influences that usually affect other pressure standards. By selecting a metal-gas system with a two-phase field, the system becomes an even simpler and more reliable standard. Gibbs Phase Rule states that in such a binary metal-gas system, the appearance of two phases rather than one causes a loss of one degree of freedom. Such is actually the case, wherein the degree of freedom which is lost is pressure. Thus, at a certain fixed temperature, the pressure remains constant no matter what the composition is, as long as the solid system remains in the two-phase field.

A specific working example of a reduced-pressure standard was demonstrated in the determination of the thermodynamics of the erbium-hydrogen system.¹¹ This was the original work which established that the erbium-hydrogen system had potential as a secondary partial-pressure standard. It remained to refine the thermodynamic characteristics in the two-phase region which is the study described in this report. The pressure-temperature-composition relationships in the erbium-hydrogen system were determined experimentally with a Sievert's apparatus. The procedure employed to obtain the pressure-temperature-composition relationships was to develop experimentally a family of isothermal curves of composition versus pressure. Figure 1 shows the family of isotherms for the Er-H system. The solubility relationships can be deduced from these isotherms. As the first amounts of hydrogen

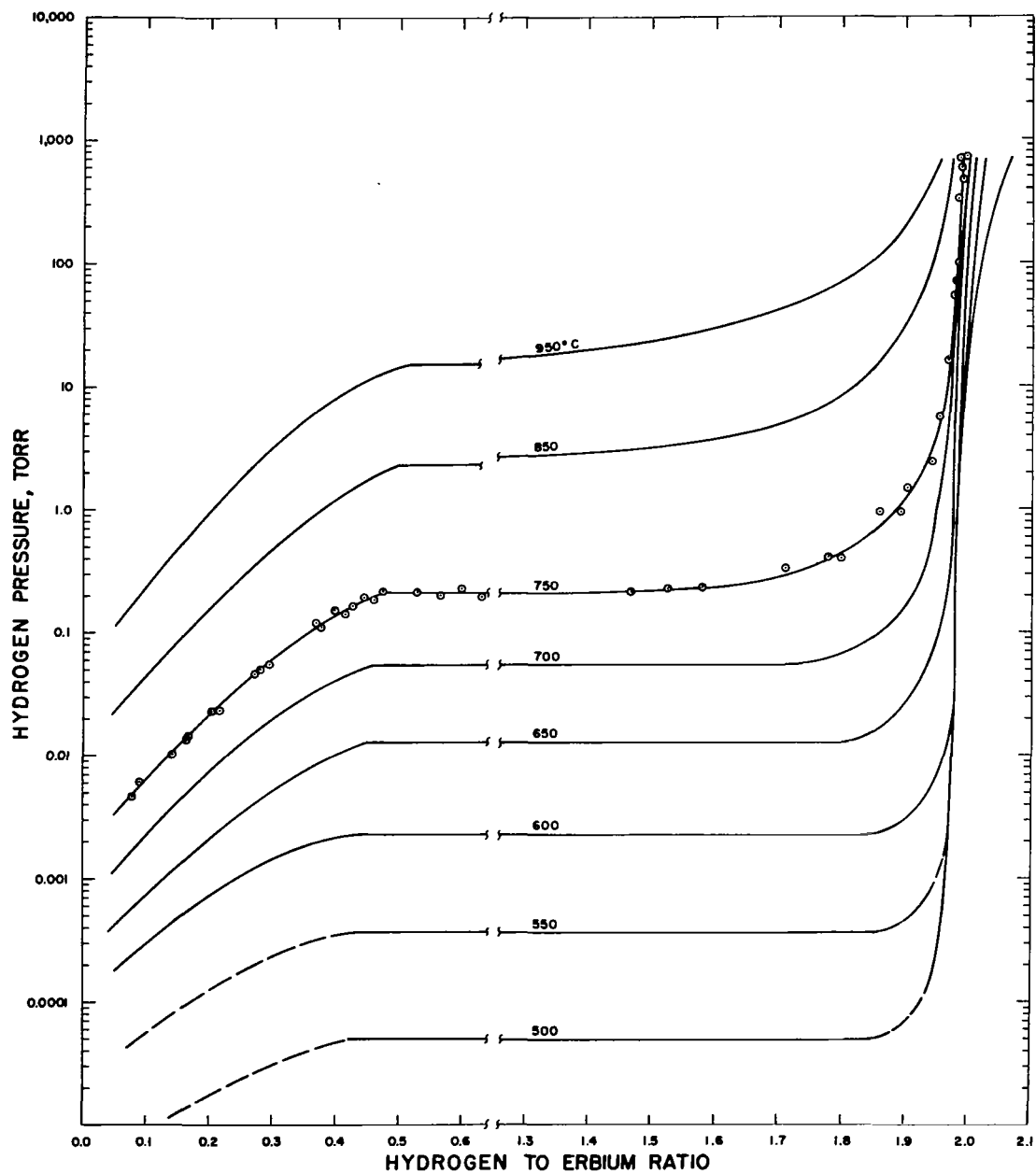


Figure 1. Family of Isotherms in the Erbium-Hydrogen Systems

are added to erbium metal, the pressure increases. At saturation of the metal solid solution, the pressure reaches a plateau. Additional hydrogen initiates the formation of the dihydride phase as the pressure remains constant in this two-phase region. As can be observed, this plateau region is quite extensive. In accordance with the Phase Rule, the appearance of another phase signifies the loss of one degree of freedom, namely that pressure is constant. The pressure again rises from the plateau at the boundary of the single-phase dihydride region. For the purposes of this research study, the plateau region is the center of interest.

The plateau equilibrium reduced pressures were plotted as $\log_{10}p$ versus the reciprocal of the absolute temperature. A straight-line relationship resulted and was quantitatively determined by computer least-square analysis. The following equilibrium-dissociation-pressure equation was obtained.

$$\log_{10}p(\text{torr}) = -\frac{11,500 \pm 70}{T} + 10.57 \pm 0.07$$

where p = equilibrium hydrogen pressure, torr
 T = absolute temperature, Kelvin

The standard deviations are included to show the degree of fit to the straight lines. Three times the standard deviation will include 99.7% of the data points. Figure 2 shows the plot of the log of plateau equilibrium pressure versus reciprocal of absolute temperature. Also included are the dissociation lines for families of constant composition in the solid-solution region.

On comparing the dissociation equation to the van't Hoff equation,

$$\frac{d \ln K}{d \left(\frac{1}{T} \right)} = - \frac{\Delta H^0}{R} \quad (1)$$

one observes that the slope is related to the enthalpy of the reaction occurring at the plateau, and the intercept is related to the entropy of the reaction. By manipulating the van't Hoff equation, it can be brought into the form of the dissociation equation as follows:

K = equilibrium constant for plateau reaction.

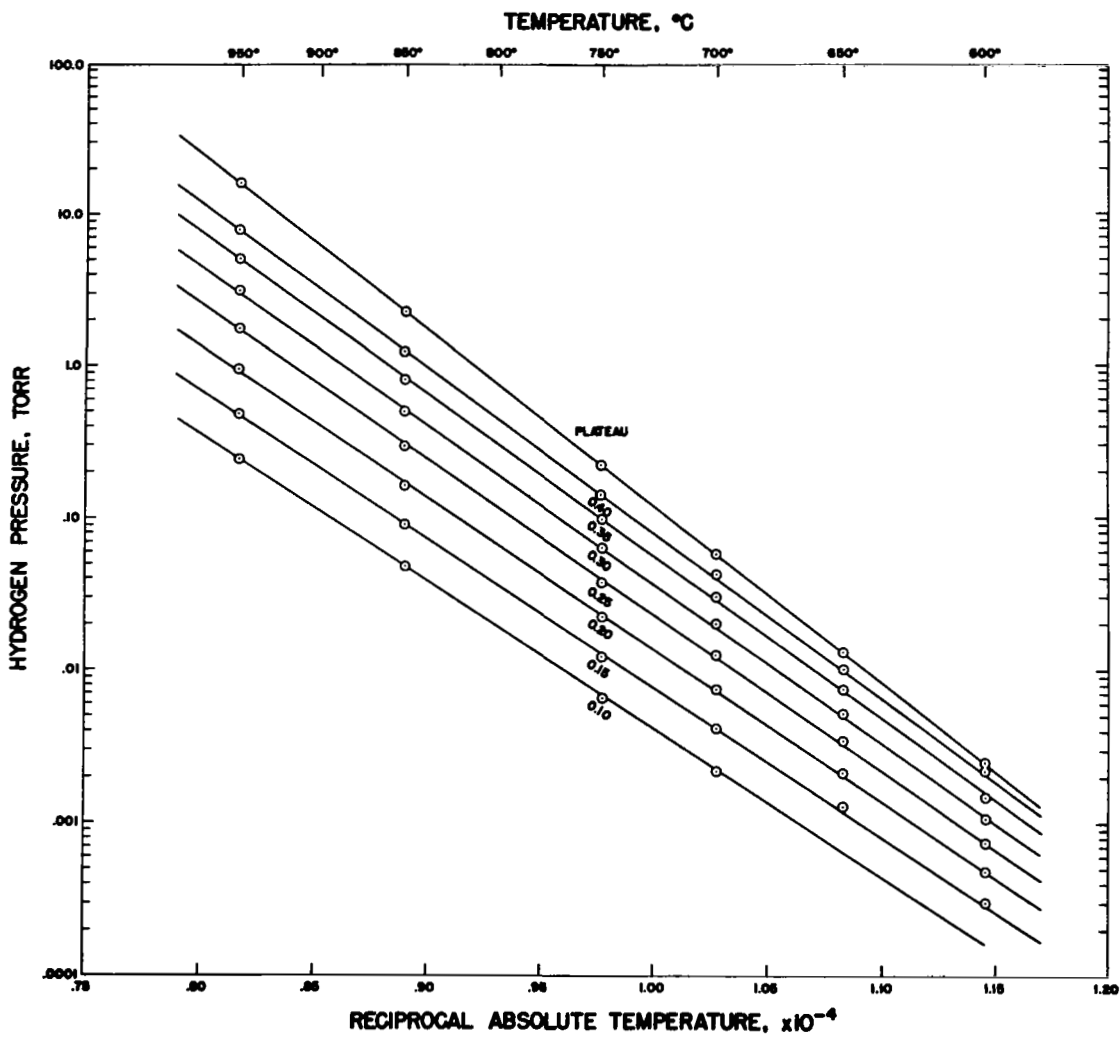
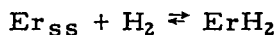


Figure 2. Isopleths in the Erbium-Hydrogen System

The plateau reaction is:



$$K = \frac{a_{\text{ErH}_2}}{P_{\text{H}_2} a_{\text{Er}_{\text{SS}}}} = \frac{1}{P_{\text{H}_2}} \quad (2)$$

$$\frac{d(\ln p)}{d\left(\frac{1}{T}\right)} = \frac{\Delta H}{R} \quad (3)$$

Integrating:

$$\log p = \frac{\Delta H}{2.303 RT} + \text{Constant} \quad (4)$$

This is now in the same straight line form as the dissociation equation. It can be readily seen that the slope of the line, $\Delta H/2.303 R$, will give the enthalpy of formation of the dihydride. The above equation can be manipulated into the Gibbs-Helmholtz type equation, as follows:

$$\Delta F = -RT \ln K$$

$$\Delta F = RT \ln p$$

From Equation (4),

$$2.303 RT \log p = \Delta H + \text{Constant} \times 2.303 RT$$

$$\Delta F = \Delta H + \text{Constant} \times 2.303 RT$$

$$- \frac{\Delta S}{2.303 R} = \text{Constant}$$

Thus, the constant is related to the entropy of the formation of the dihydride. These values for the erbium-hydrogen system are $\Delta H = -52.6 + 0.3 \text{ kcal/mole of H}_2$ $\Delta S = -35.2 + 0.3 \text{ cal/deg. mole of H}_2$.

EXPERIMENTAL

Materials. - Erbium metal was initially obtained from two sources to survey metal quality. The as-received metals were in the form of solid ingot

and sponge, respectively. The reported purities in both cases were 99.9%+, by weight. As a check on the purity, metallographic analysis was conducted on these metals. The relative quantity of second phase appearing in the structure of erbium is an excellent indication of the degree of contamination from such elements as oxygen, fluorine, nitrogen, and tantalum, which are the principal contaminants. After the sponge metal was arc melted and compared structurally with the ingot metal, the sponge was selected for use for this study. This erbium metal was almost entirely free of second phase, whereas the ingot metal had considerable second phase. Another advantage was had in the sponge metal, in that, the rate of attaining metal-gas equilibrium was decidedly greater. Therefore, the commercial source providing the sponge metal was selected to provide the erbium which was henceforth used, since it was felt to be the best quality.

Specimen Preparation. - The as-received erbium sponge was sectioned into small pieces about 0.2 gr in size. No other surface preparation was conducted, since it was felt that more contamination would be added than could be removed. These samples were then stored in a vacuum desiccator until ready for loading in the apparatus. All handling was done with clean tweezers after cutting the appropriate sample size. Prior to loading, the sample was weighed on an analytical balance to the nearest 0.1 mg. To prevent contact with the quartz reaction chamber, the sample was placed in a 1-mil-tungsten-foil capsule. There is practically no reaction at the temperatures employed in this study with tungsten. There would be a reaction with the quartz if this protection were not accorded. The capsule and specimen were then placed in the quartz tube at the bottom of the closed-end section.

Sievert's Apparatus. - A good deal of time was taken to design and construct the modified Sievert's apparatus. This was one of the primary objectives of the program; that is, to provide equipment which would best obtain the required data under a carefully controlled and reproducible environment. Much attention was given to maintaining cleanliness and to the measurement of temperature and pressure. Since the basis for this study was to establish a new approach to establishing reduced-pressure secondary standards, the variables in the process had to be accurately established.

A schematic drawing of the modified Sievert's apparatus inclusive of all the component parts, is presented in Figure 3. Figures 4, 5, and 6 show several photographic views of the apparatus. As can be seen, the apparatus was mounted on an angle-iron-frame cart with retractable

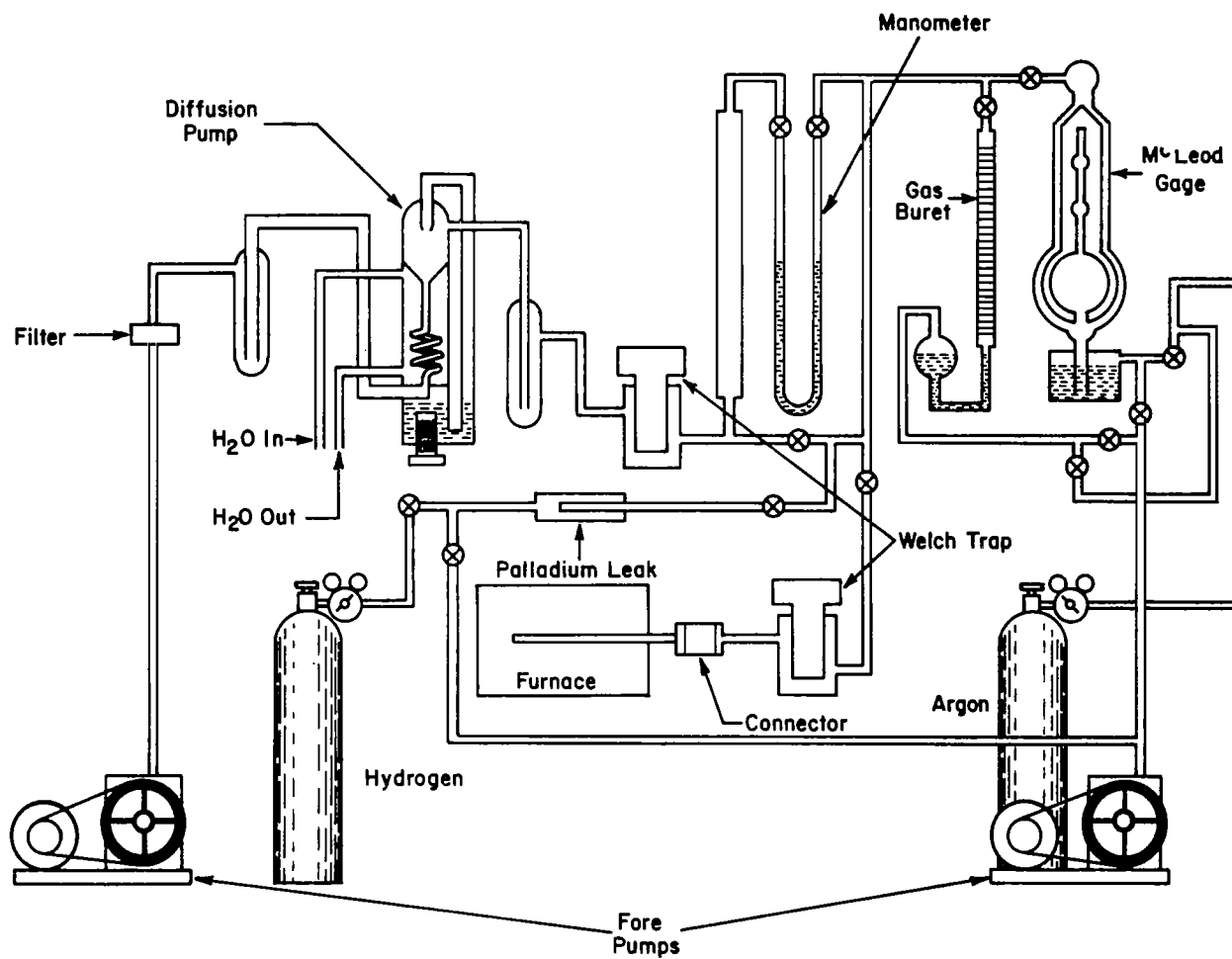


Figure 3. Schematic Diagram of the Sievert's Apparatus

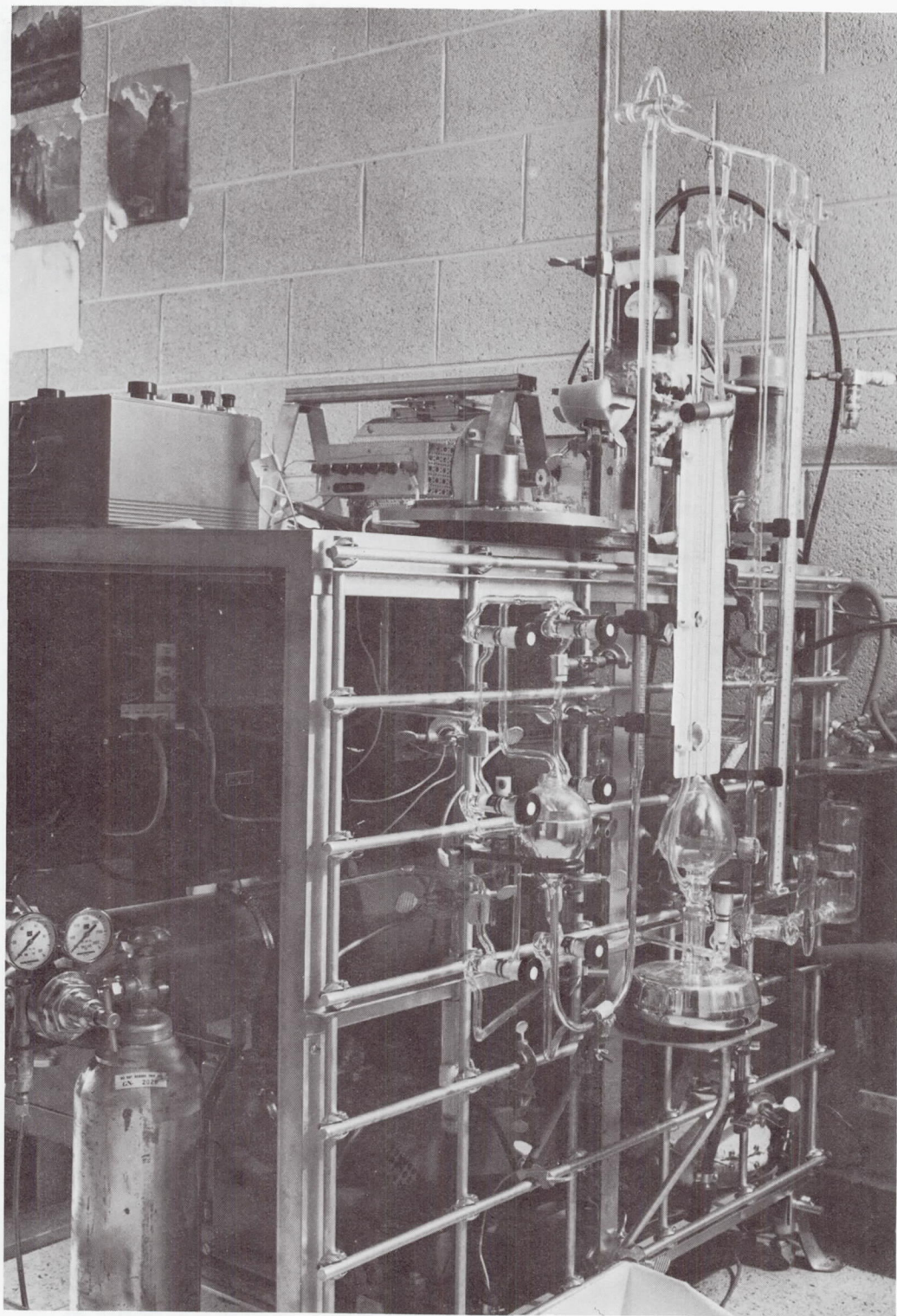


Figure 4. View of the Sievert's Apparatus

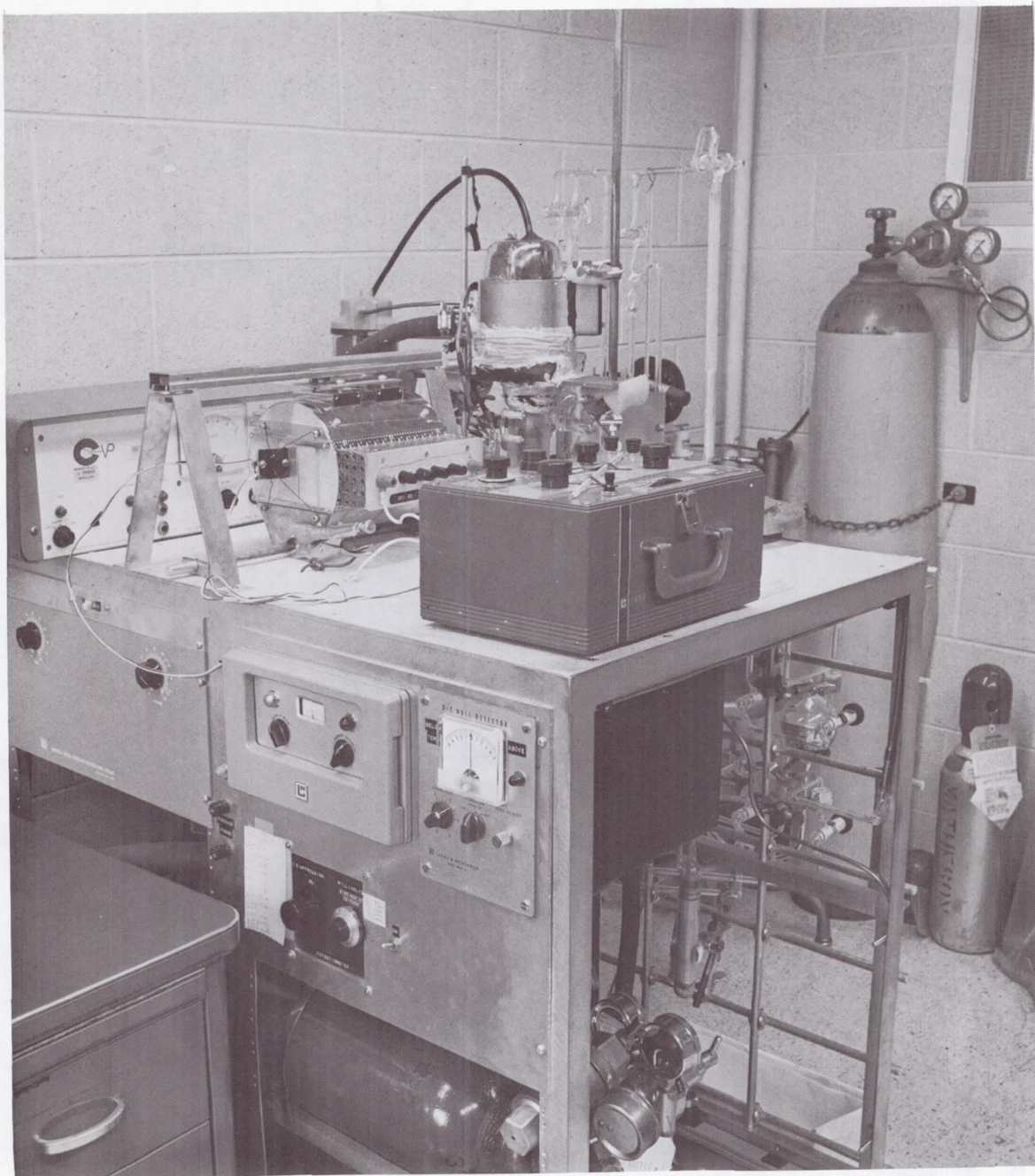


Figure 5. View of the Control System

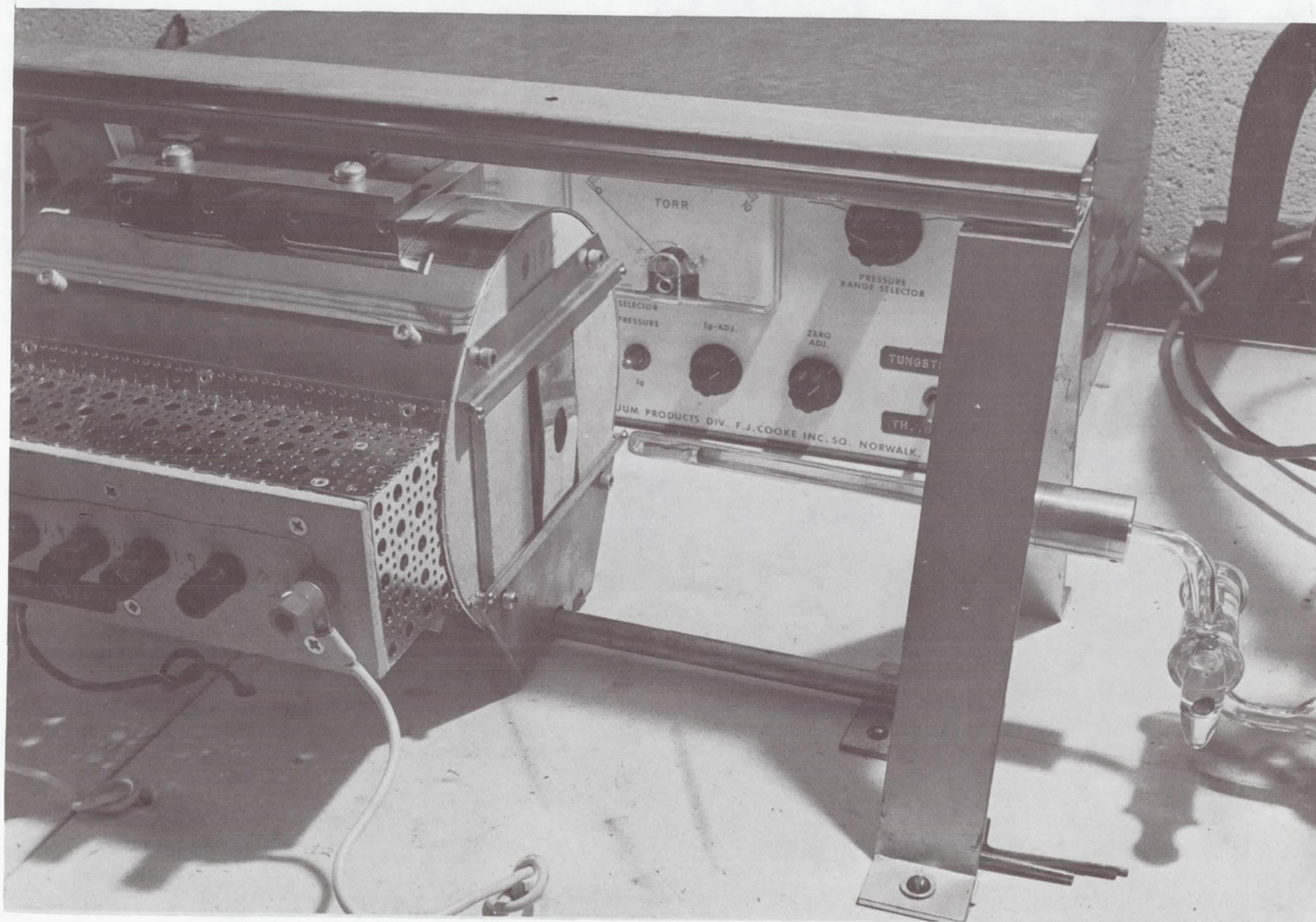


Figure 6. View of the Furnace and Reaction Chamber

wheels. Figure 4 shows the glassware side of the system, and Figure 5 shows the instrumentation and control side. The furnace and the reaction chamber can be seen in detail in Figure 6. The temperature-control system for the furnace was selected after a study of the available systems from the various manufacturers. The unit felt to be the best for this application was a Set-Point Control System. It provided precision temperature control with range flexibility and fast response. It had an adjustable zero suppression and excellent reproducibility of control and control-point setting. The system consisted of 1) a Set Point Unit, 2) a DC null detector, 3) a current-adjusting-type control unit, and 4) a final-control device. The Set Point Unit was a DC potentiometer in the range of 0 to 55 millivolts. A 1000 count, 10-turn dial provided a 5-millivolt span in steps of 5 microvolts, and a suppression switch provided 10 steps of 5 millivolts. Each setting was reproducible to 0.05%. Reference-junction compensation was automatic for chromel-alumel thermocouples which were to be employed. The signal from the thermocouple was bucked against the potentiometer, and the deviation, plus or minus, was fed to the DC null detector. The sensitivity was continuously adjustable to a maximum of 1.0 microvolt per scale division. Since the response of chromel-alumel was 40 microvolts per degree centigrade, this unit detected deviations as small as 1/40 degree centigrade. The signal from the DC null detector was fed to the current-adjusting-type control unit. The control signal was amplified to generate control actions. The unit consisted of a bridge and potentiometer feedback circuit. Its 0 to 5 milliamperes DC output was fed to the final control device. This component consisted of a silicon-controlled rectifier. Its function was to vary the AC output voltage to the tube furnace over a range of 0 to full-rated output in accordance with the 0 to 5 milliamperes DC control current from the current-adjusting-type control unit. The degree of furnace control to be expected was ± 0.1 degree centigrade. The chromel-alumel control thermocouple was calibrated and traceable to Bureau of Standards calibration at 50°C intervals between 550 and 900°C. In addition to this control thermocouple, another independently calibrated thermocouple was employed to measure the exact temperature of the specimen with a Precision Potentiometer. A mock run was initiated at a temperature of 700°C, as measured by the control thermocouple, about midrange of the temperature to be employed in the study. According to the null-point detector reading and its specified sensitivity, the degree of control was ± 0.01 °C. However, another potentiometer was used with the independent thermocouple designed to represent the specimen temperature which is limited to ± 0.25 °C.

The pressure measurements were made in the range of 1×10^{-3} to 10 torr with a McLeod gauge, Model No. GM-100A. The GM-100A covered the 1×10^{-5} to 10 torr range on three scales. On the 0 - 10 torr scale the measuring errors were no larger than $\pm 1.1\%$ of indicated pressure on the 0 - 1 torr scale, $\pm 1.2\%$ of indicated pressure and on the quadratic scale, $\pm 1.5\%$ of indicated pressure. The level measurements were made with a precision cathetometer which was capable of reading to 0.05 mm on its scale.

In the design of the Sievert's system, the criteria employed throughout the design considerations were as follows:

1. To use all Pyrex in the Sievert's apparatus proper.
2. To use components in this section, all of which were bakeable.
3. To use no grease in joints or stopcocks.
4. To avoid migration of mercury and to reduce mercury vapor to a minimum by cold trapping.
5. To obtain the best temperature control possible.
6. To obtain the best pressure measurement possible.

In general it was desired to keep the system as clean as possible from outgassing sources. This was the foremost criteria, as can be evidenced from the items 1 through 4. There was no exception to No. 1. All items were Pyrex glass. The Sievert's system proper consisted of the quartz reaction chamber, the GM-100A McLeod gauge, a precision gas buret, and a mercury manometer. The buret was 100-ml capacity with 0.1-ml graduations. A new high-vacuum, bakeable stopcock was first used throughout the system. Stopcocks had Teflon plugs and were sealed by Viton-A, O-rings. They were bakeable to 150°C . However, these stopcocks had to be replaced, since they later were found to be a major source of outgassing even after baking. They were replaced with high-vacuum stopcocks which were lubricated with Apiezon "N" high-vacuum grease. This grease was reported to have a vapor pressure below 10^{-9} torr. The outgassing problem was therefore reduced to a negligible quantity from these stopcocks. Since mercury had to be employed for the pressure-measuring system, mercury vapor could not be avoided. However, incorporated in the design was a cold trap between the quartz reaction chamber and the rest of the system. The cold trap

was operated continuously, so that mercury vapor was not present in the reaction chamber. This provided a mercury-free atmosphere over the heated surface of the erbium. Residual condensibles were removed in addition. This step was felt to be mandatory to provide the best data. The cold trap was a Series 3150 Cryogenic Refrigerator with the cold finger attaining a temperature of -140°C . It operated continuously by means of compressed air. It eliminated pressure fluctuations due to warm-up and evaporation of frozen, trapped material, as is the case with liquid nitrogen. Another of these traps was placed between the vacuum source and the Sievert's system to eliminate the mercury vapor from the mercury diffusion pump and oil vapor from the forepump. The Pyrex-glass cold trap was built around the cold finger was placed in the dead air space of the cold trap so that the cryogenic unit was independent and outside of the vacuum system. The trapping occurred on the inner glass finger. This arrangement was desirable because the trap could be easily baked, and vibrations from the operation of the cryogenic unit were not transmitted to the glass system. For added precaution, a foreline trap was incorporated to prevent oil backstreaming. The mercury diffusion pump was a three-stage pump capable of an ultimate vacuum of 5×10^{-8} torr. The addition of the cold traps reduced this considerably. Convoil 20 was used in the forepump as added insurance for low oil-vapor pressure in this region of the system.

The source of high-purity hydrogen was the best available. This consisted of hydrogen which was boil-off from liquid hydrogen obtained from the Bureau of Standards in Boulder. It was analyzed and contained less than 1 ppb of impurities. This passed through a palladium-silver filter before usage in the Sievert's system. The gas buret is normally operated with air on the other side of the system as is the McLeod gauge. Instead, purified argon was used to operate both of these devices. This eliminated any possibility of air or dust getting into the mercury and diffusing through to the Sievert's system.

The quartz reaction chamber consisted of a closed-end tube about 9 mm in diameter. It was inserted in a hole through a Lavite plug and into an Inconel heat sink. In turn these were both contained within the 1 in. diameter hole in a tube furnace. The Inconel heat sink was employed to even out thermal gradients and to eliminate temperature cycling from the control system. This assured an extremely even temperature control. A chromel-alumel thermocouple fit into the opposite end of the furnace through another Lavite plug. The Lavite plugs at either end of the furnace served as insulators to keep the longitudinal heat loss down. The thermocouple tip was placed against the closed end

of the quartz reaction chamber which was intended to accommodate the thermocouple. Immediately inside the quartz chamber, the specimen was placed. The temperature of the specimen was very accurately read with this arrangement.

The Nichrome-wound tube furnace was provided with a means of sliding the furnace back and forth. The quartz tube, being stationary, would either be in or out of the furnace as the furnace was moved from one position to the other. This feature facilitated loading and unloading specimens in the quartz reaction chamber. The furnace mounting unit consisted of a heavy-duty framework with two roller systems and a track on which the furnace was bolted. The travel of the furnace was restricted by metal stops at either end so that the furnace could be brought back to the same position.

The initial pump-down vacuum of the Sievert's apparatus was measured with Miniature Ionization gauges placed in two positions in the system, immediately outside of the furnace tube and in the main vacuum manifold. These gauges were used in addition to the McLeod gauge.

On the same glass rack as the Sievert's apparatus, a small bake-out unit for tungsten-foil capsules was set up. It consisted of a small furnace, a quartz tube, and a small forepump. The tungsten capsules were brought to 900°C for bakeout under vacuum, cooled, and stored in vacuum prior to their use.

Hydriding and Equilibration Procedure. - As standard procedure the Sievert's apparatus was intermittently leak-checked between runs with a helium leak detector and always found to be helium leak tight. Prior to the hydriding run itself, the Sievert's apparatus was evacuated to its ultimate vacuum with the two cold traps in operation and the tube furnace heated to the required temperature, but not yet moved onto the reaction chamber. Thus, the erbium specimen was at room temperature at this stage. The system nominally pumped to a vacuum in the low 10^{-7} torr region. A light bakeout of the system glassware to about 150°C was then conducted with the exception of the reaction chamber. The reaction tube was independently baked by moving the tube furnace onto the quartz tube for about 30 sec at 700°C. The specimen did not have an opportunity to heat for this period of time so it could not be contaminated by the out-gassing from the tube, albeit small. The system was then alternately purged with high-purity hydrogen and evacuated to clear the system of any minor remaining contaminating gases. The hydriding was initiated at this stage. From the weight of the specimen, the amount of hydrogen

gas required to bring the composition to 50 atomic percent hydrogen was calculated. The composition relationship used henceforth in this report instead of atomic percent is the ratio of hydrogen to erbium, or 1.0 H/Er in this case. This value was selected because it is approximately midway between the solubility limits of the metal solid solution and the dihydride phase. Thus, the region is two-phase where the experiments were conducted to assess the potential of the metal-gas system as a reduced pressure standard. The phase equilibria and their relationship to the requirements for a standard will be discussed in more detail in a subsequent section. An amount of gas was then admitted into the precision gas buret, and its volume and pressure were measured to determine the quantity. By admitting or removing small amounts of gas, the correct quantity to bring the erbium metal to 1.0 H/Er was obtained. At this point, the furnace was moved onto the quartz reaction tube containing the erbium specimen. Evacuation of the system was continued for about 5 min. while the specimen was coming to temperature. A temperature of 700°C was arbitrarily selected to conduct the initial hydriding to composition. The stopcock to the vacuum source was then closed and the gas in the buret admitted. The only components of the system involved in the equilibria measurements from this point on were the manometer, the McLeod gauge, the quartz reaction tube and interconnecting tubing. The cold trap between the quartz reaction tube and the manometer and McLeod gauge was maintained at about -140°C. The temperature-pressure equilibria study was initiated at this stage. The equilibrium point was taken at 700°C and the temperature adjusted to the next level, usually about an increment of 25°C above. Equilibrium was allowed at this temperature, the pressure measured, and the next level was set. This procedure was continued up to 950°C, and then the cycle reversed downward in similar temperature increments to 550°C. Before the run was completed, many thermal cycles between these limits were conducted to obtain the temperature-pressure equilibria data. There was no indication of hysteresis observed in the data obtained in the downward temperature cycles as compared with the upward cycles. This was further proof that equilibrium was attained. An average run was conducted for a week before the specimen was removed and replaced with another. However, this was only a precautionary measure to be assured that excessively long periods of time at temperature did not contaminate the specimen. Several runs were carried out for as long as two weeks without any problems. Each run was cycled up and down an average of about eight times. Again the equilibria did not shift as a result.

RESULTS AND DISCUSSION

Based on the pressure-temperature-composition study of the erbium-hydrogen system described in the "Background" section, this study was undertaken with the prime objective of constructing a more sophisticated Sievert's apparatus and obtaining highly refined pressure-temperature data therefrom in the two-phase region. The statistics of the data were then to be carefully analyzed and assessed for application as a secondary reduced-pressure standard. The range of temperature selected was from 550 to 950°C with the resulting pressures being from 10^{-4} to 10 torr, or a difference of 5 orders of magnitude in pressure.

A total of 24 independent runs were made during the course of this program representing 412 individual data points. However, a portion of the runs were conducted with the Teflon-bore, greaseless stopcocks in the system. It was determined that these were outgassing excessively, causing the statistics of the error analysis on the van't Hoff plots to be rather poor. When these were replaced with high vacuum, all-Pyrex stopcocks, the internal outgassing was reduced to a negligible amount. The statistics of the error analysis were reduced noticeably. Therefore, the earlier data were discarded. As a result, the data presented in this final report are from 12 independent runs representing 252 data points. These data are tabulated in Table I with each data equilibrium point presented in terms of the temperature and the equilibrium pressure. The points are given in the sequence of the thermal cycling that was employed. A least-square computer analysis was conducted for each run on a Burrough's 5500. The least-square analysis was formulated for the van't Hoff equation which represents the data obtained in hydriding. A more complete treatment of the thermodynamical relationships involved were presented in the Background. Briefly, however, the van't Hoff equation is as follows:

$$\log_{10}p(\text{torr}) = -\frac{A}{T} + B$$

where p is the plateau hydrogen pressure in equilibrium with the solid, T is the absolute temperature, and A and B are constants representing the slope and the intercept of the straight line, respectively. Table II is a summary of the van't Hoff equations for the 12 runs, listing the equation constants and the standard deviation for each.

TABLE I

Equilibrium Pressure-Temperature Data

Run No. 1		Run No. 2 (Cont.)	
<u>Temperature, °C</u>	<u>Pressure, torr</u>	<u>Temperature, °C</u>	<u>Pressure, torr</u>
747	0.268	551	0.0005
857	3.150	656	0.019
901	6.98	654	0.016
807	1.16	652	0.018
703	0.075	651	0.017
601	0.0037	750	0.290
553	0.005	748	0.275
		748	0.270
		747	0.275
		848	2.62
		846	2.50
		843	2.48
		845	2.49
		902	6.97
			Run No. 3
		747	0.275
		848	2.63
		903	7.11
		808	1.20
		698	0.067
		603	0.0037
		550	0.0005
		655	0.018
		751	0.296
		902	6.90
		806	1.16
		698	0.083
		602	0.0037
		549	0.0005
		653	0.0185
		847	2.53

TABLE I (Cont.)

Run No. 4		Run No. 6	
<u>Temperature, °C</u>	<u>Pressure, torr</u>	<u>Temperature, °C</u>	<u>Pressure, torr</u>
747	0.260	747	0.270
846	2.52	847	2.33
902	6.90	902	7.20
806	1.15	807	1.11
697	0.062	698	0.068
602	0.0033	601	0.0036
549	0.0004	551	0.0005
654	0.018	654	0.019
749	0.286	750	0.297
846	2.62	847	2.67
901	6.96	901	7.10
551	0.0005	805	1.10
656	0.019	698	0.066
750	0.290	602	0.0038
849	2.71	549	0.0005
902	7.00	655	0.019
806	1.15	750	0.295
700	0.070	847	2.65
604	0.0037	902	7.12
Run No. 5		Run No. 7	
747	0.267	747	0.278
808	1.20	848	2.75
902	6.96	895	6.52
844	2.45	805	1.14
745	0.254	698	0.068
547	0.0004	601	0.0035
605	0.0039	548	0.0004
654	0.018	654	0.019
702	0.0735	750	0.286
810	1.23	848	2.63
551	0.0004	897	6.48
656	0.0195	698	0.069
748	0.270	547	0.0004
847	2.63	549	0.0005
903	6.90	552	0.0005
806	1.06	655	0.020
699	0.066	751	0.310
602	0.0036	849	2.77
551	0.0005	903	7.34
654	0.018	805	1.10
749	0.280	698	0.070
848	2.69	601	0.0037
903	7.00	551	0.0006

TABLE I (Cont.)

Run No. 7 (Cont.)		Run No. 9 (Cont.)	
<u>Temperature, °C</u>	<u>Pressure, torr</u>	<u>Temperature, °C</u>	<u>Pressure, torr</u>
654	0.019	602	0.0038
751	0.310	652	0.0175
848	2.73	747	0.282
901	7.20	859	3.53
Run No. 8		Run No. 10	
747	0.278	744	0.271
845	2.66	858	3.50
901	7.27	893	6.74
806	1.11	793	0.897
698	0.068	703	0.081
601	0.0035	601	0.0040
551	0.0005	651	0.0180
655	0.018	745	0.276
748	0.286	856	3.40
848	2.79	899	7.48
902	7.28	793	0.893
805	1.11	552	0.0008
552	0.0005	653	0.0195
602	0.0042	747	0.289
702	0.075	855	3.39
809	1.19	905	8.41
902	7.39	792	0.862
844	2.57	697	0.071
745	0.270	600	0.0040
651	0.018	650	0.0170
547	0.0005	746	0.290
Run No. 9		858	3.63
		899	7.63
549	0.0005	Run No. 11	
604	0.0040	746	0.260
723	0.146	792	0.850
795	0.666	899	7.22
895	6.72	803	1.11
857	3.32	701	0.075
743	0.256	600	0.0034
551	0.0005	651	0.0165
652	0.017	748	0.280
746	0.282	859	3.40
857	3.45	551	0.490
894	6.62	606	0.0040
793	0.883	706	0.0845
704	0.082		

TABLE I (Cont.)

Run No. 11 (Cont.)		Run No. 12	
<u>Temperature, °C</u>	<u>Pressure, torr</u>	<u>Temperature, °C</u>	<u>Pressure, torr</u>
807	1.20	859	3.29
900	7.18	903	6.63
855	3.21	807	0.996
745	0.261	704	0.0720
647	0.0150	748	0.252
651	0.0165	860	3.15
546	0.0005	902	6.65
604	0.0037	808	1.10
706	0.088	704	0.071
		602	0.0033
		549	0.0004
		551	0.0005
		654	0.0175
		751	0.278
		903	6.73
		807	1.10
		705	0.745

TABLE II
Summary of van't Hoff Equations

<u>Run No.</u>	<u>No. of Points</u>	$\log_{10}P(\text{torr}) = -\frac{A}{T} + B$	
		<u>A</u>	<u>B</u>
1	21	-11,450 ± 50	10.57 ± 0.05
2	7	-11,550 ± 130	10.70 ± 0.13
3	36	-11,500 ± 40	10.65 ± 0.04
4	16	-11,450 ± 70	10.64 ± 0.08
5	19	-11,540 ± 60	10.71 ± 0.06
6	23	-11,550 ± 60	10.72 ± 0.06
7	19	-11,450 ± 50	10.62 ± 0.05
8	28	-11,500 ± 40	10.69 ± 0.04
9	21	-11,450 ± 50	10.65 ± 0.05
10	18	-11,550 ± 80	10.75 ± 0.08
11	23	-11,433 ± 70	10.65 ± 0.07
12	21	-11,540 ± 50	10.73 ± 0.05
Total	252	-11,490 ± 18	10.668 ± 0.019

All of the runs were then analyzed collectively. The total least square analysis of data is presented in the same table. The equation obtained was as follows:

$$\log_{10}P(\text{torr}) = -\frac{11,490 + 18}{T} + 10.668 \pm 0.019$$

This shows a decided improvement over the initial studies reported in the Background section which led to this study. The standard deviations of the slope and intercept have been reduced by factors of 3.9 and 3.7, respectively. The Coefficient of Determination is 0.999. This means that 99.9% of the variation in the dependent variable can be explained by the independent variable. This is a measure of the predictive value of the independent variable. The Standard Error of Estimate is 0.03. The

error amounts are now felt to be of the caliber required for standards consideration. The advantages of the erbium-hydrogen system as a secondary standard or a calibration device for hydrogen gas are felt to be numerous and distinct, now that the error analysis has proven worthy. They are as follows:

1. Once the dissociation pressure equation is established and standardized, one can calculate the corresponding temperature setting for any desired pressure. Then, by adjusting the temperature to the appropriate value, the pressure can quickly be attained.
2. There is no hysteresis in the hydrogen pressure. The system can be cycled any number of times, and the pressure will return to the value as prescribed by that specific temperature.
3. In this temperature range, pressure equilibrium is very rapid.
4. The equilibrium pressure for each respective temperature is independent and unaffected by the volume or geometry of the system. There is an exception to this when the composition is shifted off the plateau by having a very small hydride specimen and a very large system.
5. The partial-pressure plateau in the two-phase region is very extensive, and shifts in overall hydrogen composition do not affect the pressure at any specific temperature.
6. Contamination from outgassing or inleakage does not seriously affect the equilibria established according to the dissociation equation.
7. A metal-gas system such as the erbium-hydrogen system will establish accurate reduced pressures which bracket the manometer, the McLeod gauge, and the ionization gauge in a continuous manner. One doesn't need a separate calibrating system for each type of measuring device.
8. There is no reason to believe that a standardized dissociation equation for such a system prepared in the higher pressure range where good primary standards exist could not be extrapolated to lower pressures with a high degree of confidence in accuracy. This is so because of the immutability of the thermodynamic properties. It is known that in the lower temperature regions, where the lower equilibrium pressures obtain, no phase changes occur in either the metal

solid solution or the hydride. Furthermore, the solubility boundaries change slowly with temperature. Therefore, the enthalpy and entropy values for the reaction across the two-phase region are not expected to change over quite a range of temperature. This means the dissociation equation can be confidently extrapolated. This feature is probably one of the most important advantages to be gained using this type of system. For the first time, a reliable secondary standard can be available where before absolutely nothing existed. For instance, Table III below shows some of the calculated hydrogen reduced pressures in the high- and ultrahigh-vacuum region.

TABLE III
Extrapolated Hydrogen Reduced Pressures

<u>Temperature, °C</u>	<u>Pressure, torr</u>
450	4.65×10^{-6}
350	1.17×10^{-8}
300	2.70×10^{-10}
250	3.70×10^{-12}
200	9.30×10^{-15}

Having such a capability to establish rather well-known pressures in this range opens some exciting possibilities to the ultrahigh vacuum technologist.

9. One distinct advantage of this technique is that an in situ calibration can easily be devised for any ultrahigh vacuum apparatus, where an immediate calibration of the measuring apparatus can be available. All ionization gauge calibration is currently done on another piece of equipment, the gauge physically removed from the calibration equipment, and placed into service in another apparatus. Since the original calibration can be easily affected by so many factors, it would be surprising if the calibration would last long enough to make the first vacuum measurements accurately. In use, the gauge undoubtedly shifts calibration. However, with a metal-gas system adjunct to the primary apparatus, the gauge could be recalibrated at any time desirable.

10. The system is inexpensive to build and relatively simple in design and use. The temperature range can be easily attained and measured with commercially available thermocouples. The components for the apparatus to contain the system are commercially available. There are no electronics involved, which considerably lessens the chance for error. The erbium metal is not expensive for the amount required.
11. The erbium can be hydrided to the appropriate composition, removed to air, and transported to another system without affecting its dissociation relationships.
12. Other metal-hydrogen systems exist which give somewhat different equilibria. This would give a lot of flexibility in selecting the appropriate temperature and partial pressures for the specific application.
13. The range of temperatures over which the wide spread of pressures exist are easily measured. A chromel-alumel thermocouple can be employed which has a very high EMF per unit temperature increase. Also, standard materials of construction for the furnace chamber can be employed because the highest temperatures required are not at all severe.
14. The simulation of any hydrogen reduced pressure atmosphere is feasible in any type of chamber or system by simply placing a small heating source and a prehydrided specimen in the chamber with a means for measuring the temperature of the hydride.

Metal-oxygen and metal-nitrogen systems may be developed for secondary partial pressure standards in the same manner. This would make available standardization or calibration sources of all of the common contaminating gases. A feasibility study was included in this program of several candidate metal-oxygen and metal-nitrogen systems to assess their potential. A literature survey was first conducted to select likely prospects in both types of systems. A bibliography of the literature survey is presented in the Appendix. The systems are presented in Table IV. The table lists their thermodynamic stability, expected temperature range of operation, and melting point. Out of this group, two metal-oxygen and two metal-nitrogen systems were arbitrarily selected. These were the Pd-O and Cu-O systems and the Cr-N and V-N systems. The principal objective was to determine the kinetics of absorption, and the equilibrium pressure level at 800°C for an amount of gas equivalent

TABLE IV
Metal-Oxygen Systems

<u>System</u>	<u>Reaction</u>	<u>Stability</u> <u>ΔF_{298}</u>	<u>Expected Temp.</u> <u>Range, °C</u>	<u>Melting, °C</u>
Fe-O	$4\text{Fe}_3\text{O}_4 + \text{O}_2 \rightarrow 6\text{Fe}_2\text{O}_3$	-96 kcal/mole O_2	700-1200	>2000
Cu-O	$2\text{Cu} + \text{O}_2 \rightarrow 2\text{CuO}$	-61	400-1000	1083
Pt-O	$2\text{Pt} + \text{O}_2 \rightarrow 2\text{PtO}$	-22	50-300	1769
Pd-O	$2\text{Pd} + \text{O}_2 \rightarrow 2\text{PdO}$	-28	120-400	dec 900
Os-O	$\text{Os} + \text{O}_2 \rightarrow \text{OsO}_2$	-49	300-600	dec 600
Rh-O	$4\text{Rh} + \text{O}_2 \rightarrow 2\text{Rh}_2\text{O}$	-37	400-900	dec 1100
Ir-O	$4/3\text{Ir} + \text{O}_2 \rightarrow 2/3\text{Ir}_2\text{O}_3$	-28	300-600	dec 1200
Cr-N	$2\text{Cr} + \text{N}_2 \rightarrow 2\text{CrN}$		400-900	1900
V-N	$2\text{V} + \text{N}_2 \rightarrow 2\text{VN}$		700-1200	1730
Mo-N	$4\text{Mo} + \text{N}_2 \rightarrow 2\text{Mo}_2\text{N}$	-21		2610
Si-N	$3/2\text{Si} + \text{N}_2 \rightarrow 1/2\text{Si}_3\text{N}_2$	-77		1410
Ba-N	$3\text{Ba} + \text{N}_2 \rightarrow \text{Ba}_3\text{N}_2$	-73	600-800	704
B-N	$2\text{B} + \text{N}_2 \rightarrow 2\text{BN}$	-59	800-1600	2040
Cu-N	$6\text{Cu} + \text{N}_2 \rightarrow 2\text{Cu}_3\text{N}$		> 450	dec 450
Sn-N	$3\text{Sn} + 2\text{N}_2 \rightarrow \text{Sn}_3\text{N}_4$		> 360	dec 360
Ge-N	$3\text{Ge} + 2\text{N}_2 \rightarrow \text{Ge}_3\text{N}_4$			959
Pt-N				1769
Pd-N				1559

to about 30 atomic percent. All systems were able to absorb the quantity of gas admitted in a reasonable period of time and reach what was felt to be an equilibrium state. The kinetics of absorption were considerably slower than with the hydrogen system. Rather than attaining equilibrium in minutes, it was a matter of hours or days for the oxygen and nitrogen systems. The pressures were in the range of 10^{-2} to 1 torr at 800°C for these systems. Time did not allow for definitive studies. However, these systems appear to be feasible systems for future study.

CONCLUDING REMARKS

A new and unique approach to reduced-pressure standardization and gauge calibration was studied and identified as a technique with considerable merit in this respect. The technique employs the characteristics of a metal-gas system, wherein, the gas is in equilibrium with the solid at known and highly reproducible reduced pressures. The system studied was the erbium-hydrogen system. It was found that the thermodynamic properties of this metal-gas couple lend themselves admirably to secondary standards or calibration applications. The technique employs the pressure-temperature relationships in the two-phase region of the system, where erbium solid solution coexists with erbium dihydride. A mathematical relationship exists in the two-phase region between pressure and temperature which thermodynamically is described as the van't Hoff relationship. The relationship was determined to be as follows:

$$\log_{10}p(\text{torr}) = - \frac{11,490 \pm 18}{T} + 10.668 \pm 0.019$$

A carefully designed and constructed Sievert's apparatus was employed to establish this equation. The equation is a straight line relation between the logarithm of pressure and reciprocal of absolute temperature. The data used to establish the van't Hoff equation were treated by a computer least-square analysis to determine the error limits. As can be observed the standard deviations are very low. Knowing this equation, one need only attain the appropriate temperature and then calculate the resulting reduced pressure. The technique has many advantages over other more traditional types of standards or calibration sources. The salient advantages are its degree of reproducibility over long time periods, its coverage through the manometer, McLeod, and ionization gauge reduced pressure ranges while employing only one system, its complete absence of the dependence on any electronic systems which gives it an absolute stability, and its relative simplicity both in equipment and use.

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APPENDIX

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